Kurt J. Lesker PVD Manual

THINGS TO NOTE

Check/observe the cryo temperature, it should be in the range of 11.5 - 13 K. If it is out of range it could mean the cryo will need to be regenerated.

Remember when pumping down turn on pumps before opening valves, and when venting close any valves before turning off any pumps.

Computer password is ‘jiaogroup’

CWare dep software info user: nano, password: nano123 → Hit ‘Enter’ twice to log in

NO DI water for chillers, it is not good for the piping.

To help equalize pressure in the main chamber change crucible positions, open/close the e-beam shutter and move window knobs. This gets the air (or lack thereof) in the chamber moving around and helps the pressure to equalize.

Main chamber pressure should be around 10^{-8}, but if it has just been opened and then pumps down it may only reach 10^{-7}. 10^{-8} usually requires pump down overnight.

Prior to depositing, pour the water from the dehumidifier into the two ground holes behind the PVD. <50% is bad, dehumidifier can get down to 35%

Note that for the software, Green text/numbers are a reading while white characters are changeable.

The e-beam power supply requires at least 10 minutes to warm up, if you are planning to use the e-beam, make sure to turn on the e-beam power supply unit at the bottom of the rack, it is the unit with the large breaker-style switch.

Under the ‘Motion’ tab the 1st platen motor should be “Referenced”. This is indicated by the small green-shaded box. If the box is not green, then simply click the home button for the 1st platen motor to reference it.

10 Angstroms = 1 nanometer, 1kA = 100 nm

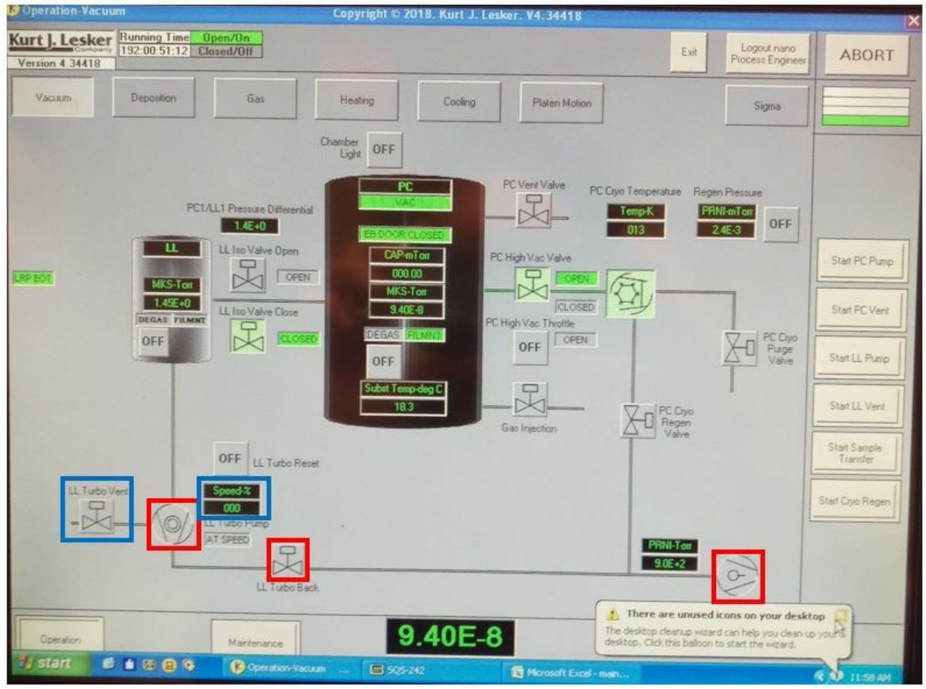
For any ion process (sputtering, plasmas) the cryo needs to be throttled to prevent ions from getting into the cryo chamber and reacting with the activated carbon inside of the pump. Before starting any plasma or sputtering procedures ensure the cryo is throttled by clicking PC High Vac Throttle.

Only use vacuum safe tape - Kapton Tape -, in addition, when using tape **<u>DO NOT</u>** use the heater.

LOADING SAMPLES

1. Turn on the nitrogen and argon gas in the other room using the valves above the tanks, **not** on the regulators.

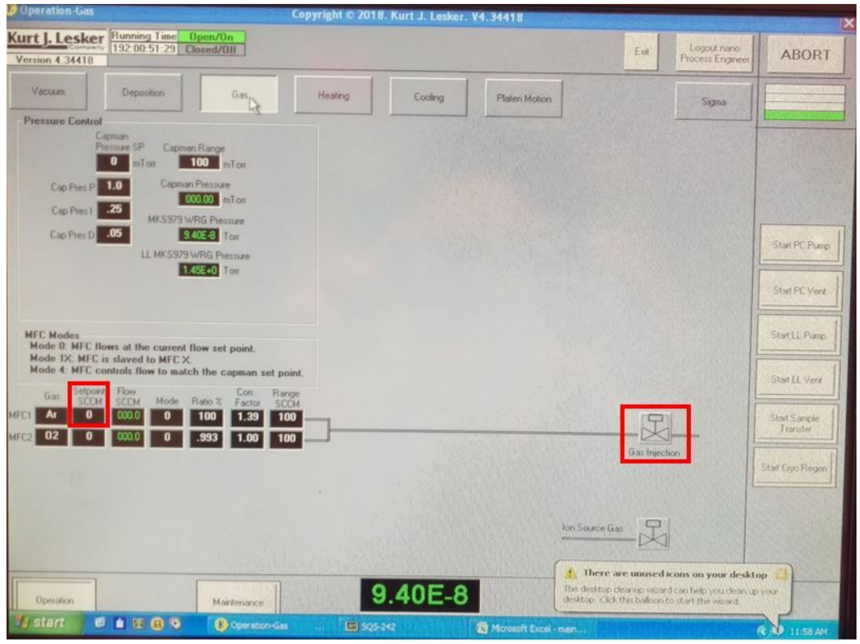
2. The turbo pump and rough pump need to be off in order to vent the load lock and load samples. Under the 'Vacuum' tab, turn off the turbo by clicking LL Turbo Pump (red box), close the turbo valve by clicking LL Turbo Back (red box) and then turn off the rough pump (red box). Once the turbo spins down below 75 percent speed (blue box), click LL Turbo Vent (blue box) to vent the load lock with nitrogen.



3. Once vented, remove the wafer holder from the loading station by rotating it counterclockwise and pulling out. Carefully clamp down the wafer on the holder making sure that it is clamped securely on the holder. Put the wafer holder back on the loading station and lock it by rotating it clockwise.

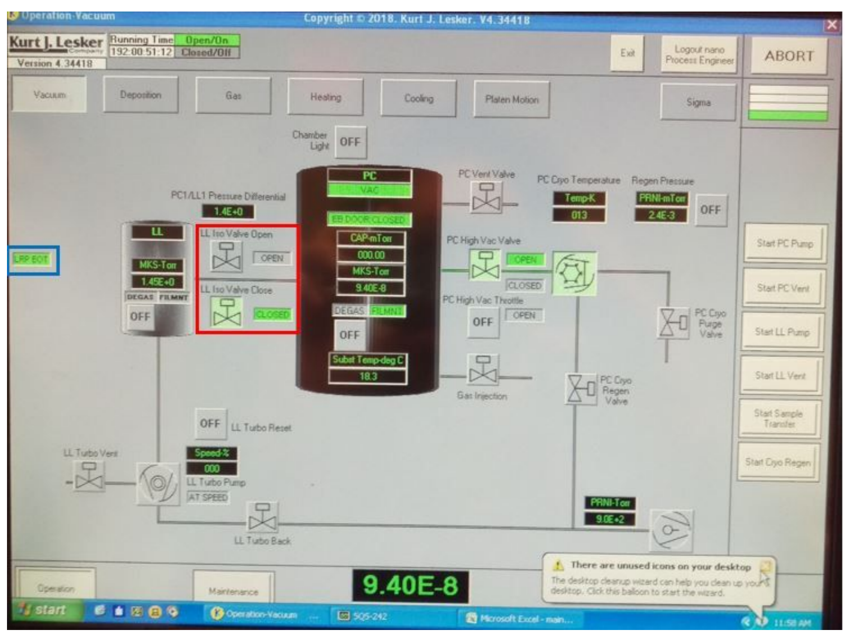
4. Close the LL chamber door, turn on the rough pump, and open the LL Turbo Back valve while holding the LL chamber door shut until it seals from the vacuum. Wait until the pressure in the load lock reduces to 10^{-1} torr. Once the pressure has dropped to 10^{-1} torr in the load lock, turn on the turbo pump and wait until the load lock pressure further reduces to 10^{-5} torr.

