

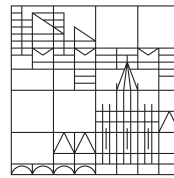
Fabrication of a Superconducting Single-Electron Transistor

Projektpraktikum Report

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1 Introduction

Single-Electron Transistors (SETs) are built by coupling a metallic island with two weak links. These devices show an effect which is called Coulomb blockade, where electronic transport is suppressed for low bias voltages. The reason for this blocking effect is the charging energy of the island which is equivalent to the energy one has to pay to add/remove an electron to/from the island. An additional parameter to control the transport is a gate voltage. Fabricating SETs out of superconductors instead of normal metals opens up the possibilities for further effects.

Even though normal- and superconducting SETs have been investigated since the 90s, there are still not fully understood, yet there is steady progress. More recent publications have shown for example how Coulomb oscillations vanish when the charge fluctuations increase as a consequence to have a junction with almost perfect transmission [1] and how Coulomb blockade and multiple Andreev reflections (MAR) interact [2].

Most SETs are produced with tunnel barriers (e.x. aluminum oxide) as weak links to the island. The SET to be studied here is therefore special since one of the oxide barrier has been replaced by a mechanically controllable break junction (MCBJ) which allows to adjust the resistance of the junction *in situ*. Other than with an oxide barrier, which has many channels with low transmission, a MCBJ can form an atomic contact with almost perfect transmission.

To understand all superconducting SETs (SSET) better, samples with superconducting break junction, but a normal conducting lead to the oxide barrier, have lastly been investigated. This report thus describes the fabrication of SSN-type SETs using aluminum as superconductor, aluminum oxide as insulating tunnel barrier and copper as normal metal. The next step of this research project, which is continued within the scope of the Collaborative Research Center SFB 1432 is to irradiate an all superconducting SET with microwaves to perturb the system in a controlled way. The sample fabrication for these SSETs is done exactly analogous with the only difference being that, the copper is replaced by another layer of aluminum.

The present report explains the steps necessary to fabricate thin film MCBC-SETs with aluminum and copper providing some more backgrounds than typical fabrication chapters. It also addresses various problems and solutions as well as changes and improvements in the fabrication process. To make the fabrication process a closed matter, some things as for example the improved polishing or exchanging old resists for new ones, which were actually done later during the work on my master thesis, are included in this report. An overview of the fabrication process can be found in table 4.1 in the appendix.

2 Fabrication procedure

The SSET samples are so called lift-off devices and are prepared using the technique of electron-beam lithography (EBL). This nanofabrication method generally requires the steps of coating a substrate with electron sensitive resist, exposure, development, thin film deposition and lift-off. A peculiarity of our sample is that it was designed to be operated in a mechanically controlled break junction (MCBJ) setup. Therefore, we begin the fabrication process by coating the substrate with a sacrificial layer, which is etched in a last step in order to form the freestanding constriction necessary for the MCBJ technique. Sample fabrication using EBL to produce lift-off devices is generally a well known procedure. A nice visualization for the fabrication of break junction samples can be found in [3]. Figure 2.1a shows the break junction mechanism, where a rod is pushing the sample against two counter rods and therefore bends the substrate. This pulls the freestanding constriction apart and forms an atomic contact. An SEM image of an etched break junction SET can be found in figure 2.1b.

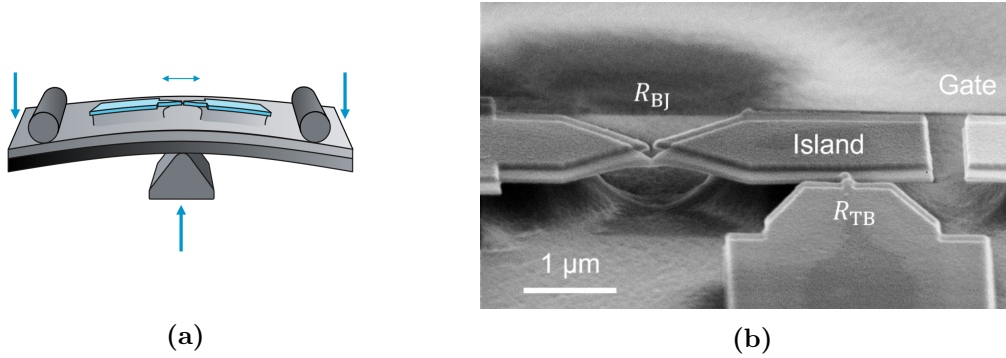


Figure 2.1: (a) Mechanically controllible break junction mechanics showing how a sample is bent to tear the narrow freestanding constriction apart in order to form an atomic contact. The image was adopted from [3]. (b) SEM image of a SET with a break junction.

2.1 Preparing the wafer

As the samples are bend in the MCBJ set up their shape is very elongated (dimensions of 3 x 18 mm). However, this is very unfavorable for coating them with a thin layer via spin coating, because pileup effects at the edges come into play more strongly here. Hence, all fabrication steps up to the last iteration of spin coating are done on a larger wafer (dimensions of 50 x 65 mm) before cutting the samples down to their final size.

2.1.1 Polishing

In order get good quality results from electron-beam lithography, we need a very planar, yet not atomically smooth, substrate. Already micrometer-scale height variations cause

focus problems, on the other hand an atomically flat substrate will cause adhesion problems for the coating layers. To get a sufficient uniformity of height the bronze wafer is polished mechanically. Prior to that one has to ensure, that the protective plastic foil and glue residues from it are removed entirely, the wafer is fixed properly to a block of metal using double sided tape. Cooling the wafer down with liquid nitrogen makes the plastic foil brittle and therefore eases taking it off. The same trick can be used to remove the wafer from the sticky tape after polishing. Glue residues are best removed with acetone.

