

The Rezanator, Jr.

The Sputtering system for

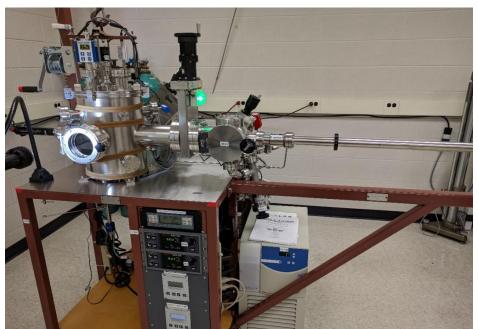
The Advanced Undergraduate Laboratory

Michigan State University
Department of Physics and Astronomy

Quick Operation Manual

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Overview of DC-Magnetron Sputtering System:

Sputtering is a Physical Vapor Deposition vacuum process used to deposit very thin films onto a substrate for a wide variety of commercial and scientific purposes. Sputtering occurs when an ionized gas molecule is used to displace atoms of a specific material. These atoms then bond at the atomic level to a substrate and create a thin film. Several types of sputtering processes exist, including: ion beam, diode, and magnetron sputtering.

In a magnetron sputtering application (such as Rezanator), the high voltage is delivered across a low pressure gas (usually Argon) in order to create high-energy plasma. This plasma emits a colorful halo of light often referred to as a "glow discharge" and consists of electrons and gas ions.

These energized plasma ions strike a target composed of the desired coating material. The force causes neutral atoms to eject from the target material and bond with those of the surface of substrate.

Because sputtering takes place in a high-energy environment, it creates a virtually unbreakable bond between the film and its substrate at the atomic level, creating one of the thinnest, most uniform, and most cost-effective films possible.

To enable ignition of the plasma, the gas pressure is typically maintained in the range of 1-20 x10⁻³ Torr which also depend to the type of sputtering source and nature of target.

DC-sputtering such as the Rezantor is limited to conducting materials like metals and doped semiconductors. The reason is that bombardment with positive ions would quickly charge up the surface of an insulating target material and cause the ion current to die off. Instead, for insulating materials, a radio frequency AC-voltage is applied to the target to prevent the charge buildup associated with DC-magnetron sputtering. This technique is called RF-magnetron sputtering.

This system will be used for depositing Superconducting Niobium (Nb) and Normal Metal Aluminum (Al) films onto a $\frac{1}{2}$ " x $\frac{1}{2}$ " substrate material such as Silicon (Si). First we will make Superconducting Niobium (Nb) and will measure the superconducting transition temperature (Tc). Then, we will make the insulating films such as Aluminum Oxide (Al₂O₃) through direct oxidization using ArO₂ gas. With this sputtering system students will be able to make SIS and SIN junctions. S: superconductor; I: insulating; N: normal materials



Identify Valves and Learn About Their Functions-part 1 What happen if you forget to Open or Close it when it needed



What are the function of Gate Valves?

Gate valve is a vertical disc with an "O" ring that seals and separate one chamber or area from its neighbor.

Gate vale A is largest in the Rezanator system that is backed up with a mechanical pump and maintain the low pressure in the main chamber.

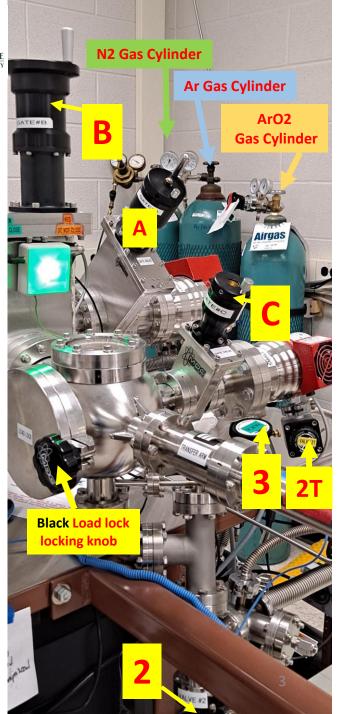
When we insert Ar gas for sputtering, we close it almost 2/3 of the way to maintain the partial pressure of about 3-6x 10^{-3} Torr of Ar gas.

If you do not close it 2/3ed, sputtering guns will have hard time to produce plasma and Nb power supply will blink and Gun won't start. If you close it too much, thedeposition rate goes down due to scattering.

Gate valve B separate the main chamber from the load lock and will stay closed during Loading, Unloading and oxidizing the Al to become Al₂O₃ (insulator). It will stay open during sputtering. Danger: You have to make sure the sample transition rod is in the load lock all the way, otherwise as you close the gate it will bend and damage the transition sample rod.

If you forget to close it during Loading, Unloading and oxidizing, the air, or N2, or ArO2 exsit in the load lock area will rush into the main chamber and it will damage the large turbo pump.