5. Flow 2 sccm of argon into the main chamber by selecting the ‘Gas’ tab, and inputting 2 in the setpoint box for MFC1 (red box). Click ‘OK’ and then click the gas injection button (red box). Wait until the pressure in the main chamber is above the pressure in the load lock chamber, usually once the main chamber reaches $5x10^{-5}$ torr. A higher pressure in the main chamber when opening the load lock will ensure that gas flows from the main chamber into the load lock. This avoids introducing unwanted particles that may have settled in the load lock from being introduced into the main chamber.



6. Once the primary chamber pressure is greater than the pressure in the load lock, click LL Iso Valve close and then LL Iso Valve open (red box) to open the load lock door, it is a 2-step process.

7. Slide the electromagnetic handle to the left which moves the wafer holder into the primary chamber. In order to attach the wafer holder the stage motor must be turned off. Select the ‘Motion’ tab and turn off the 2nd platen motor. Next screw the sample into the stage by turning the handle clockwise direction until it is snug and then slowly pull the handle away. Make sure the electromagnet is pulled all the way back and into the load lock then close the load lock. If it's not moved all the way back, the LRP EOT on the left will not turn green and you will not be able to close the load lock. Close load lock isolation valve, Stop the flow of argon gas by setting the MFC1 gas setpoint to 0 under the ‘Gas’ tab and clicking on the gas injection button.



REMOVING SAMPLES

1. Under ‘Motion’ tab, select position 6 and click on Go To station. Again make sure the button indicates motion with green. Stop rotation if you used it, push the Fwd button to do this, also turn off the cryo throttle if sputter was used.

2. Under the ‘Gas’ tab, flow 2 sccm of argon into the primary chamber. Wait until the pressure in the primary chamber goes above the pressure of load lock chamber before opening.

3. Open the load lock by clicking LL Iso Valve open. Push the stage holder inside the main chamber and bring the wafer holder to the load lock. Close the load lock by first clicking on LL Iso Valve open and then LL Iso Valve close. If the load lock does not close, make sure EOR indicator on the left side of the screen is turned green. If not, then pull back the electromagnet handle all the way back and try again. Set argon flow rate to 0 and click gas injection.

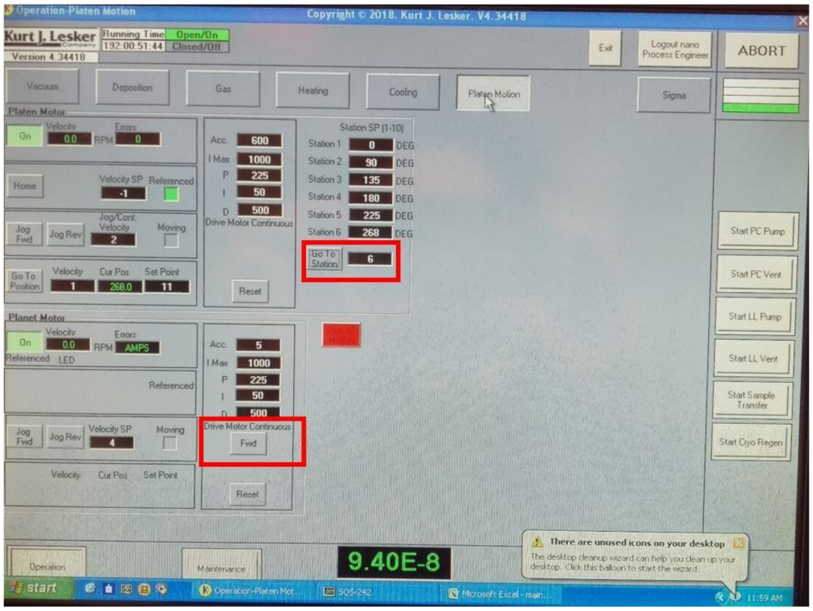
4. Turn off the turbo pump, close the LL Turbo Back Valve and turn off the rough pump. Wait until the turbo spins down below 75 percent. Then press LL Turbo Vent button to vent the LL chamber.

5. If done using the PVD for the day, replace the wafer holder into the LL arm and close the LL door. Turn on the Rough Pump and open the LL Turbo Back Valve. Wait until the pressure reaches 10-1 torr before closing the LL Turbo Back Valve and turning off the Rough Pump.

6. Before leaving the PVD room make sure of the following:

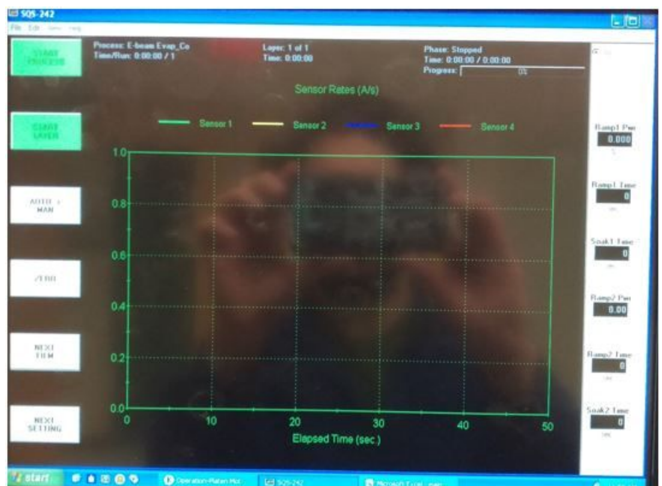
1. All lights off - In the room as well as the PVD chamber itself.
2. No gas is flowing - Close valves for any gases in use
3. No movement in the PVD - Stage rotation or otherwise
4. Power supplies turned off

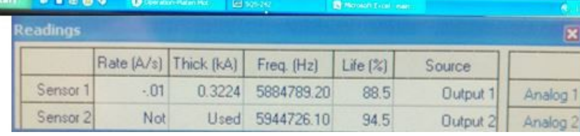
E-BEAM DEPOSITION



1. Select the ‘Motion’ tab and click the input box (red box) next to the Go to Station. Select the appropriate station position on the number pad and click OK. Choose the position 4 (opposite of e-beam). Press the Go to Station button to move the stage, the button will turn green. If the stage doesn’t move when the button is pressed, press again. Rotate if desired (red box).

2. Open up Deposition Control software named ‘SQS-242’ and click on process tab, click select correct program and then choose the correct deposition program from the list. Next, go to the view tab and click on sensor readings. The sensor reading window will provide information on the deposition rate and deposition thickness on the sample.

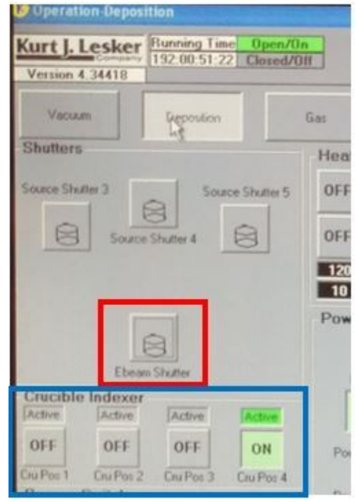




NOTE: Never operate the e-beam if the pressure is greater than $5x10^{-7}$ torr, if pressure reaches $5.3x10^{-6}$ torr during operation, stop immediately.

3. Make sure the power supply for the e-beam is turned on. If it was not turned at the beginning, turn it on and allow it to warm up for 10 minutes (Red box). Turn on the sweep control box (blue box).