Formerly, the polishing was done using a hand drill machine, which was mounted on a stand horizontally, and a polishing tissue attachment with solid polish. Unfortunately, the grain size of the polish was unknown and as the bloc where the wafer is glued onto is hand-guide, the varying contact pressure may cause rather height variations on a scale of couples of millimeters. This is why we changed to polishing all manually using wet sandpaper of different roughness on a glass slide to have a very flat surface and apply pressure evenly. Namely, we used sandpaper of with the following grits: 600, 800, 1000, 1500, 2000, 2500, 3000, 4000 and liquid polish with grain sizes of $3\text{ }\mu\text{m}$ and $0.25\text{ }\mu\text{m}$. The conversion from grit to grain size can be approximately done by the following formula

$$\text{grain size in }\mu\text{m} = \frac{25400}{\text{grit}}, \quad (2.1)$$

as the grit number is defined as number of meshes per inch. With this new polishing method we reach comparable shininess if not better for less corrugation over all. Another advantage is the reduced noise exposure due to not using the drill. An inconvenience on the other hand is that the time for polishing increased from 15-20 min to more than one hour. If there is a need for multiple wafers one could consider to bring the sanding machine back into operation again.

After the mechanical polishing the wafer is removed from the metal block and cleaned with analytical acetone and a lint-free tissue. An ultrasonic bath in analytical acetone and rinsing with analytical isopropanol (IPA) completes the polishing process.

2.1.2 Polyimide layer

The next fabrication step is to coat a layer of polyimide (PI), which serves multiple purposes. Firstly, PI is electrically isolating with a high dielectric strength compared to other materials while being thermally conductive at the same time. Secondly, the layer of PI can be etched easily using RIE to form a freestanding constriction, which is required for the MCBJ technique. Furthermore, polyimide is stable over a large range of temperature including cryogenic temperatures and temperatures well above room temperature.

In order to obtain a smooth layer of PI, which again is necessary for good quality EBL, the layer of polyimide is applied using the method of spin coating. For this purpose the wafer is spun on a rotating plate and PI is spread evenly across the wafer by the centrifugal force.

To start this process one should take the polyimide (Durimide 115A by Fujifilm Elec-

tronic Materials) from the freezing compartment of the fridge in P504, defrosting takes a few minutes. Meanwhile, one can blanket the spin coater with aluminum foil to facilitate the cleaning afterwards and cover the chuck with parafilm including four tiny holes in a diamond shape to ensure good suction of the wafer. The convection oven is preheated to 135 °C and the vacuum oven prepared to load the wafer. After baking the wafer out on a hot plate (100 °C for 1 min) to get rid of remaining water on the surface, the wafer is centered on the spin coater and held in position by a vacuum pump. Once the position of the wafer is balanced and the surface was blown clean with nitrogen, the wafer is covered with approximately half a glass vial with rolled rim of PI. When spreading the polyimide roughly with a needle parallel to the surface it is important to make sure not to have any air bubbles close to the center, as they would move towards the center instead of outwards. **Program 3** will ramp the spinner up to 5000 rotations per minute (rpm) within 3 s and hold that for 90 s after a spread cycle of 300 rpm / 90 s. All changes of the rotation speed here as well as for the following spin coatings are applied in short ramps of 3 s. The spread cycle parameters and the duration for the spin coating of the PI differ from the Fujifilm data sheet, but they have proven to give good results and were therefore unchanged. The temperatures for baking the wafer, on the other hand, have been slightly changed according to the data sheet when comparing to previously used temperatures [4]. After the spin coating, we soft bake the wafer in the convection oven at 135 °C for 5 min and hard bake 400 °C for 30 min in the vacuum oven afterwards. This high temperature is needed to convert the polyamic acid into polyimide. Strictly speaking it should only be called polyimide after the hard baking. As the vacuum oven had some issues that are now solved, I wrote a short manual on how to operate it properly. The manual can be found in figure 4.1 in the appendix. The result of this fabrication step is a polyimide layer with thickness of approximately 2 μm .

2.2 Electron-beam lithography

Even though most of part the structures in our sample design could be written by a laser lithography system as well we only use the electron-beam lithography (EBL) system available in the nano.lab (Zeiss CrossBeam 1540XB). The main reason for this is that this way we only have to perform one single step of lithography and therefore don't have to deal with alignment issues of a subsequent lithography step. Plus, we can have superconducting leads as well.

2.2.1 Sample Design

The sample design was principally adopted from LAURA SOBRAL REY and was previously implemented with Neomicra. A sketch of the sample design taken from her thesis is shown in figure 2.2. As the Neomicra system is no longer supported we switched to Raith, respectively Elphy Plus. We copied the dimensions of the SET structure and adapted the leads for a 18 mm \times 3 mm sample. Therefore we use a small writing field of 100 μm \times 100 μm for the small structures and place it in the center of the sample. This writing field is then embedded in a larger one (1000 μm \times 1000 μm) which is centered as

well. More write fields are used to cover the rest of the leads. To ensure good stitching, the large writing fields overlap by 50 nm per design.