<u>Gate valve C</u> separate the small turbo pump from load lock. We use it only if we need fast pumping of the load lock. It will <u>stay closed during Loading</u>, <u>Unloading and oxidizing</u> the Al. <u>If you forget to close it during Loading</u>, <u>Unloading and oxidizing</u>, <u>the air</u>, <u>or N2</u>, <u>or ArO2 will rush into the small turbo pump and blade will shatter.</u>





Identify Valves and Learn About Their Functions-part 2 What happen if you forget to Open or Close it when it needed



Function of other important valves:

<u>Valve 2</u>: Use for direct pumping of the load lock from the mechanical pump (roughing pump) during loading and during pumping out the ArO₂ after oxidization.

All other valves, 2T, gate valve C, gate valve B and valve 3 must be kept closed during pumping.

If you forget to close:

<u>2T and /or Gate valve C</u>: Small turbo pump will shut down and need to be started again by turning oF and on from back of the unit.

Valve 3: If stay open, N2 gas will keep coming into the load lock and you can not achieve low pressure.

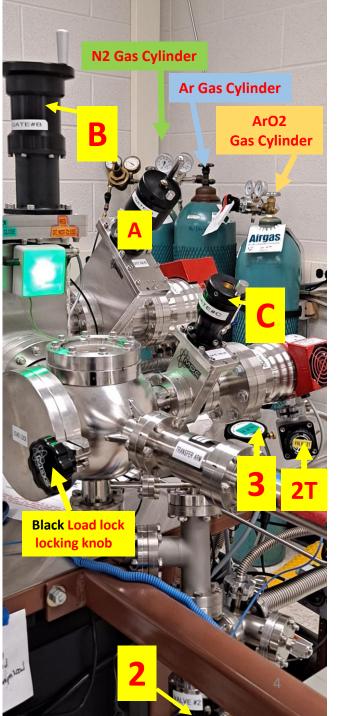
Valve 3: only for inserting N_2 or ArO_2 into the load lock.

All other valves: T2, gate valve C, gate valve B and valve 2 must be closed.

If you forget to close it: similar symptom as valve 2 disfunction.

<u>Valve 2T</u>: is located at the end of the small turbo pump and connected to the smaller roughing pump and Opens only for faster pumping of the load lock along with the Gate valve C.

<u>If you forget to close it during Loading, Unloading and oxidizing, the air, or N2, or ArO2 will rush to the turbo pump and will shut down and possible damage to the blade.</u>





Identify Valves and Their Functions-part 3



Ar gas Flowmeter

Ar gas valve: is the 3ed and final valve for controlling the flow of Ar gas. It connect to the Flowmeter which is on the top of frame to monitor and control the amount gas that goes into the chamber.

If you forget to close it, there will be leak into the chamber and you will not be able to pump down the system to low 10⁻⁷. Also we run out of Ar gas slowly!!

Dry Pump: Is a pumping instrument that does not use oil for creating vacuum; however, needs short term maintenance and shorter lifetime than a mechanical (roughing) pump. There are one unit for this system; for direct pumping of the main chamber during maintenance and can pump down to $\sim 10^{-3}$ Torr.

Roughing Pump: Roughing pump is used for continue pumping of the main chamber with assistant of the turbo pump for reaching to higher vacuum (lower pressure) usually down to low 10⁻⁸ Torr.

Caution must be taken not to use it directly on the main chamber without the turbo pump which will cause oil vapor back-flow into the chamber and create a significant contamination (disaster!!)

ArO₂ gas valve: is an "ON"-"OFF" valve that controls the flow of Ar(80%) O₂(20%) gas.

It is used only for oxidization of Al during the growth of NIS or SIS junctions and goes only into the Load lock.

If you forget to close it, there might be leak along the tubing and it gets empty within a few days.

Nitrogen (N_2) gas valve: is an "ON"-"OFF" valve that controls the flow of N_2 gas. It is used only for purging the Load lock during loading and unloading the sample. Stay on OFF position any other times

Ar gas valve

Dry pump and roughing pump

> ArO₂ gas Valve





N₂ gas Valve

What are the Gages:

- 1-IG: Ion Gage, Located on the top of main chamber and capable of measuring the pressure inside of the main chamber between 10⁻³ and 10⁻¹¹ Torr.
- 2-CG1: Convectron Gage 1 located on the side of the main chamber; capable of measuring the pressure from atmospheric pressure of 760 Torr down to low 10^{-3} torr. However, you will see it shows 0, because the main chamber pressure is always lower than 10^{-3} Torr.
- 2-CG2: Convectron Gage 2, located on side of the load lock and has the same function as CG1 and it should be . 10⁻³ Torr, except when you venting the system or during oxidization.



Why we use 3 different gases?

- 1) N₂ (Nitrogen gas). It is a high purity gas (99.999% pure) and we use it only during load or unload the sample through valve #3. It is cheaper than other gases.
- 2) Ar (Argon gas). It is a high purity gas (99.999% pure) and we use it only during sputtering to produce Ar plasma by removing an electron from orbit to produce Ar ions.

Please note that the typical partial pressure of Ar gas for this system is about 3-6 x 10^{-3} Torr. If it is lower, it would be hard to create plasma and if it is higher such as $2-4 \times 10^{-2}$ Torr, the deposition rate would damp out by scattering between the Nb or Al atoms and other Ar moleculs or ions around the guns.