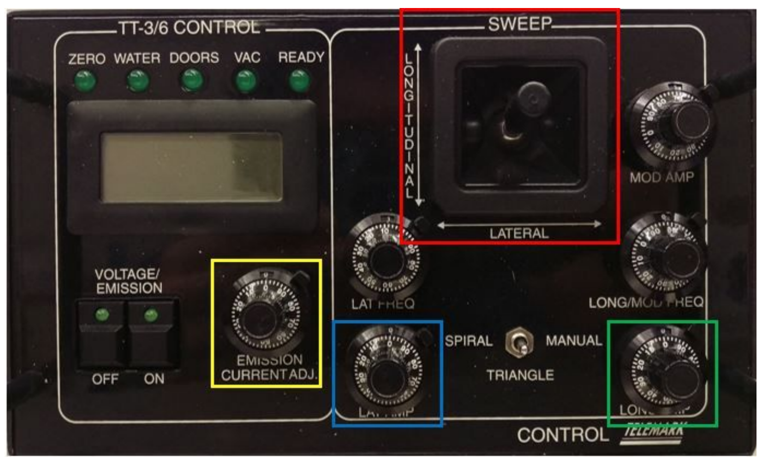
4. Make sure the e-beam crucible shield is open and then under the ‘Deposition’ tab, click on E-beam shutter (red box) to open the shutter between e-beam sample holder and the main chamber. Select the correct crucible (blue box). Peek inside the chamber through the window to check if you can spot the liner on the mirror.



5. Once e-beam power supply is warmed up, flip the switch to manual and then press ON button on the controller. Keep an eye on the cut back indicator (yellow light) on the e-beam power supply at all times. If the cut back indicator light stays on, immediately hit Off button on the controller. Dial up the voltage knob but do not exceed 12 V, on the power supply (bottom of the rack unit) and try turning the controller back on. If the cut back indicator stays off, turn off the controller and bring the voltage back down to the original value. If not, repeat this step over.

NOTE: It is normal for the cut back indicator to flash once upon startup, do not be alarmed.

6. If the metals in the crucible are in pellet form, they will need to be melted first. Slowly increase the current knob on the controller to 10 mA (yellow box). Look at the mirror inside the chamber to check if the beam is hitting inside of the liner. If not, use the stick on the controller (red box) to align the e-beam position.



NOTE: The area around the e-beam unit contains multiple very high voltage sources when in operation. This components on the chamber will arc if given the opportunity, be very cautious around these components and keep at least 2 feet from the main chamber during operation.

7. There are two additional knobs on the controller that are used to control the motion of the e-beam inside the liner. One knob gives lateral motion (blue box) and the other will give longitudinal motion (green box). On the sweep control box, check the alignment indicator to see if the lateral and longitudinal are aligned in the middle and only moving within three bars (one bar in either direction). If not, use the knobs on the controller to align the longitudinal and lateral motion by looking at e-beam through the mirror. Be sure to wear the sunshades during this step as the e-beam is very bright and will damage your eyes. Slowly increase the current by turning the current knob, 10 mA at a time waiting 30s inbetween to stabilize. At the same time checking

the e-beam alignment to ensure it is within the crucible. Lower the size of the raster by dialing down the lateral and longitudinal motion if necessary.

ATTENTION! Keep the maximum current used to below 200mA. Greater values leave more room for something to go catastrophically wrong, i.e. things break more easily at higher powers.

NOTE: Keep an eye on the main chamber pressure for any fluctuations. If the pressure increases check the e-beam raster to see if the beam is hitting the liner, as this is the primary reason for a pressure increase. If it is, turn down the current to a safe level and slowly re-align the beam to within the crucible. If the e-beam is in contact with the liner for too long it can crack the liner and cause numerous issues. A cracked liner, or overflowing crucible will be indicated by a sharp reduction in deposition rate as the metal has a thermal pathway to the water chilled copper source. You decide and good luck. If the pressure increase is an order of magnitude or greater, a cracked/faulty crucible is the most likely culprit.

8. Keep increasing the current on the controller slowly until the desired deposition rate is reached on the SQS sensor reading window. Keep note on the current value and deposition rate. Check on the mirror to see if the metal pellets have completely melted.

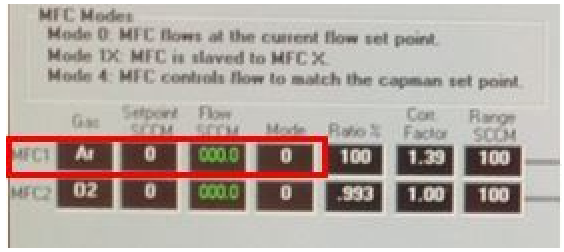
9. Once the desired dep rate is reached, and the pellets have melted completely, close the e-beam shutter. Go to the ‘Motion’ tab and change the position of the wafer to position 1. Make sure the green light is on which indicates motion. Wait until the motor stops at the targeted position and click on Zero on the left side of the SQS-142 window. This zeros the thickness of the deposited material. As quickly as possible go back to the ‘Vacuum’ Tab and open the e-beam shutter.

10. The sensor reading window will show the thickness of metal being deposited on the wafer. Keep an eye on the pressure reading of the main chamber for any abnormalities.

11. Once the desired thickness of deposited metal is reached, close the e-beam shutter and change the position of the sample back to position 4

12. Once the sample is at station 4, go back to ‘Deposition’ tab and open the e-beam shutter. Slowly bring down the current on the controller while watching through the mirror at the crucible. Sometimes the raster can move abruptly during the current reduction. Turn the controller off as well as the sweep control unit and then flip the switch on the e-beam power supply to auto mode. Put the controller away on the table and wait for the melt to cool down. Once the melt is no longer colored from the heat, move the crucible position to one that was not in use that day, a “cold” one, then close the e-beam shutter and manual shutter.

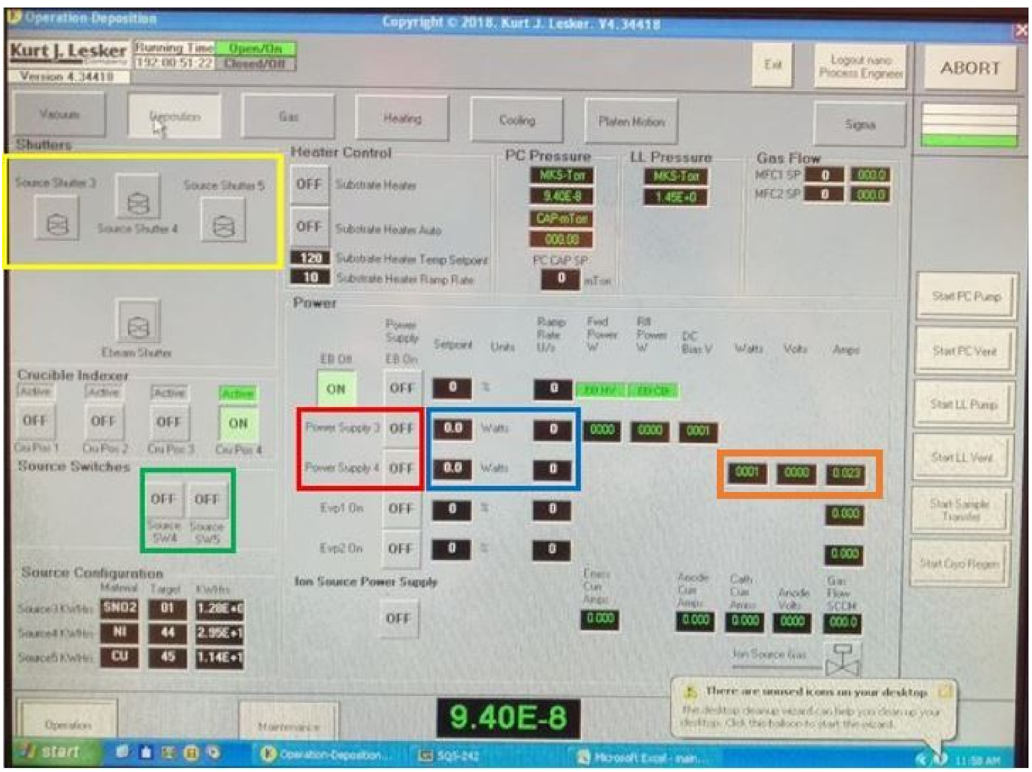
DC SPUTTER DEPOSITION



1. First thing is to throttle the cryo by clicking PC High Vac Throttle. Go to the ‘Gas’ tab and switch MFC1 to Mode 4 by clicking on the box, then set the capman pressure to 5 mTorr, and then click gas injection to flow argon into the primary chamber. Ensure that all window shutters are closed.