The superconducting island of the SSET is weakly coupled to its environment via two leads and can be controlled with a gate (coming from the right here). This SET is special, as one of the contacts, the MCBJ (coming from the left) can be adjusted *in situ*. The tunnel junction in form of a finger (touching the island from the bottom) is only connected through shadow evaporation and is fixed by the oxidation step. To protect the device from static shocks, a macroscopic shortcut connects the two bias leads and is cut through not until the sample has been loaded to the cryostat.

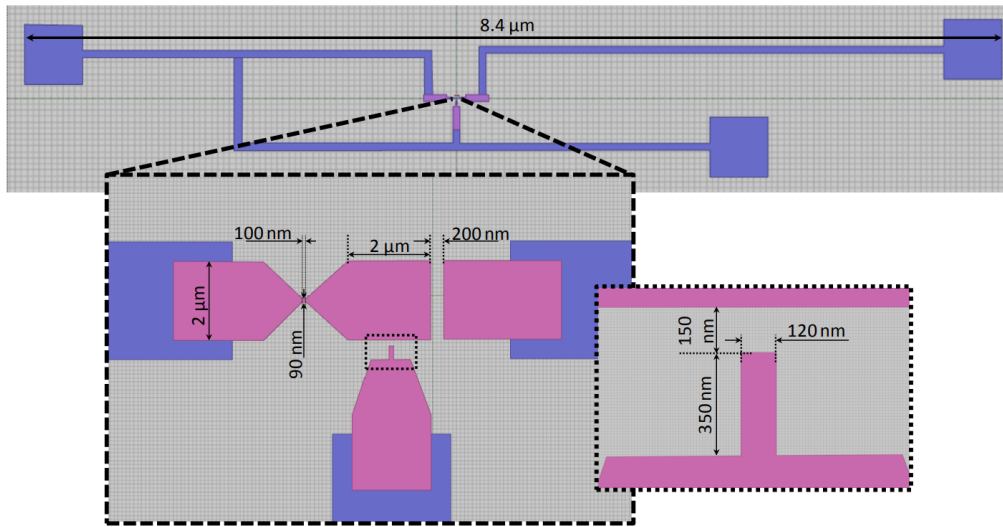


Figure 2.2: Lithography mask for a thin film MCBJ-SET using a small (pink) and large (purple) writing fields. This sketch of the sample design was taken from [5].

2.2.2 Spin coating

In the same way as the polyimide coating two layers of positive electron sensitive resist are applied to the wafer. The reason why we use a two layers of resist is briefly discussed in section 2.2.6. Other than the PI, there is no need to cover the spin coater with aluminum foil as the excess resist can easily be wiped with technical acetone.

The bottom layer of copolymer (MMA(8.5)MAA EL11 by Kayaku Advanced Materials) is designed to have a thickness of approximately 600 nm, while the top layer of PMMA (950 PMMA A4 by Kayaku Advanced Materials) is thinner with a thickness of nearly 200 nm. After preheating the convection oven to 170 °C and preparing the chuck of the spin coater as before, the wafer again is put onto a hot plate (100 °C for 1 min) to get rid of moisture and blown clean with nitrogen, once it was centered on the chuck. Then, about 90 % of the surface is covered with copolymer from the syringe (which is

stored in the fridge as well) and **Program 1** spins the wafer at 2500 rpm for 90 s after a spread cycle at 500 rpm for 5 s. Before coating the second layer, the wafer is placed on the hot plate at 150 °C for 60 s to bake the first layer of copolymer. After covering the wafer with PMMA, **Program 2** will execute the same spread cycle as before and spin up to 5000 rpm for 60 s prior to a baking of the wafer in the convection oven at 170 °C for 30 min. Just like for the polyimide, the temperature and duration for the baking have been updated according to the data sheet, whereas the spin parameters have been unchanged compared to previous works [4].

2.2.3 Wafer cutting

After completing the preparation of the entire wafer with polishing, polyimide and a bi-layer e-beam resist, the wafer can now be cut in to smaller pieces which will then be nanostructured to become individual samples. The sample size that was used in the Scheer II cryostat is 22 mm × 3 mm. Currently we use 18 mm × 3 mm as the sample holder in the BlueFors cryostat is limited to that length. However, we might change to a sample holder accepting 22 mm × 3 mm as well or even 22 mm × 4 mm to create more space and therefore make the process of contacting easier.

The wafer cutter basically cuts the bronze wafer by pressing a sharp metal edge down onto the wafer with a lever. Suitable spacers allow cutting pieces of the desired dimensions. Depending on the condition of the wafer cutter cleaning with IPA is recommended.

2.2.4 Exposure

For lithography one or more samples are loaded on the sample holder (with Faraday cup) and transferred into the CrossBeam. A rough alignment of the samples is sufficient, gluing or clamping of the samples is not necessary when transferring carefully. The workflow for writing samples has been optimized using position lists to automatize some steps and to remind the user to do certain other things. There are two position lists to be worked through, one for alignment, functions rather as a memory aid and saves a few clicks, and one for writing the complete structure where all aperture changes and stage and beam movements are automatized.

