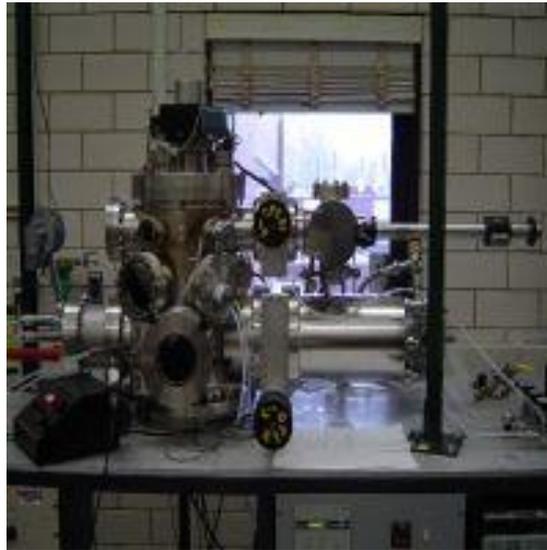


## A GUIDE TO SPUTTERING

In 1852, W R Grove presented a paper which provided the first recorded observation of sputtering, the ejection of atoms of a material resulting from its bombardment by positive ions [1]. Today, sputtering has grown to be the most widely used vacuum deposition technique, with applications in industry and experimental research. In industry, sputtering is used to manufacture magnetic storage disks in personal computers, and for producing optical coatings [2]. Our lab is equipped with a homebuilt Ultra High Vacuum (UHV) System which consists of two chambers equipped with a total of five sputtering guns, (two rf- and three dc-), two ion-mills, and an electron beam evaporator (Figure 1).



*Figure 1: Our homebuilt UHV System.*

One of the chambers is dedicated to making Nb trilayers for SQUIDs, whilst the other is a multipurpose chamber, currently used for the fabrication of S-F-S Josephson junctions [3]. The purpose of this report is to provide the user of the UHV System with guidelines on how to sputter.

The main steps in sputtering are :

- A) Substrate Preparation
- B) System Preparation
- C) Substrate Loading
- D) Sample Positioning
- E) Ion Milling
- F) Film Deposition
- G) Sample Unloading
- H) Final Checks
- I) Sample Treatment

### **A) Substrate Preparation**

The substrate used for our purpose is silicon oxide, which was pre-coated with photoresist in the cleanroom. The coating of photoresist is to minimize impurities adhering to the surface of the substrate when it is brought out of the cleanroom into the sputtering lab.

- 1) Fill one beaker with acetone
- 2) Fill another beaker with isopropyl alcohol (2-propanol)
- 3) Using a pair of handlers, transfer the substrate from a dish first into the beaker with acetone
- 4) Place this beaker into the Ultrasonix 57X machine and hit “Start”
- 5) After 1-2 minutes, hit “Stop” on the Ultrasonix 57X machine
- 6) Whilst spraying acetone on the substrate, transfer the substrate quickly into the beaker with isopropyl alcohol
- 7) After rinsing with isopropyl alcohol, dry the film with a dryer, with the film tilted downwards

The preceding steps removed the layer of photoresist coated on the substrate, and chemically cleaned it.

- 8) Take a sample holder and apply silver grease on its surface
- 9) Using 2 q-tips, press the sample down
- 10) Place the prepared sample holder under a beaker near the UHV system

### **B) System Preparation**

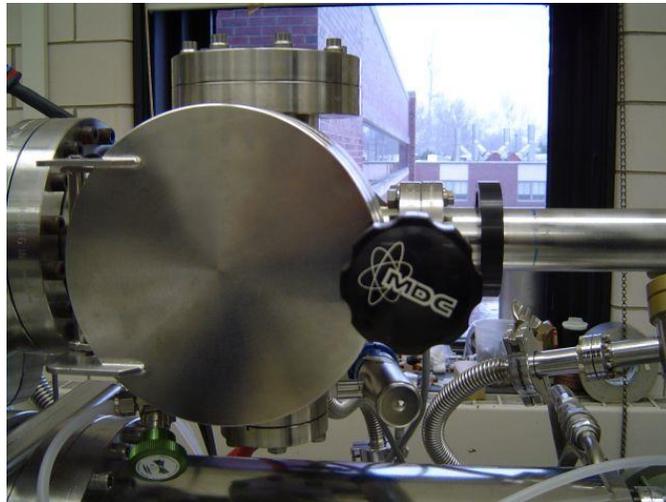
The objective of the following steps is to vent (bring to atmospheric pressure) the loadlock chamber.