2. Open the SQS software and select the appropriate program from File → Process → 5Ni, where the number represents the location of the sputter target. Also, open the sensor readings window from File → View → Sensor Readings. In the sensor readings window, sensor 2 is for sputtering.

NOTE: The type of sputter target in each location is on the whiteboard on the wall of the PVD room. There are programs for most metal/location combinations saved in the SQS program.



3. Choose the source in the Source Switches (green box) corresponding to the sputter head to be used. And turn on the respective power supply for the given target (red box). PS4 is for targets 4 and 5. Next set the ramp rate and set point (blue box). For all metals the rate should be 10 W/s with a set point of 150 W. Set the ramp rate first to avoid the power supply slamming on to the set point.

ATTENTION! The magnet on sputter 5 is not as strong as the one on sputter 4, for this reason, do not exceed 225 W when using sputter 5. For sputter 4, try and keep the power below 275 W. Typically this corresponds to 5-6 Å/s for sputter 4 and 2-4 Å/s for sputter 5.

4. Once set, hold here for roughly 30 seconds to pre-clean the chamber. This helps to remove any metal flakes and other bits that may have attached themselves to the target.

NOTE: Issues can arise with the power supply at this stage. Keep a close eye on the power supply unit itself as well as the orange box in the photo above. If the power supply shows 750 V (max voltage) but zero amps and no power, the circuit is open. This can be solved by increasing the gas flow by slightly increasing the capman pressure. If the power supply shows 0 V, 2 Amps (max amperage) and no power then there is a short somewhere and the power supply unit will arc out and start flashing. This can be caused my small metal flakes from previous depositions interfering with the targets. To try and fix, turn on the power supply to 100 W with no ramp rate to try and blow the flakes off the target. If this doesn't work, let the system cool for a while and try again.

5. After the 30 seconds pre-clean process set the ramp rate to 10 W/s and change the set point to 225 W for the actual deposition. Once the power is at the set point and the deposition rate is satisfactory, start sample rotation and move the stage to the correct location under the 'Platen Motion' tab. Open the SQS software and 'zero' the sensor, then immediately under the 'Deposition' tab open the respective shutter (yellow box) to begin the deposition.

NOTE: If the shutters are stuck closed or open the pistons that control them most likely need cleaning. Deposited metal accumulates over time until they are inhibited from moving. Use a razor blade to carefully scrape the metal away, **DO NOT USE SOLVENTS**.

NOTE: PS3 is used for RF sputtering, it is not covered in this manual.

NOTE: It will be useful to record the deposition rate and time to your desired thickness as a function of your power level in the event that the QCM malfunctions. This can also be used in future depositions to get an idea of the deposition rate at a given power level.

6. Once desired thickness is achieved close the respective shutter and ramp down to 0 at 10 W/s. Once at 0, turn of the respective power supply and source switch, and move the stage to position 6. You may move the stage while the power supply is powering down.

7. Set the capman pressure back to 0 and change the mode on MFC1 back to 0.

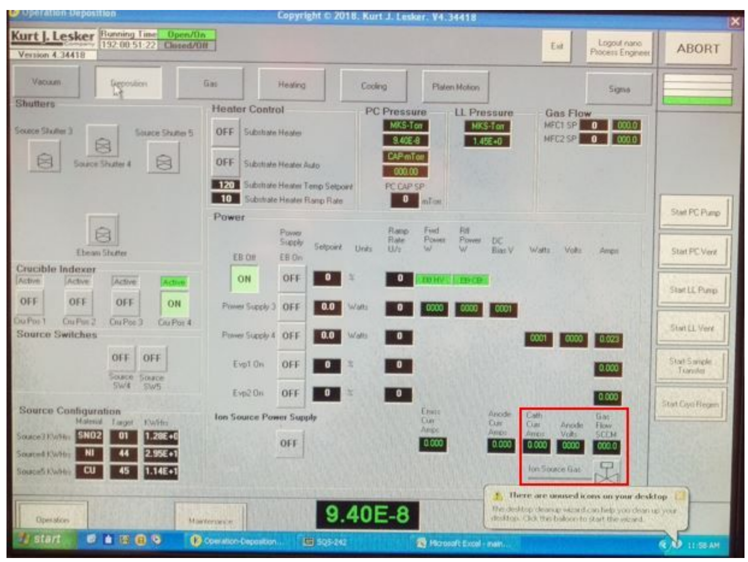
8. This process can be repeated by simply returning to step 2 and selecting the new metal.

OXYGEN PLASMA

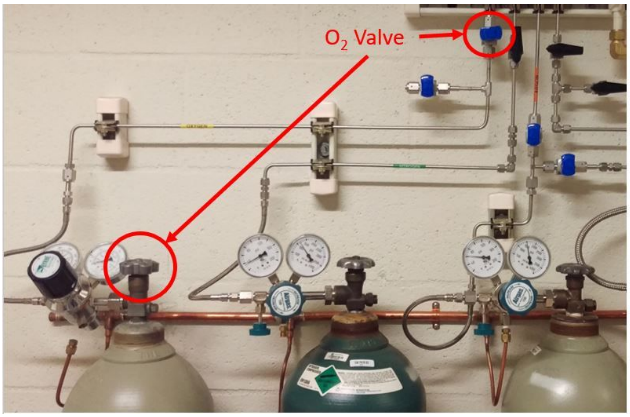
Procedure used when wafer cleanliness is believed to play a factor. Within first month of purchase wafers should be okay, after a month or so sitting the surface will need a cleaning. This is done after loading the samples but before any e-beam or DC sputtering procedures.

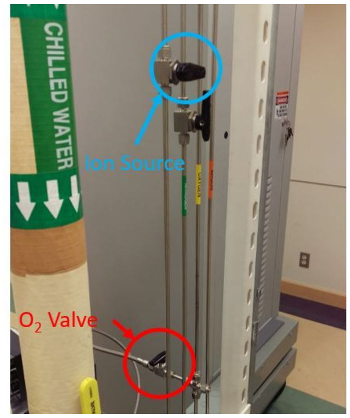
1. The cryo needs to be throttled for plasma processes by clicking PC High Vac Throttle under the ‘Vacuum’ tab then the gas line needs to be purged. This is done locally using the controllers in the PVD table itself. The top controller controls the gas flow while the bottom two control the plasma.

2. Use the dial on the gas controller to increase the flow rate to 30 sccm, hit enable on the controller and then under the ‘Deposition’ tab click Ion Source Valve to open the gas valve. The value in the box above the valve control gives the actual flow rate in the line. Once this value drops to zero the line is clean. Hit standby on the controller and turn off the ion source valve. Make sure that both manual gas valves are closed during this purge step, see the picture below.



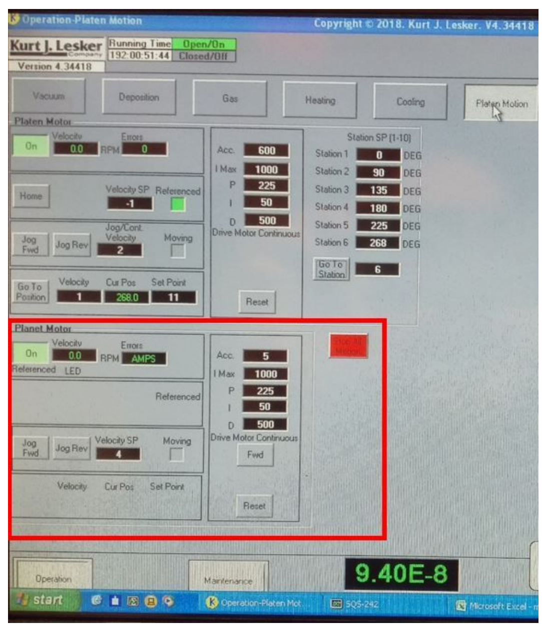
3. Turn on the oxygen **<u>at the tank</u>** in the other room and make sure that all the gas valves are oriented correctly to allow oxygen to flow to the PVD. Open the oxygen valve on the hardline mounted to the PVD table (red circle), click Ion Source Valve on the computer and set the flow rate using the dial back down to 3 sccm. The ‘standby’ button on the controller stops the gas flow. Close the ion source valve. Make sure that the ion source gas valve is closed (blue).