First, we choose the proper alignment file according to the sample length we inserted. Next, we make sure that the correct writing field and aperture for the small structures are selected. We start by writing the smallest and also most important structure (constriction for the break junction) right in the center of the sample and continue with a bigger writing field (1000 μm × 1000 μm) at the same position to connect the small structures to the leads and then continue with the writing fields for the leads. As the SEM has been optimized to operate with the 30 μm aperture, we also use this one for the most critical structures for the small writing field (100 μm × 100 μm) in the center of the sample. In addition, we use high current mode and a acceleration voltage of 10 kV even for the small writing field as the limiting parameter for resolution is the resist and not the spot size of the electron-beam. A big advantage of using high current mode is the increased depth of focus, which simplifies focusing the electron beam significantly, as

there is no need for contamination dots. These simplifications as well as different doses have been compared to the previous method of doing the EBL and showed equally good results as before. Best results could be achieved with a dose of $170 \mu\text{C}/\text{cm}^2$ for both the small and larger structures. The area line spacing and area step size for the small writing field are both set to $0.008 \mu\text{m}$ for the small writing field and $0.04 \mu\text{m}$ for the large writing field.

After choosing the correct writing field and aperture and turning the high current mode on, we measure the beam current with the Faraday cup and recalculate the area dwell time accordingly. The last time we measured it was 0.397 nA . In principal one should measure the beam current for all apertures used, but as the value for the large aperture of $120 \mu\text{m}$ changes slowly and the exact dose for the leads is not as important as for the central structure, we only measure the beam current for this aperture once in a while. For reference, the value for of the beam current for the big aperture is around ten times higher than for the small one.

To align the sample by two point angle correction we navigate to the top right corner of it and roughly focus the beam to get an acceptable image. Then, we set the origin of the UV-coordinate system to this position, mark the corner as first reference point for the angle correction and drive to to the point where we expect the top left corner, mark it as second reference point and apply the angle correction. In order not to accidentally expose the sample while moving the stage, the beam is blocked automatically. To check the alignment, we can move back to $(U,V) = (0,0)$. The alignment is precise enough, if the top right corner is within $20 \mu\text{m}$ from the expected position. Next, we move to the middle of the top edge and look for some particle on the sample to focus the electron-beam properly. This means we wobble and correct the astigmatism, then we set the beam shift to zero and adjust the working distance to 5 mm with a precision of 0.001 mm .

Now we can open the position list for writing the actual structures, select all lines and execute it. The machine will therefore start by exposing the small structures in the center of the sample, embed this in a larger writing field in the center as well and then continue to write the rest of the leads. To reduce the time the resist has to be exposed and therefore speed up the lithography we also use high current mode for the large aperture and the big writing field. After couple of minutes the machine will automatically move to the expected position of the top right corner of the next sample and change to the small aperture again. This way we can continue aligning and writing the next sample easily.

2.2.5 Development

After the lithography the sample is developed, which dissolves those parts of resist that have been exposed to the electron-beam. Therefore, the sample is held and moved gently in a glass vial with rolled rim filled with a solution of 1:3 MIBK to IPA for 25 s . To stop the chemical reaction and prevent overdevelopment, the wafer then is transferred into an other glass vial with rolled rim filled with IPA for 1 min and blown dry with nitrogen.

2.2.6 Shadow Evaporation

In nanofabrication one often wants to combine two different materials by a clean contact meaning that no contamination or oxide layer should lie in between the two materials. Therefore, it is necessary to deposit the two layers without breaking the vacuum. In our case we actually want an oxide layer to create a SIN or SIS interface. However, the oxidation has to be done in a very controlled way to tune the oxide barrier to the desired resistance. The method we use to contact two materials under vacuum conditions is the Niemeyer–Dolan technique [6] which is also known as angular evaporation or shadow evaporation. Here, a suspended mask with large undercut is used for evaporation at different angles. This is why we coated our sample with a bi-layer resist in the first place. A sketch of the shadow evaporation is shown in figure 2.3. To achieve a very directional metal deposition the distance between crucible and sample holder is quite large with ~ 60 cm. That is also the reason why we use evaporation instead of sputtering.

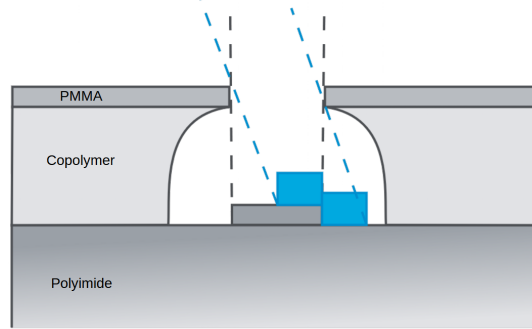


Figure 2.3: Visualization of the shadow evaporation, where two layers of material are deposited on top of each other at two different angles. Note that the top layer has to be thicker in order to have a continuous film across the edge of the bottom layer.

The evaporator we use for our shadow evaporation is usually referred to as the „old evaporator“ and is located in P502. Special features of this machine are the possibility to rotate the sample holder (which is of course necessary for shadow evaporation) and a thermal evaporation crucible alongside the electron-beam evaporator.

For best results it is recommended to do the development of the samples directly before loading for evaporation. Furthermore, the sample holder should be cleaned with IPA and heated to 100°C to get rid of moisture. Ideally, the loading of the samples to the load lock is done in the evening, so that the chamber can be pumped during the night before transferring to the evaporation chamber. This has proven to enhance the quality of the aluminum films. To do the shadow evaporation correctly, one has to make sure, that the finger contacting the island from the bottom (see figure 2.2) is oriented towards the transfer hook of the sample holder.

We start by evaporating 6N aluminum from the thermal evaporator. As this crucible is not located directly underneath the sample holder unlike the electron-beam evaporator, we tilt the sample holder by 4° to effectively evaporate the first layer of metal perpendicularly. Experience has shown, that there is no problem to keep the crucible

filled with aluminum and there is no need to empty the crucible every time as done in the past. On the contrary, always having enough metal available and being able to evaporate couple of nanometers on to the shutter first also helps the quality of the aluminum film. Especially the end phase, when only few aluminum was left in the crucible, resulted in uncontrollable rates and therefore caused problems to reach a certain film thickness.