- 1) Ensure that the loadlock chamber gate valve is closed, and the gate valve for the cryopump is open. The gate valve for the cryopump is open all the way at this stage for the cryopump to maintain a high vacuum in the main chamber.
- 2) Shut off the turbo pump by pressing on button A of the TCP 21 electronics (Fig 2).



*Figure 2: Electronics of the UHV system*

- 3) Close the butterfly valve (located behind the entry point of the loadlock chamber). This isolates the roughing pump from the system.



*Figure 3: Lock of loadlock chamber and nitrogen valve*

- 4) Loosen the lock of the loadlock chamber (Figure 3), and rotate the lock to the right of the sample entry point.
- 5) Open the nitrogen valve (the green tap beneath the loadlock chamber, Figure 3) Do this slowly so that there is no sudden buildup of pressure within the loadlock chamber.
- 6) Check that the reading A of the 307 Vacuum Gauge Controller reads  $7.7 \times 10^2$  (atmospheric pressure) before opening the loadlock chamber.

### C) Substrate Loading

Having vent the sample chamber, we are now ready to load the sample.

- 1) Screw the sample holder onto the transfer rod. Ensure that the nuts on screw from the transfer rod are not falling off.
- 2) Turn the sample face down using the magnet (Figure 4) on the external part of the transfer rod.

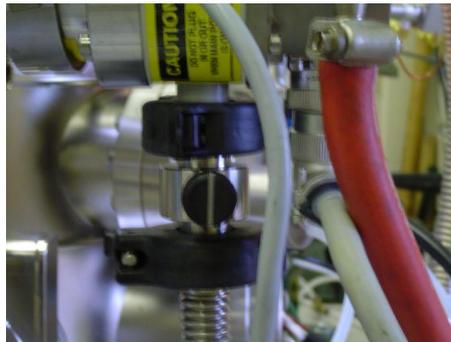


*Figure 4: Magnet used to adjust sample position*

- 3) Close the loadlock chamber and lock it.
- 4) Turn off the nitrogen valve

Before one transfers the sample from the loadlock chamber into the main chamber, a low pressure is desired. The change from atmospheric pressure to the low pressure in the sputtering chamber is effected in two steps (5 & 6).

- 5) First, the butterfly valve is opened as shown in Figure 5.



*Figure 5: An opened butterfly valve*

- 6) When the second reading on the 307 Vacuum Gauge Controller reads  $5 \times 10^{-2}$  or lesser, push button ON of TCP 121 to turn the turbo pump on.
- 7) Once the TCP 121 bar is fully lit (luminous green), the turbo pump is up to speed, and the loadlock chamber valve can be opened to allow equilibration of pressures (At this point, the first reading on the 307 Vacuum Gauge Controller will increase initially, but should drop eventually)

#### **D) Sample Positioning**

This is probably the most technically challenging task; the user is required to align the transfer rod and the two bolts, slotting the sample holder (See Figure 6) into one of the forked ends of the internal support. The user should be cautious not to let the sample run into the shutter.



*Figure 6: A sample holder*

#### **E) Ion Milling**

The purpose of ion milling is to clean the surface of substrates. After fitting the sample holder into the forked end, the sample would have been in a position to be ion-milled, since the ion-mill is located directly under the point where the sample is transferred in from the loadlock chamber.

- 1) The ion-mill compartment is linked to tap 3 (Figure 7), which is in turn connected to a cylinder of Ar gas.
- 2) Open tap 3

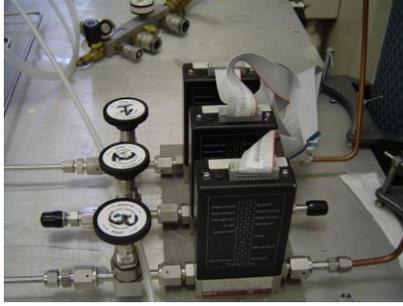


Figure 7: Taps linked to gas cylinders

- 3) On the Model 2478 4-Channel, flip “Channel 3” up (Figure 8)
- 4) Wait until the pressure on the ION gauge reads  $2-3 \times 10^{-4}$ .