3) ArO_2 (Argon-Oxygen). It is mixture of 80% Ar and 20% oxygen. We use it only in load lock (with all gate vales and regular vales closed) for oxidization of interlayer aluminum (circular part) to become Aluminum Oxide (Al_2O_3). It must be evacuated from the load lock after certain time of oxidization.

Regulator: Regulator is a mechanical device that shows the total pressure inside of the gas cylinder and also allow you to choose the output pressure.

Gas Flowmeter: Gas flow meter is must have unit to control the glow of the gas into the chamber. **Observation Window:** The observation window is a very helpful part of main chamber that allow you to see inside of chamber and illuminate the inside. It seals the chamber by a Viton O-ring.

FTM Rotational Knob: This knob is located on the top and center of main chamber which is for rotating the FTM inside of the chamber and place it on the top of either one of sputtering gun to measure the deposition rate. Please Note that you rotate it ONLY 90 degrees to right for Nb or Left for Al





Sample Loading: (For loading a new chip only). If already loaded go to Next page

This is a very sensitive part of operation and care must be taken to follow the procedure carefully and "In-Order" to avoid any kind of disaster including damaging the turbo pumps and the dry pumps.

- 1) To avoid air leakage into the main chamber and pumps and damaging them, make sure the Gate B, Gate C and Valve 2 are CLOSED. Please Note that valve 2T that allows pumping the small turbo is always open unless it is indicated to be closed. Always Clockwise(CW) is closining.
- 2) Open the Gray and Black valve on the Nitrogen gas cylinder (CCW will open).
- 3) Open valve 3 AND black On/Off valve to flow N₂ gas into the load lock. See next page to identify the black N₂ valve
- 4) Lose the load lock black door knob and wait to feel N2 gas coming out from door.
- 5) Take the sample holder out and load a new Si chip in the holder (Polished side facing down).
- * Do not tighten the middle screw on the bridge of the sample holder. Check for mask flexibility. Tighten the other two to hold down the Cu heatsink which goes on the top of the Si chip.
- 6) To load the sample holder into load lock, tilted it and inserted into Sliding rod, The bridge should face the top window, while the yellow handle at the end of the sliding part face down.
- 7) Close the load lock door, but do not tight the black locking knob. Next, close valve 3 (as well as On/off N2 valve-no more N_2 flow) See next page to identify the black N_2 valve.,

Then tighten the door **black knob**.

Now, Close valve 2T now to prevent the high pressure of load lock goes into the turbo pump and damage it.

8) Then Open valve2 (pumping) and check CG2 (convectron 2) value. It should reach to mid 10⁻³ Torr.

To see CG2 as 1×10^{-3} Torr quickly, When CG2 reached mid 2×10^{-2} or mid 10^{-3} Torr, close valve 2 and open Valve 2T and open Gate C. With this action, the small turbo pump helps for faster pumping of the load lock and will go to low 1×10^{-3} Torr.

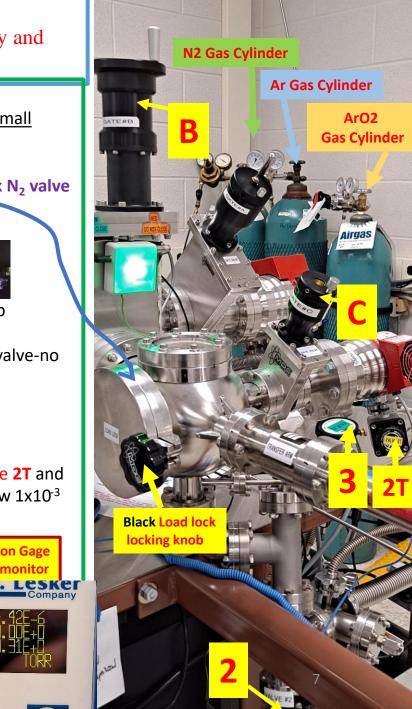
Now the load lock and the main chamber are ready to be connected to each other. For that:

9) Close Gate C (small gate) and open Gate B. when you open the gate B, notice that IG value

will change to mid 10-6 or possibly high 10-7.

10) Now, if you wait 10-20 min, IG value reach to 1x 10⁻⁷ torr.

Please Note that if IG is 10-7, you see **CG1** is **zero**. Because it is reading the pressure of the main chamber which is lower than 10⁻³ and that is the limit of Convectron Gage. Lower pressure will show up as Zero.



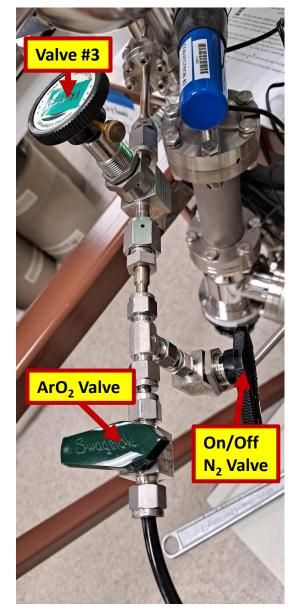


Important tips for the Nitrogen gas line needed for purging and ArO2 gas line which is needed During oxidization of Al.



After you are done with loading your sample make sure that the Nitrogen black valve shown in the photo is closed and stay closed until you want to take out your sample. The arrow should point horizontal.