The thermal evaporator can be ramped up automatically by a motor turning the wheel and should only be stopped around 57 A when the vessel glows orange to wait for freshly loaded aluminum pellets to melt. Following [7] we initially aimed for an evaporation rate of 6 Å/s, however we had problems to get stable rates. In the end we settled for 94 A/~ 6.7 V as this gave stable rates of approximately 2.5 Å/s within a few minutes. We evaporate 60 nm or more, as we observed the freestanding constriction to bend for thinner a film thickness.

After ramping the current down again, the samples are transferred back to the load lock for oxidation. For that purpose, the gate to the pumping line is closed and all valves of the oxygen line are opened beginning with the one at the wall and ending with carefully opening and closing the needle valve to reach a pressure of 3 mbar. When a timer of 3 min has run down, the gate is reopened again to pump the oxygen out of the load lock and the oxygen line for which the needle valve is opened again. Once the pressure is low enough the samples can be transferred back into the main chamber. Due to the partial pressure of oxygen the pressure required for transferring is 1×10^{-6} mbar instead of 5×10^{-6} mbar.

To evaporate a shadow of the structure on top of itself and therefore contact the island with a finger from above we set an angle of 34° . The shadow of the break junction is unwanted and was thus designed in a way, that it is at least partly evaporated to the wall of the resist. The deposition on the mask additionally narrows the constriction, which causes the shadow of the break junction to rip off when breaking for the first time. All the other shadow duplicates of the structure do not disturb.

When evaporating copper one has to slowly warm up the metal with the electron-beam, 3 min per 3 mA is a good guide value. For 33 mA we get a relatively stable rate of approximately 2.5 Å/s. If the first layer of aluminum had a thickness of 60 nm, the second layer has to be somewhat thicker in order to be continuous along the edge of the first layer. Taking into account that due to the angle α of the sample holder the effective thickness is reduced by a factor of $1/\cos(\alpha)$. For the 34° we have applied, this means we should evaporate approximately 105 nm to ensure good continuity across the edge of the first layer. Analogous to the speed ramping up the current, it should also be reduced to let the copper cool down gently. When refilling copper nuggets it is important to know, that they have to be pre-etched with diluted nitric acid ($1 \text{ HNO}_3 + 5 \text{ H}_2\text{O}$).

Checking the evaporated samples with the profilometer we noticed a significant discrepancy between the film thickness we evaporated and the one we measured. The profilometer always measured a thickness higher than expected with a factor that varies between two and four. We first suspected a problem at the evaporator, but after double checking every parameter we now think the profilometer cannot be trusted. A possible explanation therefore is that the polyimide is just too soft and thus falsifies the measurement. Viewing the sample from an angle with the SEM supports this finding, here

the film thickness we read from the evaporator looks reasonable.

While evaporating aluminum onto the samples, there were several issues. For one, some of the aluminum films were very grainy as shown in figure 2.4. This problem could be solved by paying more attention to good vacuum conditions for the evaporation, which means pumping the load lock to a pressure significantly lower than the one required for transferring to the main chamber. The easiest way to ensure good vacuum before transfer is to load the samples in the evening before evaporation, so that the load lock can be pumped properly during the night. We also tested to evaporate while cooling the sample holder with liquid nitrogen (reaching temperatures down to -150°C), which has not shown any significant effect.

A second problem, we noticed in the fabrication process is that the precision of the evaporated structures depends strongly on the post-development time. This is unexpected, as the bath in IPA after development should in principle only stop the development process. An issue connected to this is that the resist masks were collapsing as shown in figure 2.4a. A consequence of which is that the shadow i.e. the second layer of evaporation is distorted as the shape of the collapsed mask changes with the evaporation angle (figure 2.4b). We were able to solve this problem by buying new resists, and coating it as specified in the data sheet. Both, the PMMA and the copolymer have been expired for several years. Most likely a combination of the expired resists and the too low baking temperature of the copolymer have caused bubbles in the resist as shown in figure 2.5a and thus let the masks collapse.

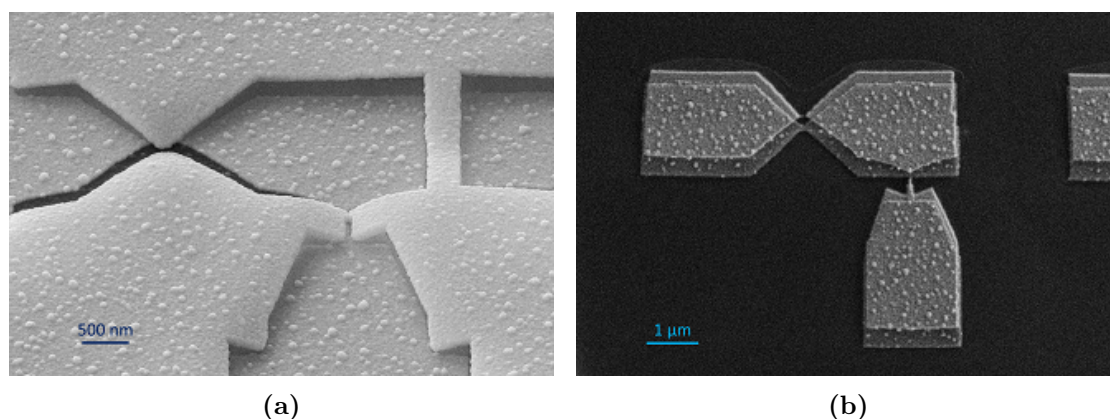


Figure 2.4: (a) Collapsing masks caused problems during evaporation. The problem could be solved by buying new resists. (b) Test structure showing an impure aluminum film and a distorted shadow which was caused by collapsing masks.