4. Before starting the plasma clean make sure the sample is facing away from the plasma position (position 2) and that the cryo is throttled by clicking PC High Vac Throttle under the ‘Vacuum’ tab.

5. Close any and all window shutters, turn off the main chamber light if necessary and enable the gas flow on the local controller by clicking enable. Also make sure that the plasma source shutter is manually closed, this is the black knob on the left side of the PVD.



6. Open the ion source valve and start the stage rotation under the ‘Platen Motion’ tab by selecting the Fwd button in the planet motion section. Then turn on the two plasma power supplies. These are the two units below the local gas controller, turn on the middle unit first, then the bottom one. The plasma control is done through these controllers **not** via the computer.

7. Briefly move one of the window shutters to check that a plasma is in fact present before moving the stage to position 2 under the ‘Platen Motion’ tab.

8. Once the stage as moved fully into position 2, open the manual shutter and wait for 30 seconds, then close the shutter. Hit standby on the plasma power supplies and confirm they are off on the ‘Deposition’ tab, to the left of the ion source valve. Turn off the gas at the controller as well as the ion source valve.

9. The chamber needs to be cleaned of oxygen before further use. Under the ‘Gas’ tab flow 15 sccm of argon, click Gas Injection and then open the cryo throttle by clicking PC High Vac

Throttle button. Allow argon to flow for 30 seconds before setting flow rate to zero and clicking Gas Injection.

10. At this point there will still be oxygen floating around inside the chamber. To help remove this oxygen and reduce the pressure, e-beam some titanium with your sample pointed away until you reach a reasonable dep rate. At this point the pressure should drop and stabilize, indicating the titanium has bonded with most of the oxygen to form titanium dioxide.

VENTING THE PRIMARY CHAMBER

Venting the primary chamber is required for changing the e-beam emitter or swapping crucibles in addition to many other maintenance procedures. These steps are typically done the day/night before e-beam depositions to give the chamber time to pump down to the appropriate pressure.

NOTE: **ALWAYS** use gloves when opening and handling anything in the primary chamber as any water and oil from your skin can greatly affect the environment inside the chamber.

1. In order to open the e-beam emitter chamber the main chamber must be vented, this is done with nitrogen. Make sure the nitrogen is turned on and under the 'Vacuum' tab click PC High Vac Valve to close the cryo and then PC Vent Valve to vent the primary chamber.

2. Once the chamber is vented, turn off the power at the wall with the large lever on the small breaker. It is the small breaker mounted highest on the wall. Once the power is off, carefully pull out the e-beam rack making sure the cables are away from the pinch points and that the e-beam shutter is not hitting the chamber.

3. Perform maintenance as needed and for the e-beam chamber be careful not to pinch the cables. Make note of any changes on the whiteboard.

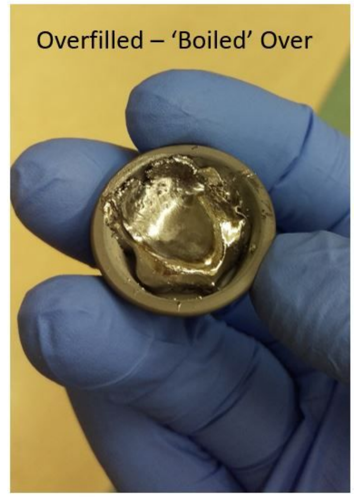
4. Begin the pump down process by venting the LL and then opening the LL Iso Valve. Then turn on the rough pump and open the Turbo Back valve under the 'Vacuum' tab. In order to decrease the pump down time, flip the rough pump ballast dial to position II. This allows more air to flow through the pump, increasing the pumping speed. Wait until the pressure in the main chamber reaches 10^{-1} torr. Once at 10^{-1} torr, flip the ballast dial back to 0 and flow 3 sccm of argon for 2 minutes before clicking LL Turbo Pump to start the turbo pump. Turn off the argon and wait until the chamber reaches 10^{-6} torr. Once at 10^{-6} torr click PC High Vac Valve to open the cyro to the primary chamber. Wait approximately 5 minutes before closing the LL Iso Valve and allowing the chamber to pump down to 10^{-8} torr overnight.

Alternative. Start rough pump at ballast position II. Once at 10-1 torr, flip to ballast position 0 and turn on the turbo. When the turbo speed reaches 100%, flow 2 sccm of argon for 10 minutes. If the pressure is above 5x10-4 torr after this period, wait until it reaches this point before continuing. Once at 5x10-4 torr, open the cryo and leave for 15 minutes, then close the load lock and wait an additional 5 minutes. After 5 min. Turn off Turbo, Close Turbo Back valve and turn off the rough pump. Leave the cryo to pump on the main chamber.

5. Once the LL Iso Valve is closed turn of the Turbo, Turbo Iso Valve, and the Rough Pump.

E-BEAM CRUCIBLE FILLING

Fill crucibles roughly 60-80%. Too much metal can flow up and over the walls resulting in a thermal path where the heat/energy of the e-beam is directed towards the hearth, away from the melt.

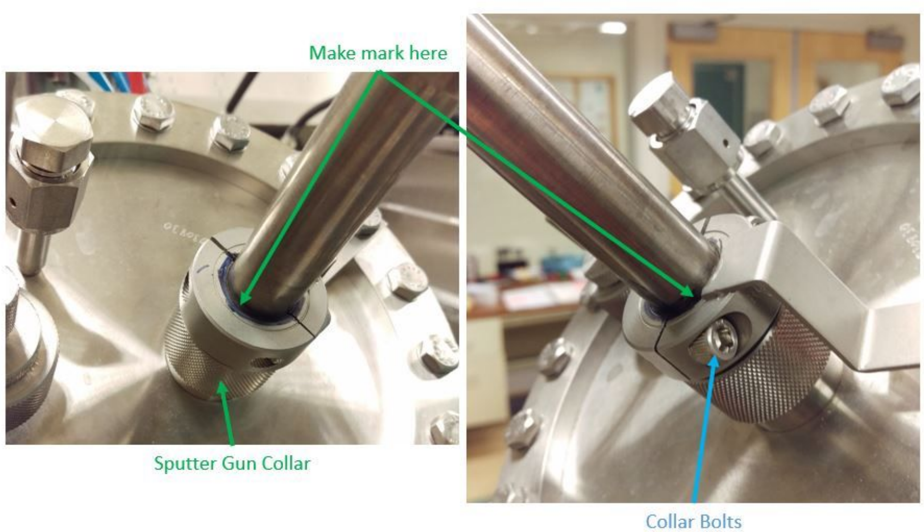


Aluminum 'wets' very easily and can creep up the sides of the crucible, fill roughly 60% to prevent/reduce this.

SPUTTER TARGET SWAP

Sputter target changing requires venting and opening the primary chamber. Excessive arc-ing or the inability to generate and sustain a plasma are indications a target change could be required.

1. Once the chamber is vented, unscrew the shutter control collar for the target to be changed and make a note of the mark on the target holder shaft. This mark should be just above the collar clamp on the shaft (green arrow).



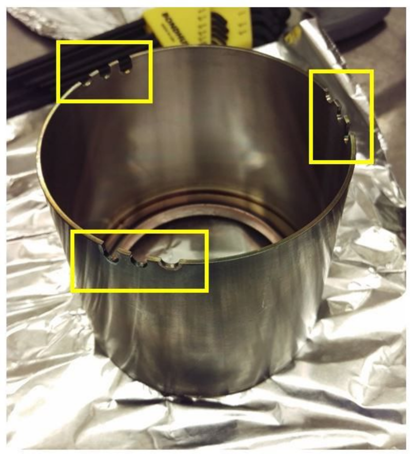
2. Hold the shaft in place and slowly

loosen the 3/16” allen bolts on the collar (blue box). Be careful as once the bolt has been loosened the shutter will start to fall into the chamber, hold it in place. Once the bolts are loose, slowly lower the shaft into the primary chamber

3. Once the target assembly has been lowered, remove the three allen bolts that secure the dark-space shield (DSS) and remove. Next remove the four flat head screws that hold the target retaining ring in place. The ring as well as spacers (depending on the target) will drop down, make sure they do not fall into the chamber. Make note of how many spacers are used for the given target and replace with the same number. If using a new target, consult the whiteboard on the wall to determine how many spacers and the DSS position for a given target thickness.