For oxidization, the Green ArO_2 gas line should be open and make sure that the N_2 Valve is closed. Also, after the load lock chamber reached the expected atmospheric pressure, Close the valve.



Depositing the Niobium (Nb) or Aluminium (Al) bottom layer



Make sure the Guns Shutter is on Closed position AND Your Sample is still in the load lock area.



This step is only for turning the guns ON



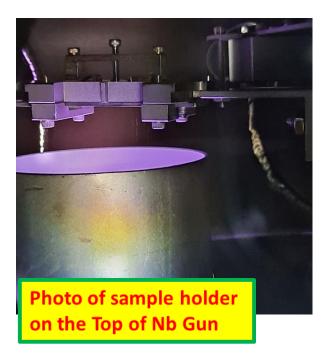
- 1) First make sure IG should be around 3.10⁻⁷ torr or lower. Open the Ar gas main valve (gray) and the side blue valve.
- 2) (* Most of time both are open).
- 3) Also open the 3^{ed} Ar gas valve below the flowmeter facing the south wall. Notice IG goes up to mid 10⁻³ Torr
- 4) Turn ON the chiller. It is interlocked to both Nb and Al guns. They will not turn on if no water flow.
- 5) Turn ON the MDX power supply for both Nb and Al guns from back of the units.
- 6) Slowly increase the power on Nb to 100 W and Al to 50W and
- 7) push the start button on the left side you should see a purple color plasma like shown in the photo.
- 8) For example, if you measure DR for Al as 2.5, you can use it as 2.2 to calculate deposition time because of heat during measurement when FTM is on top of Al gun.
- 9) If it did not light up, check pressure of IG to be set at 4-6x10⁻³ Torr and also if you open the chimney it will help.
- 10) Choose program **F1** on the FTM for **Nb** (choose program- then **F1** –enter- program) and after measurement of the deposition rate for Nb, Change it to **F2** for **AL**.
- 11) Rotate the FTM on the top of **Nb (CW)** or **Al (CCW)** to measure deposition rate.
- 12) The typical deposition rate for Nb is about 3 Å/Sec and Al about 2 Å/sec.
- 13) After the deposition rate measurement is done, move the FTM to back of the chamber, away from the path of the sample holder
- 14) Now you are ready for deposition of Nb or Al or both.



Function of chiller: To supply cooling water to cool down the sputtering guns.

Chiller must be running to turn on the sputtering guns.







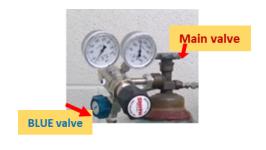




chiller







If MDX is blinking, this means you need Ar gas, it should be open or if you have gas and still blinking, you can open chimney.



A) First Experiment is to Deposit ONLY Nb, for measuring Transition Temperature (Tc) of Nb from Normal State to Superconducting State



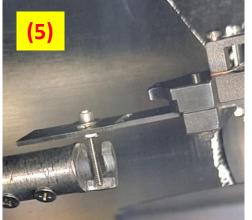
- **
 Make sure the chimney (Shutter) is on closed position.
- 1) Make sure the mask is on the first narrow (normal position without doing anything after loading sample) strip of the sliding mask (Fig 1). You can check it from front large window by rotating 90° toward window.
- 2) Rotate 180 to have the yellow indicator be on the bottom of the load lock arm and the substrate facing down.
- 3) Gate B should be open. Slide the sample holder on the top of Nb gun(facing down) and Open the chimney and record time for making 150 nm (1500Å).
- Calculate how many second expose to Nb, 1500 (Example: time(sec)=thickness of Nb (1500)/3), if deposition rate is 3Å/sec.
- 4) Pull out the sample back into the load lock area. After deposition, Close the shutter (Chimney).
- 5) Move the wobble stick all the way toward the middle of chamber and secure it with the black screw at the end of the wobble stick. Fig 4
- 6) Slide the sample holder rod toward the wobble stick.
- 7) Rotate 90 degrees to see the mask and observe the screw will go inside of U shape opening of the wobble stick Fig 5 and 6.
- 8) Very slowly pull out the sample rod to move the round part of the mask to the middle. The center of round hole must be aligned with the strips on the top and bottom of the sliding mask [see Fig (7)].
- 9) You should see the circular

 pattern now. Repeat step 2 and deposit 100nm (1000 Å) Nb again.
- 10) Move the sample holder to the top of Nb gun. Open chimney and after 1000/3= 5 min deposition, Close the shutter (Chimney)
- 11) Repeat step 5 to 9. This time you will see the vertical final top pattern.
- 12) For final top Nb strip see next page

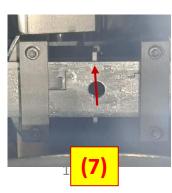














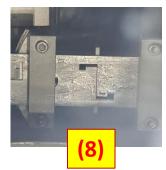
A) First Experiment is to Deposit ONLY Nb, for measuring Transition Temperature (Tc) of Nb from Normal State to Superconducting State



