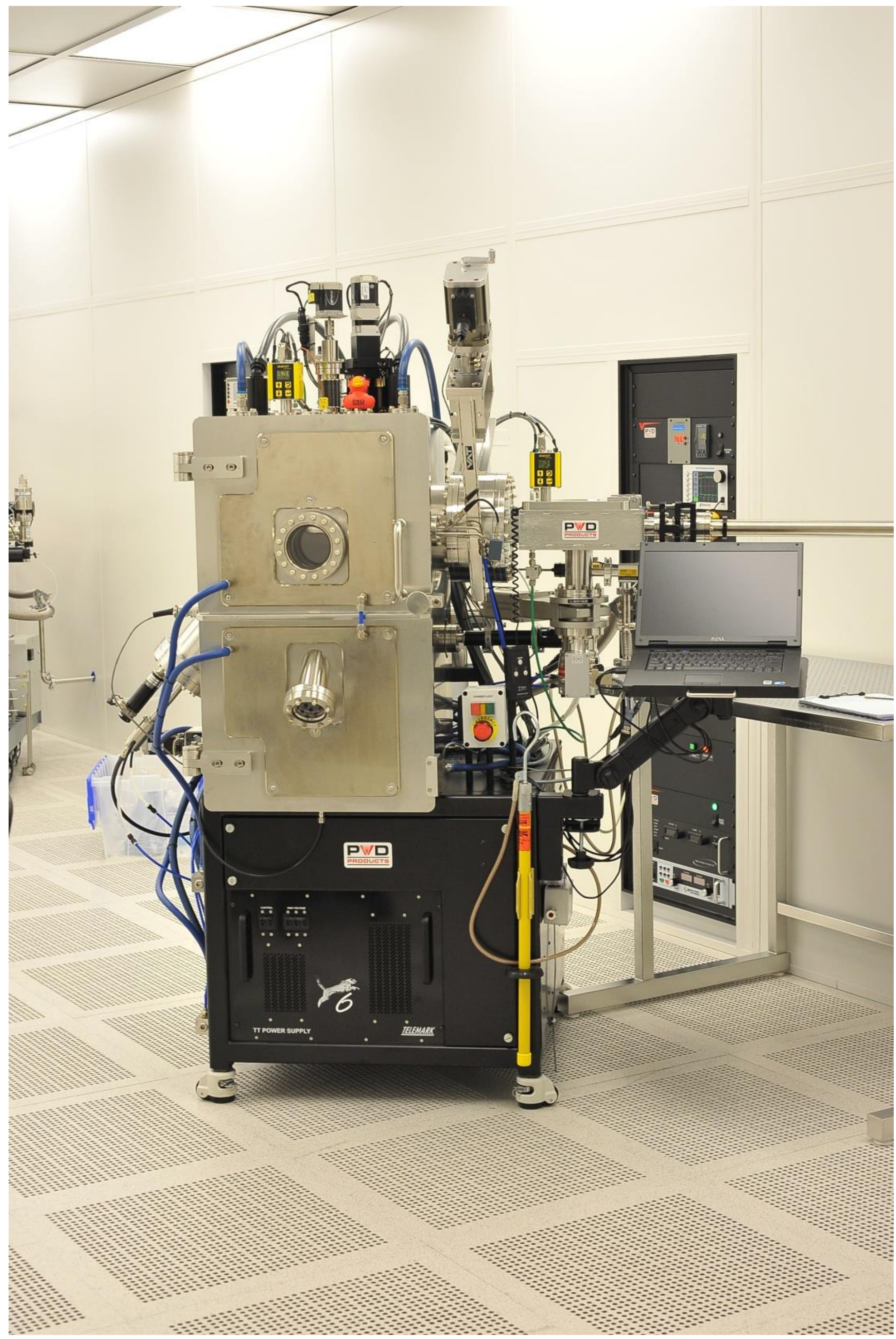


Standard Operating Procedure

For

PVD E-Beam (Version. 4)

Introduction

The PVD Electron-Beam Evaporator (E-Beam) thin film deposition machine uses a magnetically guided and collimated stream of electrons generated by applying high voltage to a large tungsten filament to heat, melt and vaporize materials in a vacuum chamber. This vaporized material streams out from the source and is deposited on all surfaces within line of sight in the chamber. At present only metals can be deposited using the PVD but in theory anything that can be melted and vaporized can be deposited. The E-beam has the advantage of being able to achieve high deposition rates and thicker films because of its large supply of deposition material in the chamber. The substrate receiving the film is far removed from the energies of the vapor source and so the deposition process is gentler on the sample. The process does, however, require a relatively high-quality vacuum and so extended pump-down times are required any time the main chamber is opened to change the source material.

Reservations are required at least 24 hours in advance for the machine and any new material not on the deposition list must be pre- approved. Deposition materials are typically loaded, refilled and cleaned for the day between 8 and 9 am; after materials loading, the chamber will typically be ready for use by 10 am.