DSS Mounted w/ Target

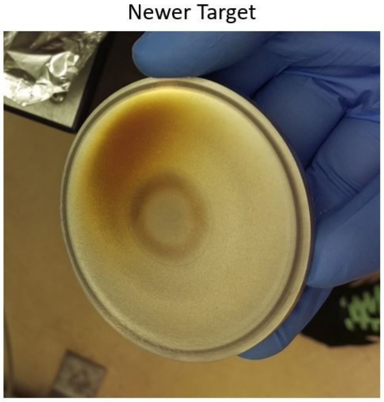
DSS Removed



Target, Spacer Rings, Retaining Ring

4. The target is held on the holder with a magnet, simply slide the target off to the side to remove. It will seem very tough to slide, it just needs to be muscled out. You can do it.

Newer Target



Used Target – Racetrack’d

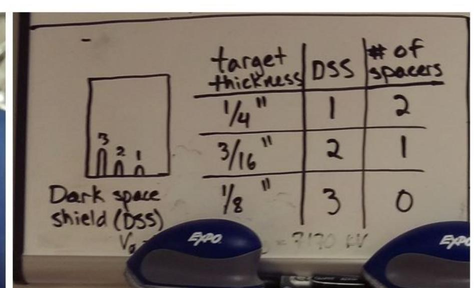


Chart for # of spacers and DSS position based on target thickness.

NOTE: Installation is the reverse of removal with a few notes. Make sure the DSS is installed into the correct bolt slots by checking the whiteboard (picture above) and that it is tight and secure. One of the main reasons for arc-ing during sputter deposition is flakes of metal that come into contact with the DSS and target, causing a short.

5. Reattach the correct number of spacers (based on target thickness → whiteboard) and the retaining ring to the target and then bolt the DSS in place. Once the DSS is installed, check the resistance with the multimeter between the DSS and target. This value should be infinite, and the multimeter with make a noise. If it does not, then check and possibly re-seat the DSS.

6. Close the shutter manually and make sure that it is aligned correctly. It needs to sufficiently block the target during deposition but also stay out of the way of other components in the chamber. This is especially important for target 5 as the shutter is very close to not only the load lock but sputter target 4 also.

NOTE: When swapping in a used target that has been sitting **OR** after the chamber has been vented or sat at atmosphere make sure to run the sputter for 1 minute or two with the shutter closed, and then another minute or two with the shutter open **BEFORE** you deposit on a sample. Oxides form very easily on the targets and can cause issues with the deposition.

GENERAL CHAMBER MAINTENANCE

Asher filament: Filament used to generate plasmas. Move the shutter by hand and unbolt the filament by hand (red boxes), install is reverse of removal. Be sure to not bend or break the new filament by overtightening. **Replace roughly every 20 plasmas** (filaments are cheap).

Quartz Crystal Monitor (QCM): Crystal used to monitor and determine the thickness of the deposited films. QCM is held via a friction fit, press out the old QCM from the rear and press the new one in on the front. QCM holder itself can be removed, it simply clips to the bracket. There are two types used, the e-beam uses an alloy QCM while the sputter uses a gold QCM. **Replace once remaining life reaches 80%** (estimated, use best judgement).

TOOLING FACTOR ADJUSTMENT

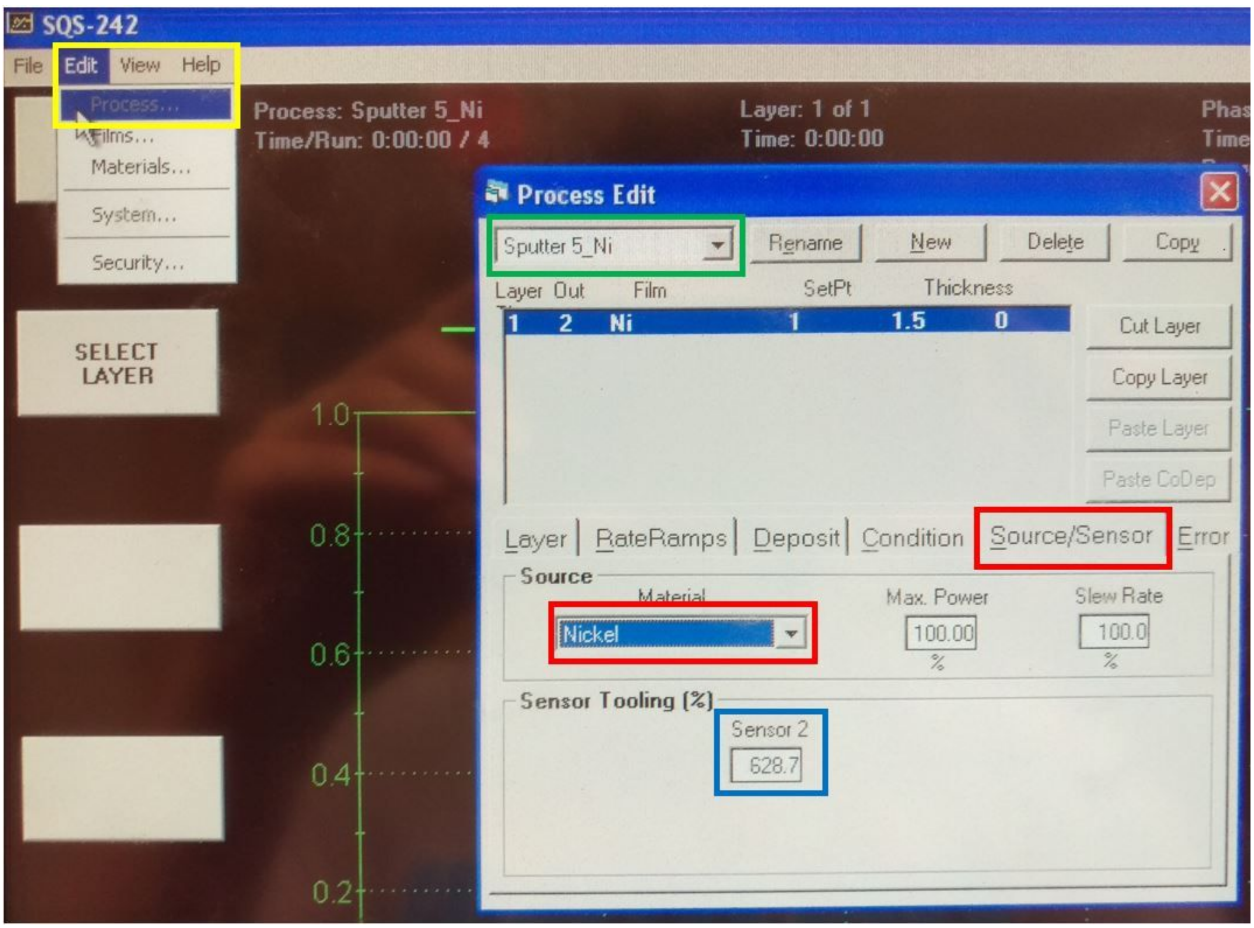
Changing the tooling factor is required when using a crucible of new material, after adding more material to a crucible, and swapping or re-orientating a sputter target.

1. Place either a piece of tape (vac-safe only) or clamp a wafer on top of the wafer to be deposited. Load the wafer in the chamber.

2. Deposit some amount of material onto the wafer and record the value displayed on the computer.

3. Remove the wafer from the chamber as well as the tape or second wafer from its surface. This leaves a clean step at the material-bare wafer interface.

4. Using profilometry, electron microscopy or other method the average step height (ASH) is determined. This is the deposited material's thickness.