- 9) Bring the wobble stick again all the way toward the middle of the chamber and engage it with sample holder screw. Rotate 90 degrees to see the mask and observe that the screw will go inside of U shape opening of the wobble stick. You will be 3^{ed} position of mask
- 10) Slowly pull out the sample rod to move the final narrow pattern (Fig 8). The narrow part of pattern must be aligned with the strip on the top and bottom of the sliding mask [see Fig (8)].
- 11) After seeing the final mask , proceed and deposit final 150nm (1500Å) of Nb.
- 12) After deposition close the chimney.
- 13) Pull back the sample rod into the load lock and close the gate valve B.
- 14) Gently decrease Nb power value then Turn OFF the Nb gun
- 15) Let the rod and sample holder back into the load lock chamber. Still your sample is under the vacuum.
- 16) Make sure the gate valve B, gate valve C and Valve 2 are CLOSED.
- 17) Also make sure the Nitrogen tank valves and the black On/Off valve of N_2 is in open position.

Note that the CG2 is around 10⁻³ now.

- 18) Loose the load lock knob;
 - Open valve 3 to flow N₂ gas into the load lock until CG2 value be at atmospheric pressure of about 760 Torr.
- 19)Wait to feel N₂ gas coming out from load lock door, then close the vale 3 and reopen it only one turn to avoid any leakage of air into the load lock area
- 20) Let the sample holder cool down for 10-15 min.
- 21) Turn OFF MDX and Chiller. Close Ar gas on side.
- 22) Take the sample holder out and load a new Si chip in the holder.
- 23) Close gate B, close valve 2. gate C was already closed.
- 24) Open N_2 gas and valve 3 while you are unloading and loading a new chip. Otherwise close all N_2 related valves.



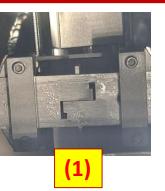


B) NIS junction –Step 1 (AI)

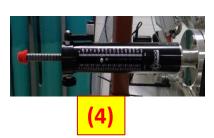
Deposition of Al , Al₂O₃, and Nb

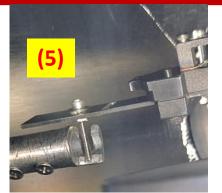


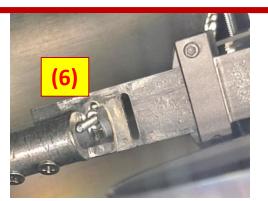
- ** Make sure the chimney (Shutter) is on closed position.
- 1) Make sure the mask is on the first narrow (normal position without doing anything after loading sample) strip of the sliding mask (Fig 1). You can check it from front large window by rotating 90° toward window.
- 2) Rotate 180 to have the yellow indicator be on the bottom of the load lock arm and the substrate facing down.
- 3) Gate B should be open. Slide the sample holder on the top of Al gun(facing down) and Open the chimney and record time for making 150 nm (1500Å). Calculate how many second expose to Al, 1500 (Example: time(sec)=thickness of Nb (1500)/2), if deposition rate is 2Å/sec.
- 4) Pull out the sample back into the load lock area. After deposition, Close the shutter (Chimney).
- 5) Move the wobble stick all the way toward the middle of chamber and secure it with the black screw at the end of the wobble stick. Fig 4
- 6) Slide the sample holder rod toward the wobble stick.
- 7) Rotate 90 degrees to see the mask and observe the screw will go inside of U shape opening of the wobble stick Fig 5 and 6.
- 8) Very slowly pull out the sample rod to move the round part of the mask to the middle. The center of round hole must be aligned with the strips on the top and bottom of the sliding mask [see Fig (7)].
- 9) You should see the circular pattern now. Pull Back the wobble stick, otherwise the sample holder can not go on the top of Al Gun
- 10) For circular mask, measure Al DR again. Then deposit 50 Å of Al and close the shutter.
- 11) Close the shutter (Chimney) and turn OFF the Al gun.
- 12) Pull back the sample rod into the load lock and close the gate valve B.
- 13) Make sure the gate valve B, gate valve C and Valve 2 are CLOSED.
- 14) Turn of the Ar gas on the left side of chamber.
- 15) Go to the next page for oxidization process of Al.

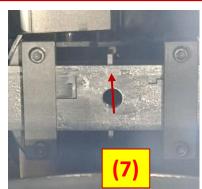














B) NIS junction – Step 2 Oxidization (Al₂O₃)

Deposition of Al , Al₂O₃, and Nb



- 1) Open the Gray and Black valve on the ArO₂ gas cylinder (CCW is open).
- 2) Open the green ON/OFF valve which is next to Valve #3 (see the photo).
- 3) Very slowly, open valve 3 (before opening it, CG2 is 10-3), then open valve 3 and watch CG2 on the Kurt
- J. Lesker gauge goes up to about 60 Torr, then close valve3.
- 4) Wait for 15 min. While waiting, Close all ArO₂ gas valves (3 of them).
- 5) Check 2T should be closed to able to pump.
- 6) Open Vale 2 to take out ArO₂ from Load lock and check CG2 value. When it reached mid 2x10⁻² Torr,
- 7) Close valve 2 and open Valve 2T and gate Valve C . With this small turbo pump, the load lock will quickly go to low 1×10^{-3} torr.
- 8) Wait 20 min, Now the load lock and the main chamber are ready to be connected to each other.
- 9) Close gate valve C and open Gate valve B.
- 10) IG1 should change to mid or low 10⁻⁷ torr. If not, wait little bit longer to reach there.
- 11) Open the side Ar gas valve
- 12) Turn ON the MDX power supply of Nb from back of the unit.
- 13) Slowly increase the power on Nb to 100 W
- 14) Push the start button on the left side you should see a purple color plasma.
- 15) Measure DR for Nb and calculate deposition time.
- 16 Now you are ready for the deposition of Nb.