2.2.7 Lift-off

To lift-off the resist and the unwanted metal on top of the resist, a sample is placed in a glass vial with rolled rim filled with acetone and put on a hot plate (60°C for 1 h or longer), which accelerates the lift-off process. The glass vial should be capped and

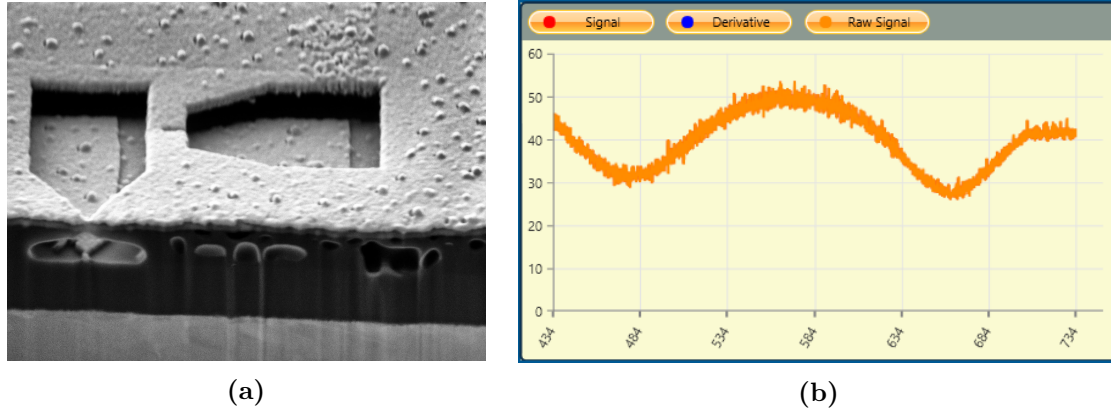


Figure 2.5: (a) FIB-cut of a sample before lift-off showing the different layers of bronze, polyimide, copolymer with bubbles, PMMA and evaporated metal on top. (b) Graphical visualization of the laser endpoint from the reactive-ion etching machine showing the reflected intensity of the laser interferometer in percent in the course of time to track the etching process.

labeled with your name and the chemical inside. In order to help the lift-off process one can use a pipette to flush residuals of resist/metal. To complete the lift-off, the sample is first rinsed with IPA and blown dry with nitrogen.

2.3 Reactive-ion etching

The final step of sample fabrication is the etching in order to form a freestanding constriction which is necessary a controlled breaking in of the constriction. For etching we use the Oxford Instruments PlasmaPro 80 ICP RIE available in the nano.lab with etching parameters tuned to isotropic etching which enables undercutting the metallic structures to ultimately undercut the constriction of the break junction completely. Other than wet etching, the (dry) reactive-ion etching process can be controlled very precisely, it is especially easy to terminate it. As this machine is new, the recipe from the old RIE had to be adapted according to our needs. We use oxygen plasma with the maximum flow rate of 50 sccm, maximum pressure of 250 mTorr, maximum ICP RF power of 400 W and low acceleration voltage of 1 V. It has proven to be beneficial to allow higher powers and higher tolerances as well as to start with less pressure in order to get a stable plasma reliably. A more detailed view of the process parameters is presented in figure 2.6, which is a screenshot showing our program „MCBJ SSET Unteraetzen v5 @ 100 °C“ taken from the control software. The most influential parameter to the etching process turned out to be the temperature of the table heater. An increased temperature really helps the isotropic etching, but also makes the etching very fast (~ 20 s at 200°C), which has a negative effect on reproducibility. We now have set the temperature to 100°C which allows good control over the process that now takes approximately 15 min and can be monitored live with a laser interferometer. The laser has a wavelength

of $\lambda = 660 - 680 \text{ nm}$ and can detect a change of thickness of the polyimide layer. As the light is reflected on the top interface of the PI layer and on the bottom interface/bronze substrate, the optical path difference is

$$\Delta = 2 \cdot d_{\text{PI}} \cdot n_{\text{PI}} = \lambda \cdot m \quad (2.2)$$

with the thickness d_{PI} of the polyimide layer and the refractive index $n_{\text{PI}} = 1.81$ [8]. While the thickness of the PI layer decreases during etching, the reflected intensity varies roughly sinusoidally as constructive and destructive interference alternate. This can be read graphically from the laser endpoint as shown in figure 2.5b. One period (assuming 670 nm) equals a thickness of 185 nm being removed. We therefore stop the etching process manually after almost three periods which results in removing a total of about 550 nm to certainly create a proper freestanding constriction.