5. In the SQS-242 software select 'Edit' then 'Process' from the upper toolbar (yellow box). Select the correct process from the drop down menu (green box) and make sure the 'Source/Sensor' tab is selected and that the correct material is selected (red boxes). Using the current value for the tooling factor (blue box), the recorded thickness and the actual thickness (via profilometry or EM) the new tooling factor is determined using the equation below. Simply input the new tooling factor into the blue box and close the window.

$$\frac{Measured\ Thickness}{Sensor\ Thickness} = \frac{Correct\ Tooling}{Current\ Tooling}$$

CRYO REGENERATION

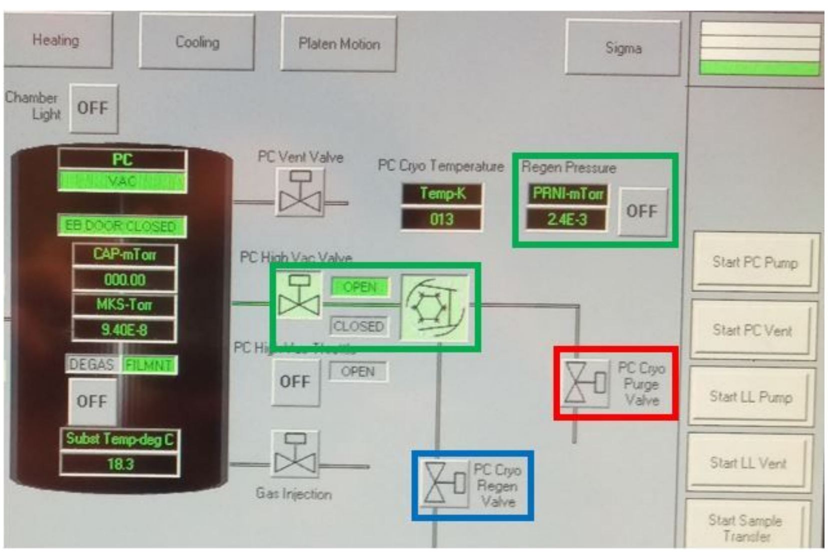
Regenerating the cryo is done when the temperature gets close or exceeds 13 K. The process involves warming to room temperature and allowing the carbon mass to off-gas all the 'stuff' trapped inside. This is an all-day process and should be done roughly every 6 months pending the amount of depositions performed. Just keep an eye on the temperature. This entire process takes approximately 5 hours, plan accordingly.

1. Turn on the nitrogen in the other room and make sure the turbo pump is off, the LL Turbo Back Valve is closed and the rough pump is off.

NOTE: It will take roughly 1000 psi of nitrogen to complete the regen procedure, make sure **BEFORE STARTING** that the tank has enough gas.

2. To start the cryo warming, close the cryo using the PC High Vac Valve and turn it off using the buttons on the program (green box in the picture below). Turn on the Regen Pressure gauge in the top right corner of the 'Vacuum' tab (green box). This gives the pressure inside the cryo itself. *(Warming from 13K to room temperature takes approximately **40 minutes**)*

3. Depending on how long the cryo has been at room temperature and how dirty it may be, the following two steps should be repeated 2-3 times in order to pump as much out of the cryo as possible.



4. Once the temperature goes above 100 K, open the PC Cryo Purge Valve (red box). There is an interlock that prevents the purge from being opened below 100 K. After the valve is opened you will hear the cryo venting with nitrogen. Once it reaches atmosphere, roughly 8-9x10^5 mtorr, it will make a popping noise. At this point turn off the PC Cryo Purge Valve.

NOTE: The gauges on this system all read in torr, **<u>EXCEPT</u>** the Regen Pressure gauge which reads in millitorr. For atmosphere this is roughly 800,000-900,000 mtorr (800-900 torr), atm is roughly 760 torr.

5. Turn on the rough pump and turn the dial on the pump to ballast position 1. This allows more air to flow through the pump, increasing the pumping speed. Open the PC Cryo Regen Valve (blue box) and allow the rough pump to pump on the cryo until the Regen Pressure reading stabilizes (roughly). Once the pressure stabilizes, turn off the rough pump and close the Cryo Regen Valve. Open the Cryo Purge Valve, you will hear the cryo venting with nitrogen. Once it reaches atmosphere, roughly 8-9x10^5 mtorr, it will make a popping noise. At this point turn off the PC Cryo Purge Valve.

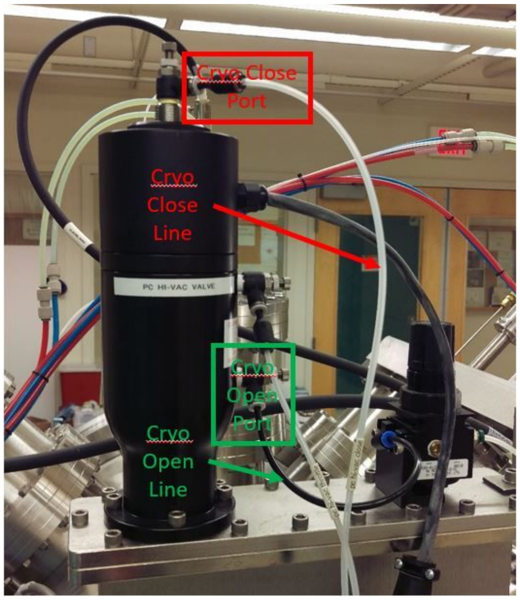
6. After repeating step 5 for the final time, open the PC Cryo Purge Valve and leave it open for 1 minute after the popping noise, (once it reaches atmosphere). After this period, close the purge valve, turn on the rough pump, turn the ballast dial back to position 0 and open the PC Cryo Regen Valve. *(Depending on how many times steps 4 and 5 are repeated this process should take approximately **30 minutes**)*

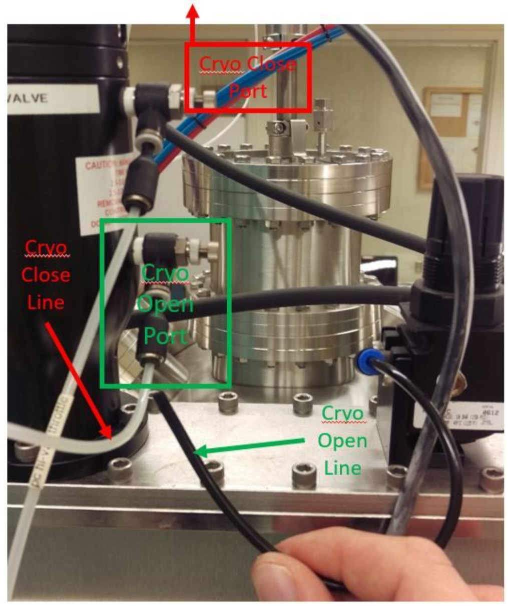
NOTE: At this point it is a waiting game for the cryo to pump down. It usually takes 1.5-2 hours but the longer it is able the pump, the better. If the rate of rise test (next step) is not within spec you will need to repeat steps 4-6 again or until it conforms to spec.

7. After the pumping period, close the PC Cryo Regen Valve and turn off the rough pump. Immediately record the Regen Pressure value and set a timer for 5 minutes. Record the pressure at 1 minute intervals as the pressure rises. The specifications need an average rise of 10 mtorr/min, with a total rise no more than 50 mtorr. If these specs aren't met, go back a repeat steps 4-6. If the rate is within spec, proceed to step 8.

8. Open the PC Cryo Purge Valve and vent the cryo, close the valve once it pops. Vent the primary chamber and the load lock as per usual, and then open the LL Iso Valve.

9. Go around to the back of the PVD table where the cryo gate valve control is (pictures below). Remove the cryo close gas line at the top of the valve control (red box) as well as the cryo open gas line connected to the lower-most port on the gate valve (black hose, green box), they are both press-fit collars, simply push the collar and pull out on the hose. Leave the cryo open hose loose and place the cryo close hose into the cryo open hose port, shown in the figure on the right. This fools the software and forces the gate valve open so it can be pumped down. Due to the design of our system everything is pumped through the load lock, necessitating everything to be open.





NOTE: This will throw an error on the software, do not be alarmed, this is expected.