How we decide about thickness of our junction?

For first experiment which is just Nb, as long as we have thick enough layer (75nm) that Become superconductor at liquid helium temperature of 4.2 K, we can make 100nm or 200nm. But the thick film usually gets rougher on the surface. For AlO₂ layer, first we make about 5nm of Al, because when we oxidize the Al, the oxygen can only defuse in about 2.5 nm and if it is thicker, we will have a very thin normal Al layer between Al (Normal) layer and insulator which will.



B) NIS junction – Step 3 (Nb)

Deposition of Al, Al₂O₃, and Nb



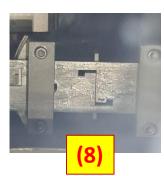
- 9) Bring the wobble stick again all the way toward the middle of the chamber and engage it with sample holder screw. Rotate 90 degrees to see the mask and observe that the screw will go inside of U shape opening of the wobble stick. You will be 3^{ed} position of mask
- 10) Slowly pull out the sample rod to move the final narrow pattern [Fig 8]. The narrow part of pattern must be aligned with the strip on the top and bottom of the sliding mask [see Fig (8)].
- 11) After seeing the final mask, proceed and deposit final 150nm (1500Å) of Nb.
- 12) After deposition close the chimney.
- 13) Pull back the sample rod into the load lock and close the gate valve B.
- 14) Gently decrease Nb power value then Turn OFF the Nb gun
- 15) Let the rod and sample holder back into the load lock chamber. Still your sample is under the vacuum.
- 16) Make sure the gate valve B, gate valve C and Valve 2 are CLOSED.
- 17) Also make sure the Nitrogen tank valves and the Black On/Off valve of N_2 is in open position.

Note that the CG2 is around 10⁻³ now.

18) Loose the load lock knob

Open valve 3 to flow N₂ gas into the load lock until CG2 value be at atmospheric pressure of 760 Torr.

- 19) Wait to feel N₂ gas coming out from load lock door, then close the vale 3 and reopen it only one turn to avoid any leakage of air into the load lock area
- 20) Let the sample holder cool down for 10-15 min.
- 21) Turn OFF MDX and Chiller. Close Ar gas on side.
- 22) Take the sample holder out and load a new Si chip in the holder.
- 23) Close gate B, close valve 2. gate C was already closed.
- 24) Open N_2 gas and valve 3 while you are unloading and loading a new chip. Otherwise close all N_2 related valves.



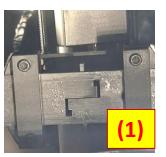


C) SIN junction – Step 1 (Nb-Al)₃

Deposition of Nb-Al, Al₂O₃, and Al

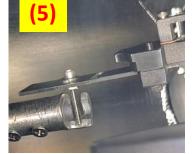


- ** Make sure the chimney (Shutter) is on closed position and sample holder stay in load lock.
- 1) Use directions in page 9 to turn on Both Nb and Al guns and measure deposition rates.
- 1) Make sure the mask is on the first narrow (normal position without doing anything after loading sample) strip of the sliding mask (Fig 1). You can check it from front large window by rotating 90° toward window.
- 2) Rotate 180 to have the yellow indicator be on the bottom of the load lock arm and the substrate facing down.
- 3) To make SIN junction, the best is to make a multilayers of Nb-Al which will have smoother surface
- 4) We want to make this multilayers: $[Nb(250 \text{ Å})-Al(25 \text{ Å})]_3-Nb(200 \text{ Å})$
- 5) Calculate the time for depositing 250 Å of Nb, 25 Å of Al and 200Å of Nb.
- 6) Slide the sample holder on the top Nb gun and Open the chimney and record time for making 25 nm (250Å).
- 7) Then slid it on top of Al gun and record time for making 2.5 nm (25Å). Do this 3 times and finish with Nb(200 Å).
- 8) Pull out the sample back into the load lock area. After deposition, Close the shutter (Chimney).
- 9) Move the wobble stick all the way toward the middle of chamber and secure it with the black screw at the end of the wobble stick. Fig 4
- 10) Slide the sample holder rod toward the wobble stick.
- 11) Rotate 90 degrees to see the mask and observe the screw will go inside of U shape opening of the wobble stick Fig 5 and 6.
- 12) Very slowly pull out the sample rod to move the round part of the mask to the middle. The center of round hole must be aligned with the strips on the top and bottom of the sliding mask [see Fig (7)].
- 13) You should see the circular pattern now. Repeat step 2 and deposit 100nm (1000 Å) Nb again.
- 14) Move back the wobble stick.
- 15) Move the sample holder to the top of Al gun. Open chimney and after deposition 50Å of Al, close the shutter (Chimney)
- 16) Go to the next page for oxidization process of Al.