Steps 1 to 4 can be understood as the initiation of the flow of Ar gas. The ion-mill should be ready to use. Procedures for further adjustments of the ion-mill can be found in the ion-mill manual.



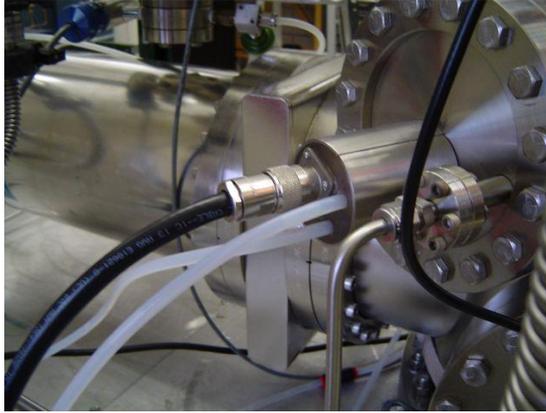
Figure 8: The ID 2500, MKS Flow Controller Model 2478, and water timer

- 5) Turn the power on ID 2500 on to ionize the Ar gas
- 6) Get a timer ready
- 7) Turn source on, wait for discharge to light, then turn beam on. These controls are found on the ID 2500.
- 8) Ion mill for 40 s
- 9) First, turn the beam off, then turn the source off, and then turn the power off
- 10) Wait for approximately 10 minutes to allow the sample to cool

## F) Film Deposition

Before coating the substrate with a film, the user has to go through the process of pre-sputtering to get rid of impurities trapped in the gun.

- 1) Check that the high voltage power cable (Figure 9) is attached to the relevant gun



*Figure 9: High voltage power cable*

- 2) Turn the water timer (Figure 8) on. This runs out in 60 minutes so that we do not waste the deionized water. If the user intends to sputter for a long time (over an hour), ensure that the water timer does not run out. This is an important step since we do not want the system to overheat
- 3) Turn the leftmost switch on Model 2478 4-Channel up. We initiate the Ar flow through channel 1. For that purpose we open the tap labelled 1, and then open the channel 1 on the 2478 flow controller
- 4) Close the cryopump gate valve until the pressure reaches  $10^{-2}$  on the baratron gauge (gauge B on Philips 307 Gauge Controller)
- 5) On the MDX-1K Channel Box, hit Power On, hit the “SETPT” button, and rotate the knob to adjust for the appropriate setpoint for power. For instance, the setpoint for Niobium sputtering is 153 W, and that for CuNi is 12 or 30 W.
- 6) Time this process (pre-sputtering) for approximately four minutes
- 7) At this point, move the substrate from above the ion-mill to above the Niobium gun. This can be done by manipulating the “jog” button of the remote control.
- 8) Leave the system to sputter for as long as required. Take note though, that sputtering for too long a time causes the sample to overheat. For sputtering over extended time periods, be sure to move the sample away from the gun to cool from time to time. Take note that the gun should not be turned off, or else one might have to presputter again.

In observing various plasmas through the inlets, a brighter color for the plasma is seen if higher power is used. A different colored plasma results from the use of different materials as plasma material.

### **G) Sample Unloading**

Before unloading the sample, the user should perform the following steps:

- 1) Turn the MDX-1K off (This is to stop the sputtering). To do that, first turn the plasma off, and then turn the power off.
- 2) Turn the Model 2478 4-Channel off (To stop the flow of gas). We do not turn the whole flow controller off. We only shut off channel 1.
- 3) Turn tap I off
- 4) Close the gate valve for the cryopump
- 5) Turn the water supply off

The sample can now be withdrawn back to its original position.

We will now wish to open the vacuum pump so we could build up the pressure within the sample chamber to atmospheric pressure

- 1) Remove the sample from the sample holder with the 2 q-tips
- 2) Remove the silver grease from the base of the sample with isopropyl-alcohol, then acetone.
- 3) Do likewise for the sample holder
- 4) Store the sample

References:

- [1] <http://physics.iop.org/IOP/Confs/Spu/>
- [2] <http://www.gencoa.com/tech/future.html>
- [3] <http://groups.mrl.uiuc.edu/dvh/sputtering.html>

Acknowledgements:

The author gratefully acknowledges the demonstrations and instructions on the use of the UHV system by S. Frolov and M. Stoutimore.