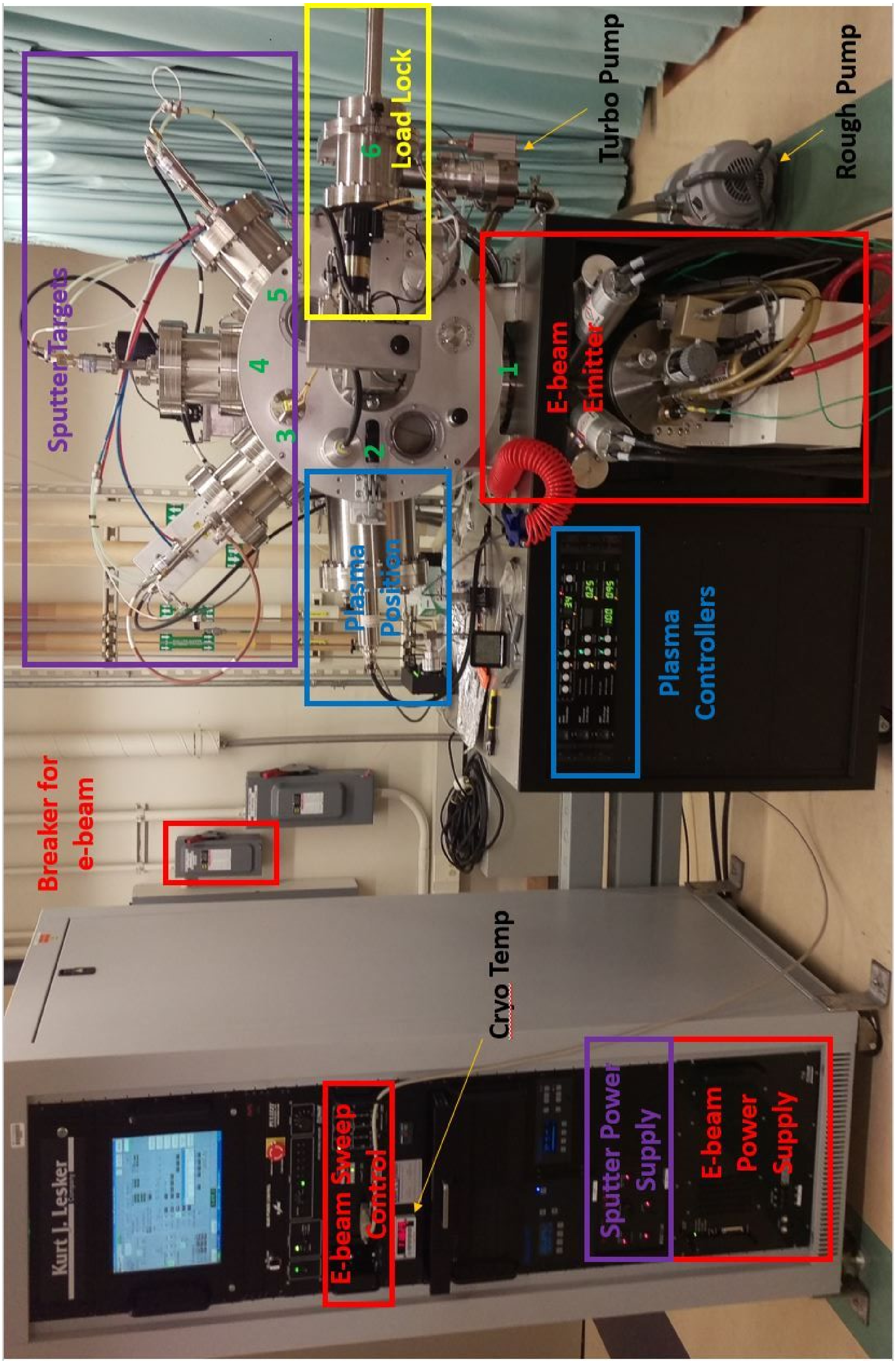
10. Turn the rough ballast dial to position 1 or 2, depending on how long you want the pump down to take, turn on the rough pump and open the LL Turbo Back Valve. Allow the system to pump to 10^{-1} torr, turn the ballast dial back to 0 and then turn on the turbo pump. *(**20 minutes**)*

11. Once the turbo has spun up to 100% speed, flow 3 sccm of argon for 5 minutes under the ‘Gas’ tab. This gets gases moving inside the chamber and should decrease the pump down time. After 5 minutes turn off the argon and allow the system to pump with the turbo and rough pumps. *(Pumping to 10-5 torr should take approximately **1 hour**)*

12. Once the pressure is at 10^{-5} torr turn on the cryo and observe that the temperature is actually decreasing. It should quickly drop to 270 K and then slow as it gets cooler. Once it reaches 270 K, swap the hoses to their original locations. It should take roughly 2 hours to cool down to 11 - 11.5 K. *(Cooling from ~285-290K down to 270K should take approximately **5 minutes**)*

13. Once at temperature, open the PC High Vac Valve and allow the cryo to pump on the primary chamber. After roughly 5 minutes, close the load lock, turn off the turbo pump, shut the LL Turbo Back Valve, turn off the rough pump and allow the cryo to pump on the primary chamber.

APPENDIX


Sputter Targets
Load Lock
Turbo Pump
Rough Pump
1
2
3
4
5
6
E-beam Emitter
Plasma Position
Plasma Controllers
Breaker for e-beam
Cryo Temp
Kurt J. Lesker
E-beam Sweep Control
Sputter Power Supply
E-beam Power Supply

E-Beam Shape

Good Beam Alignment

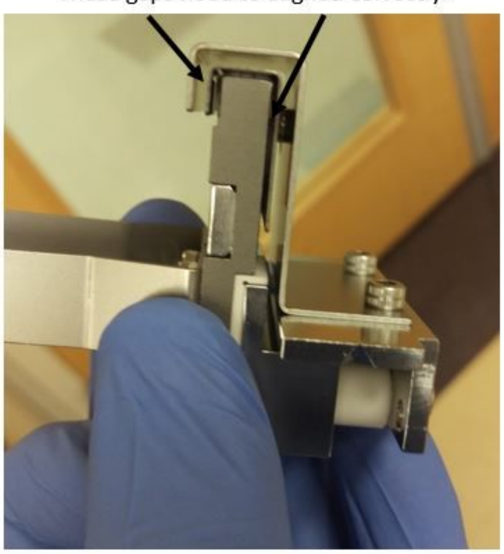
Beam Spread to Lower Right (left)

Beam Spread to Top (bottom)

Sampling of beam shapes for the e-beam. Due to the mirror and orientation of the crucibles within the PVD, directions are reversed when observing from outside the chamber. Actual directions are given in parentheses.

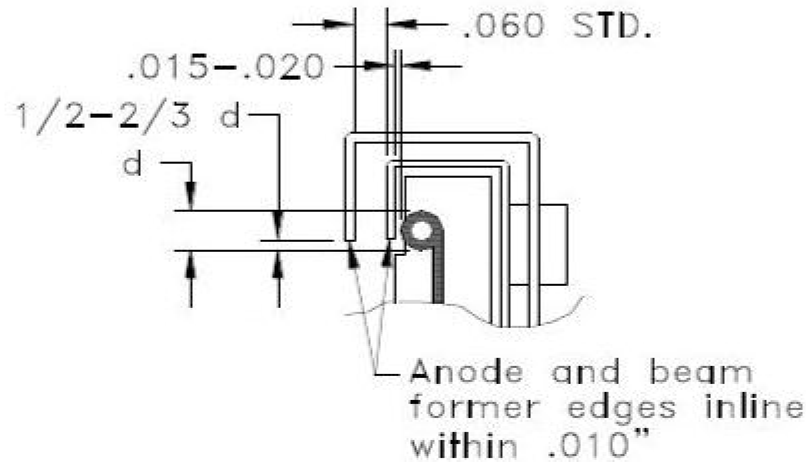
Emitter Alignment

These gaps need to aligned correctly.



The Filament should be in the center of the emitter

Copper leads need to be secure but not super tight. However the leads need to be crimped VERY well to the eyelets



Schematic of emitter gaps from the manufacturer (Telemark). The large gap, 0.060" can be reduced to 0.035"-0.040" for 5-8 kV. This increases current output

Metals On Hand - As of 9/13/19

E-Beam Pellets
- Al
- Au
- Co
- Cr
- Cu
- In - x2
- Mg
- Nb
- Ni
- Permalloy - Ni/Fe/Mo/Mn
- Ti

Sputter Targets
- Ca
- Co
- Cr
- Cu - x3
- Cu/SiO_2 - x2
- Fe
- In-Sn-O
- Mo
- Nb - x2
- Ni
- Ti
- TiN
- SiO_2

Part Information/Reference

Sputter Gun Model - TM03HS

QCMs - Both Infinicon 6 MHz

Sputter - Gold 008-010-G10

E-Beam - Alloy 750-679-G1