C) SIN junction – Step 2 Oxidization (Al₂O₃)

Deposition of Nb-Al, Al₂O₃, and Al



- 1) Open the Gray and Black valve on the ArO_2 gas cylinder (CCW is open).
- 2) Open the green ON/OFF valve which is next to Valve #3 (see the photo).
- 3) Very slowly, open valve 3 (before opening it, CG2 is 10-3), then open valve 3 and watch CG2 on the Kurt
- J. Lesker gauge goes up to about 60 Torr, then close valve3.
- 4) Wait for 15 min. While waiting, Close all ArO₂ gas valves (3 of them).
- 5) Check 2T should be closed to able to pump.
- 6) Open Vale 2 to take out ArO₂ from Load lock and check CG2 value. When it reached mid 2x10⁻² Torr,
- 7) Close valve 2 and open Valve 2T and gate Valve C . With this small turbo pump, the load lock will quickly go to low 1×10^{-3} torr.
- 8) Wait 20 min, Now the load lock and the main chamber are ready to be connected to each other.
- 9) Close gate valve C and open Gate valve B.
- 10) IG1 should change to mid or low 10⁻⁷ torr. If not, wait little bit longer to reach there.
- 11) Open the side Ar gas valve
- 12) Turn ON the MDX power supply of Al.
- 13) Slowly increase the power on Al to 50 W
- 14) Push the start button on the left side you should see a purple color plasma.
- 15) Measure DR for Al and calculate deposition time.
- 16) Now you are ready for the deposition of Al.



How we decide about thickness of our junction?

For first experiment which is just Nb, as long as we have thick enough layer (75nm) that Become superconductor at liquid helium temperature of 4.2 K, we can make 100nm or 200nm. But the thick film usually gets rougher on the surface. For AlO₂ layer, first we make about 5nm of Al, because when we oxidize the Al, the oxygen can only defuse in about 2.5 nm and if it is thicker, we will have a very thin normal Al layer between Al (Normal) layer and insulator which will.



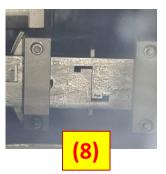
C) SIN junction – Step 1 (Al)

Deposition of Nb-Al, Al₂O₃, and Al



- 9) Bring the wobble stick again all the way toward the middle of the chamber and engage it with sample holder screw. Rotate 90 degrees to see the mask and observe that the screw will go inside of U shape opening of the wobble stick. You will be 3^{ed} position of mask
- 10) Slowly pull out the sample rod to move the final narrow pattern [Fig 8]. The narrow part of pattern must be aligned with the strip on the top and bottom of the sliding mask [see Fig (8)].
- 11) After seeing the final mask , proceed and deposit final 120nm (1200Å) of Al.
- 12) After deposition close the chimney.
- 13) Pull back the sample rod into the load lock and close the gate valve B.
- 14) Turn OFF both Al and Nb gun
- 15) Let the rod and sample holder back into the load lock chamber. Still your sample is under the vacuum.
- 16) Make sure the gate valve B, gate valve C and Valve 2 are CLOSED.
- 17) Also make sure the Nitrogen tank valves and the black On/Off valve of N₂ is in open position.

 Note that the CG2 is around 10⁻³ now.
- 18) Loose the load lock knob
 - Open valve 3 to flow N₂ gas into the load lock until CG2 value be at atmospheric pressure of 760 Torr.
- 19)Wait to feel N₂ gas coming out from load lock door, then close the vale 3 and reopen it only one turn to avoid any leakage of air into the load lock area
- 20) Let the sample holder cool down for 10-15 min.
- 21) Turn OFF MDX and Chiller. Close Ar gas on side.
- 22) Take the sample holder out and load a new Si chip in the holder.
- 23) Close gate B, close valve 2. gate C was already closed.
- 24) Open N_2 gas and valve 3 while you are unloading and loading a new chip. Otherwise close all N_2 related valves.