	Alarm Level	Stabilisation	ignite	Process	New Step
Table Heater	$\pm 2.5^\circ\text{C}$	100 °C	No Change	No Change	No Change
Table RF	$\pm 5 \text{ w}$	No Change	Power Demand 20.0 w Max Reflected Power 5.0 w Tol Time 7.00 s Min DC Bias 1.000 v	Power Demand 3.0 w Max Reflected Power 2.0 w Tol Time 7.00 s Min DC Bias 1.000 v	Off
ICP AMU	$\pm 5 \%$	Auto Park C1 0 % C2 0 %	Auto Park C1 41.0 % C2 23.0 %	No Change	No Change
Table AMU	$\pm 5 \%$	Auto Park C1 0 % C2 0 %	Auto Park C1 64.0 % C2 39.0 %	No Change	No Change
He Flow Controller	$\pm 5 \text{ Torr}$	Off	No Change	No Change	No Change
APC	$\pm 5 \text{ mTorr}$	Pressure 100.0 mTorr	Pressure 100.0 mTorr to 250 mTorr Ramp Time 5.00 s	Pressure 250 mTorr	Pressure 250 mTorr to 10.00 mTorr Ramp Time 8.00 s
O2	$\pm 5 \text{ sccm}$	50.0 sccm	No Change	No Change	Off
ICP RF	$\pm 50 \text{ w}$	No Change	Power Demand 400 w Max Reflected Power 100 w Tol Time 10.00 s	No Change	Off
Laser Endpoint		Off	Off	Monitoring Endpoint using None Threshold Rising Threshold value 5 %	Off

Figure 2.6: Screenshot from the control software of the etching machine showing all relevant parameters to the etching process. The maximum duration for etching is set to 30 min, but the process is stopped manually as soon as the desired thickness of polyimide has been removed, which is the case after approximately 15 min.

2.4 Contacting the sample

Contacting a sample is a tricky undertaking, but can be learned with a little practice. The following tips might help to avoid some mistakes. The first step is to prepare the cables by removing the insulation of the cable and soldering it to the pin connector. We

use 60 μm copper cables and a scalpel or dismantling the cables. The easiest way to prepare a twisted pair cable for the bias line is to solder the to ends of a piece of cable and twist it before cutting it in the middle. A good cable including the bond to the pin connector should have a resistance no more than 1 Ω .

As soon as the cables are ready, the sample can be fixed on the contacting plate with adhesive tape on both sides. Now one can position the cables on the contact pads and hold them in position with some putty. It is worth taking some time to position them correctly so that the ends of the cables are lying plainly on the contact pads. A high table/low chair and good lighting are helpful as well to see the tiny contact pads better and have the forearm rest on the table. The cable is connected to the contact pad on the sample through a small droplet of conducting silver applied with a toothpick that can be sharpened with a scalpel according to personal preferences. Best results could be reached when taking the conducting silver from the cap of the flask that has been turned upside down before and making several test dots on the adhesive tape before really contacting the sample. After a couple of minutes the contacts should be protected to strain by two-part epoxy resin adhesive (UHU Sofortfest). Again, test dots on the adhesive tape are recommended, as the resin tends to wet the surface right after mixing. Figure 2.7a shows what contacting a sample looks like. Even though there were some test dots made with the epoxy resin, the gluing spots got quite large due to the wetting effect.

The final result of the fabrication should look something like in figure 2.7b, which shows an SEM image at an angle of 70° of a etched sample. Checking the electrical conductivity of the sample (with a low current in order not to damage it) confirms that it has been contacted correctly.

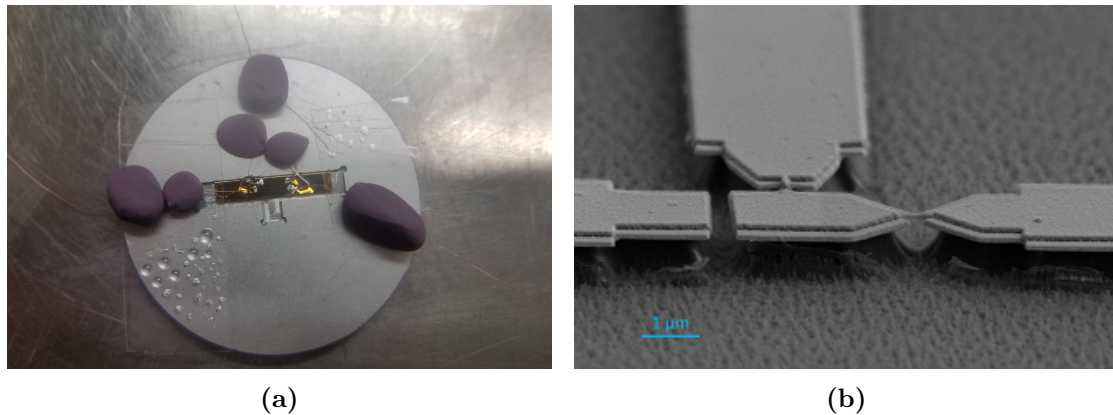


Figure 2.7: (a) Contacting a sample with 60 μm copper cables and conducting silver while held in place by adhesive tape and putty. Making test dots on the tape can help to control the dot size, but needs some practice. (b) SEM image at an angle of 70° showing a sample after the final fabrication step of etching.

3 Conclusion and Outlook

With this work we could reproduce the sample fabrication of earlier SSN-SETs while using a new lithography system and a new reactive-ion etching machine. We faced and solved many problems on the way such as collapsing masks or poor aluminum quality. Furthermore, we could improve some of the fabrication steps as for example the new way of polishing our wafers or tracking the etching with a laser interferometer.

Of course some things may change over time such as the parameters of the electron-beam lithography machine respectively the scanning electron microscope, but this report describes how to reliably fabricate MCBJ-SETs.