Safety considerations

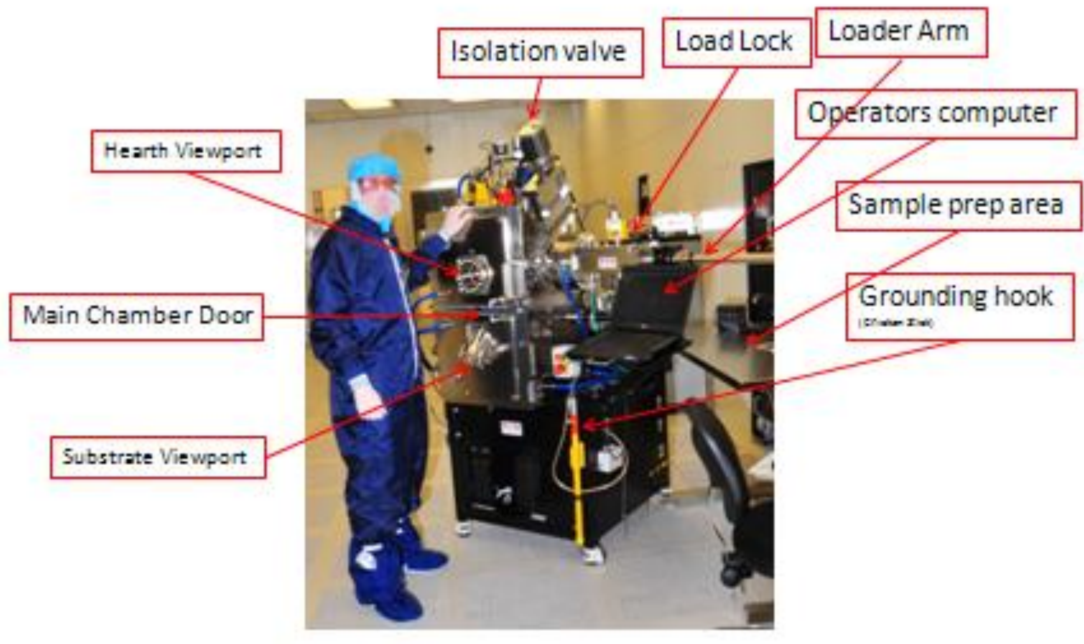
The e-beam is designed safely with interlocks that protect the user and machine, however if proper procedures are not followed there are risks to the user, the user's materials and the machine. There are open moving parts, heavy hatches, hot surfaces, and intensely bright light sources that can cause injury; and actions are required in specific sequence to not cause injury and or damage. It is the responsibility of the user to follow all current procedures and failure to do so will incur additional cost to the user should there be a mishap. Always use this SOP while processing as updates and changes in procedure will always be current here.

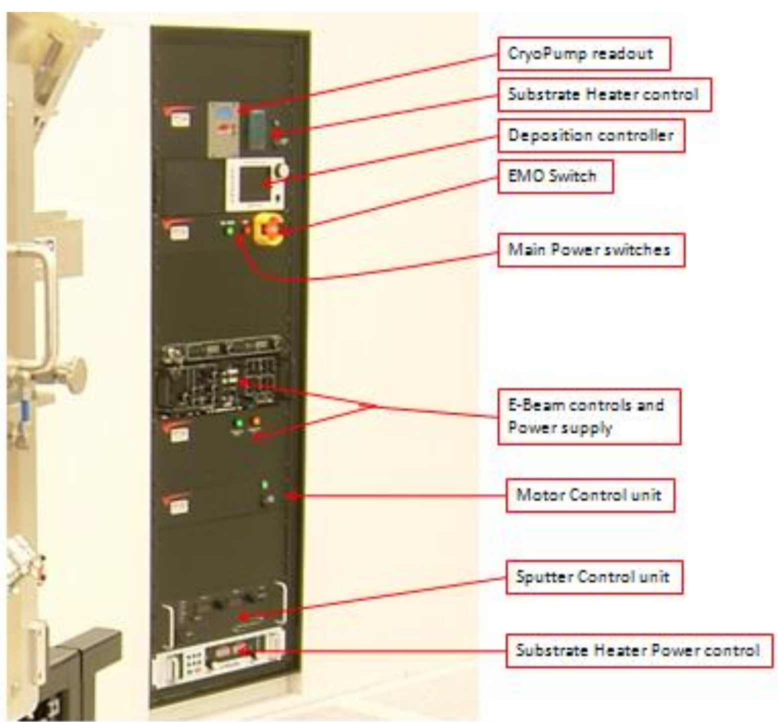
Altering the recipes beyond the evaporation run time can severely damage loaded devices and should only be done after consultation with staff. Alteration of any calibration settings, ramp times and crystal monitor settings can only be accomplished by staff.

After long intense runs the substrate can be hot; use caution when handling the substrate holder and the holder cannot be removed from the load-lock until it is below 50 C.

Visually confirm all crucible and shutter motions to ensure proper alignment and actuations; failure to do so could cause irreversible damage to your substrate.