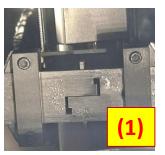
MICHIGAN STATE

D) SIS junction – Step 1 (Nb-Al)₃

Deposition of Nb-Al, Al₂O₃, and Nb

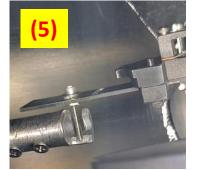


- ** Make sure the chimney (Shutter) is on closed position and sample holder stay in load lock.
- 1) Use directions in page 9 to turn on Both Nb and Al guns and measure deposition rates.
- 1) Make sure the mask is on the first narrow (normal position without doing anything after loading sample) strip of the sliding mask (Fig 1). You can check it from front large window by rotating 90° toward window.
- 2) Rotate 180 to have the yellow indicator be on the bottom of the load lock arm and the substrate facing down.
- 3) To make SIN junction, the best is to make a multilayers of Nb-Al which will have smoother surface
- 4) We want to make this multilayers: $[Nb(250 \text{ Å})-Al(25 \text{ Å})]_3-Nb(200 \text{ Å})$
- 5) Calculate the time for depositing 250 Å of Nb, 25 Å of Al and 200Å of Nb.
- 6) Slide the sample holder on the top Nb gun and Open the chimney and record time for making 25 nm (250Å).
- 7) Then slid it on top of Al gun and record time for making 2.5 nm (25Å). Do this 3 times and finish with Nb(200 Å).
- 8) Pull out the sample back into the load lock area. After deposition, Close the shutter (Chimney).
- 9) Move the wobble stick all the way toward the middle of chamber and secure it with the black screw at the end of the wobble stick. Fig 4
- 10) Slide the sample holder rod toward the wobble stick.
- 11) Rotate 90 degrees to see the mask and observe the screw will go inside of U shape opening of the wobble stick Fig 5 and 6.
- 12) Very slowly pull out the sample rod to move the round part of the mask to the middle. The center of round hole must be aligned with the strips on the top and bottom of the sliding mask [see Fig (7)].
- 13) You should see the circular pattern now. Repeat step 2 and deposit 100nm (1000 Å) Nb again.
- 14) Move back the wobble stick.
- 15) Move the sample holder to the top of Al gun. Open chimney and after deposition 50Å of Al, close the shutter (Chimney)
- 16) Go to the next page for oxidization process of Al..















D) SIS junction – Step 2 Oxidization (Al₂O₃)

Deposition of Nb-Al, Al₂O₃, and Nb



- 1) Open the Gray and Black valve on the ArO₂ gas cylinder (CCW is open).
- 2) Open the green ON/OFF valve which is next to Valve #3 (see the photo).
- 3) Very slowly, open valve 3 (before opening it, CG2 is 10-3), then open valve 3 and watch CG2 on the Kurt
- J. Lesker gauge goes up to about 60 Torr, then close valve3.
- 4) Wait for 15 min. While waiting, Close all ArO₂ gas valves (3 of them).
- 5) Check 2T should be closed to able to pump.
- 6) Open Vale 2 to take out ArO₂ from Load lock and check CG2 value. When it reached mid 2x10⁻² Torr,
- 7) Close valve 2 and open Valve 2T and gate Valve C . With this small turbo pump, the load lock will quickly go to low 1×10^{-3} torr.
- 8) Wait 20 min, Now the load lock and the main chamber are ready to be connected to each other.
- 9) Close gate valve C and open Gate valve B.
- 10) IG1 should change to mid or low 10⁻⁷ torr. If not, wait little bit longer to reach there.
- 11) Open the side Ar gas valve
- 12) Turn ON the MDX power supply of Al.
- 13) Slowly increase the power on Al to 50 W
- 14) Push the start button on the left side you should see a purple color plasma.
- 15) Measure DR for Al and calculate deposition time.
- 16) Now you are ready for the deposition of Al.



How we decide about thickness of our junction?

For first experiment which is just Nb, as long as we have thick enough layer (75nm) that Become superconductor at liquid helium temperature of 4.2 K, we can make 100nm or 200nm. But the thick film usually gets rougher on the surface. For Al₂O₃ layer, first we make about 5nm of Al, because when we oxidize the Al, the oxygen can only defuse in about 2.5 nm and if it is thicker, we will have a very thin normal Al layer between Al (Normal) layer and insulator which will.



D) SIS junction – Step 3 (Nb)

Deposition of Nb-Al, Al₂O₃, and Nb



- 9) Bring the wobble stick again all the way toward the middle of the chamber and engage it with sample holder screw. Rotate 90 degrees to see the mask and observe that the screw will go inside of U shape opening of the wobble stick. You will be 3^{ed} position of mask
- 10) Slowly pull out the sample rod to move the final narrow pattern [[Fig 8]. The narrow part of pattern must be aligned with the strip on the top and bottom of the sliding mask [see Fig (8)].
- 11) After seeing the final mask , proceed and deposit final 150nm (1500Å) of Nb.
- 12) After deposition close the chimney.
- 13) Pull back the sample rod into the load lock and close the gate valve B.
- 14) Gently decrease Nb power value then Turn OFF the Nb gun
- 15) Let the rod and sample holder back into the load lock chamber. Still your sample is under the vacuum.
- 16) Make sure the gate valve B, gate valve C and Valve 2 are CLOSED.
- 17) Also make sure the Nitrogen tank valves and the black On/Off valve of N₂ is in open position.

 Note that the CG2 is around 10⁻³ now.
- 18) Loose the load lock knob
 - Open valve 3 to flow N₂ gas into the load lock until CG2 value be at atmospheric pressure of about 760 Torr.
- 19)Wait to feel N₂ gas coming out from load lock door, then close the vale 3 and reopen it only **one turn** to avoid any leakage of air into the load lock area
- 20) Let the sample holder cool down for 10-15 min.
- 21) Turn OFF MDX and Chiller. Close Ar gas on side.
- 22) Take the sample holder out and load a new Si chip in the holder.
- 23) Close gate B, close valve 2. gate C was already closed.
- 24) Open N_2 gas and valve 3 while you are unloading and loading a new chip. Otherwise close all N_2 related valves.