The next step in the research project investigating these SETs was to make them all superconducting again and to expose them to microwave irradiation. Beside an antenna in proximity to the sample a stripline or coplanar waveguide is commonly used to couple microwaves to thin film structure on a chip. This will be discussed in my master thesis. In order to simplify the system we will do without the oxide barrier to effectively study a simple aluminum break junction in the known geometry of the SET.

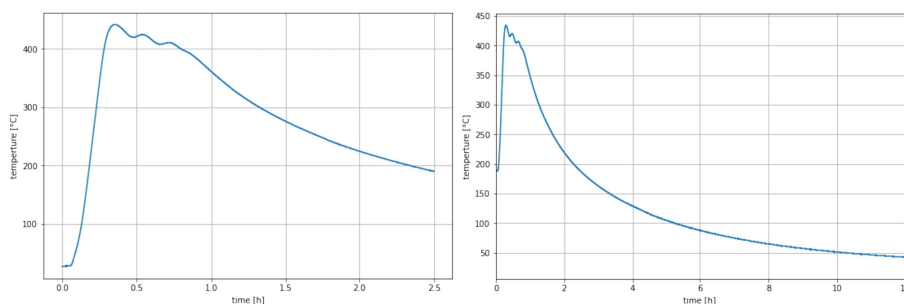
4 Appendix

User Manual – Vacuum Oven

Unfortunately, the controller does not remember the time it should stay at a certain temperature. ☹️ Proceed as the following: (values for PI hard bake)

1. Load wafer/sample to be baked, make sure valves are closed & knob for heater is off.
2. Start pumping, then tighten screws to secure lid.
3. When digital manometer is out of range ($p < 1$ mbar) start the turbomolecular pump.
You can now turn on the analog manometer to check the pressure & turn it off afterwards.
4. Check, if temperature setpoint = SP1 = **400 °C** is set to the correct value.
Use curved arrow to navigate the menu. (A hidden/second layer with more options is available when holding the MENU button and entering code: 2 = UP, UP)
5. Increase T.REMN (remaining time) to **0:30 min** and turn the knob for heater to the marked position. *Do not touch the electronics!*
6. Press UP & DOWN at the same time to start the heater. (*WRK.OP* should now be 100%)
7. Let it cool down over night while still pumping.
8. Vent carefully by opening the valve while closing the tube with your thumb.
9. Note date, name, material/recipe + parameters & pressure before opening the chamber.
Ex.: 25.01.23, Olli & Patrick, PI/bronze overnight, 30 min, 400 °C, $p_{end} = 5 \cdot 10^{-6}$ mbar.

Following these instructions, your temperature curve should look something like that:



Ancient PID-parameters, that were “good enough”: PB = 32.1, TI = 1526, TD = 254

Figure 4.1: Helpful instructions to operate the vacuum oven for baking polyimide.

Table 4.1: Summary of the fabrication of a MCBJ-SET adapted from [4].

Fabrication step	Materials, machines, parameter and procedure
Wafer preparation Polishing Cleaning	Remove plastic foil, glue bronze wafer onto polishing block Use wet sanding paper with increasing grit starting with 600 Acetone, IPA and ultrasonic bath
Preparations Drying Sacrificial layer Spinning Soft baking Hard baking	Defrost polyimide, protect spin coater with aluminum foil, cover chuck with parafilm & holes, prepare vacuum oven, preheat convection oven to 135 °C Hot plate at 100 °C for 1 min, blow with N ₂ Cover wafer with Durimide 115A, mind air bubbles Spread at 300 rpm for 30 s, spin at 5000 rpm for 90 s, ramps for 3 s (Program 3) Bake wafer in convection oven at 135 °C for 5 min 400 °C for 30 min
Drying Spacing layer Spinning Baking	Hot plate at 100 °C for 1 min, blow with N ₂ Coat wafer about 90 % with copolymer MMA(8.5)MAA EL11 Spread at 500 rpm for 5 s, spin at 2500 rpm for 90 s, ramps for 3 s (Program 1) Bake wafer on hot plate at 150 °C for 60 s
Electronic resist Spinning Baking	Coat wafer about 90 % with 950 PMMA A4 Spread at 500 rpm for 5 s, spin at 5000 rpm for 60 s, ramps for 3 s (Program 2) Bake wafer in convection oven at 170 °C for 30 min
Wafer cutting Exposure Development	Clean wafer cutter, use spacers to cut 18 mm × 3 mm Writing sample with high current mode, 10 kV, WD of 5 mm, small and big writing fields and an area dose of 170 $\mu\text{C}/\text{cm}^2$ Gently move sample a solution of 1:3 MIBK to IPA for 25 s Stop development with 1 min in IPA and blow dry with N ₂
Loading samples Shadow evaporation Lift-off	Clean and heat sample holder, load in the evening before Orient the lead contacting the island with a finger from the bottom towards the hook of the sample holder First layer: 60 nm aluminum at 4° Oxidation : In load lock oxidize with 3 mbar for 3 min Second layer: 105 nm copper at 34° Put samples in closed vial and on hot plate 60 °C for 1 h Use pipette to flush residues of resits/metal away.
Reactive-ion etching	Isotropic etching with O ₂ plasma by enhancing the chemical part through low acceleration voltage, high ICP power, high O ₂ flow and pressure and elevated temperature to remove ~ 500 nm Program (MCBJ SSET Unteraetzen v5 @ 100 °C)
Contacting	Connect contact pads to 60 μm Cu cables with conducting silver and fix them with UHU Sofortfest using a toothpick

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