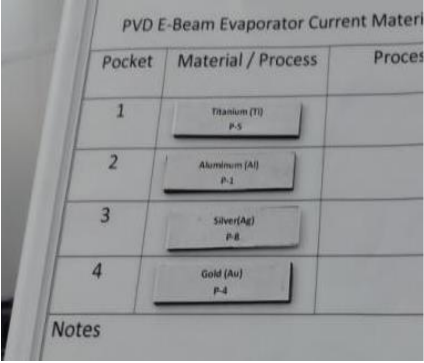
Machine "Anatomy"





Pre-operational Checks and Sign On:

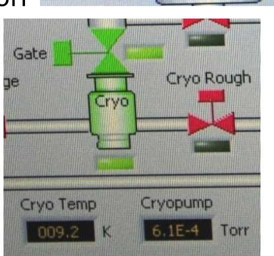
Step 1: Make sure that your deposition material has been loaded by checking the Load board.



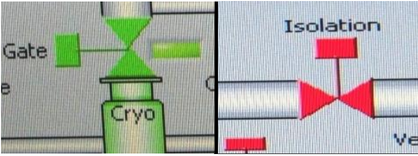
Step 2: Select the ***Chamber Tab*** on the PVD Laptop

1. Ensure that the Chamber Ion Gauge Vac $\leq 10^{-7}$ Torr

2. Ensure that the Load Lock Ion Gauge Vac $\leq 10^{-7}$ Torr



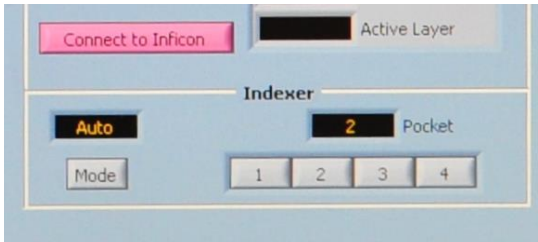
3. Ensure that the Chamber Cryo is on and is ≤ 15K
4. Ensure that the Chamber Cryo Gate Valve is open and Isolation is closed.



5. Check the Interlock lights on the left of the screen and make sure that 1, 2, 3, 5, 9, 10, and 11 are on and 4, 6, 7, and 8 are off.

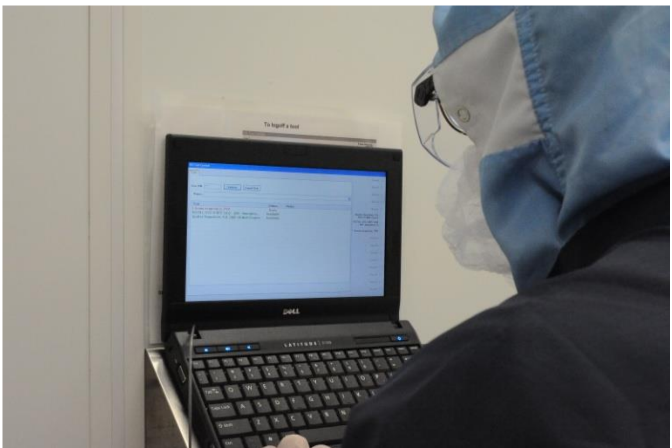
Select the ***Evaporation Tab***.

6. Ensure that the "Inficon SQC-310" mode box is set to Auto.



7. If not already connected Click on "Connect to Inficon" button.
8. The 'Rate through active layer boxes' should populate. Check that the frequency box and the % life box have data not "0" or characters. ⚠ If there is no data in these boxes, then the crystal monitor is not functioning properly and processes will not run.
9. Contact staff to fix this problem.

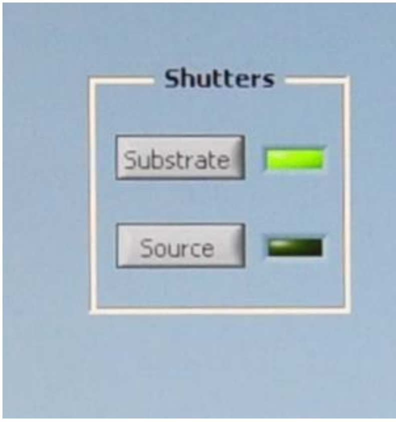
Step 3: Sign on to the TUMI; you will be required to enter in the total thicknesses of each of the materials you plan to deposit.



Step 4: Check that the interlock lights 4 and 7 on the left side of the screen are now lit.

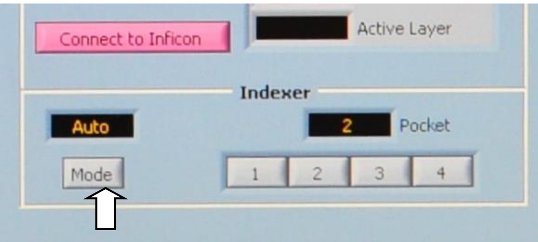
Step 5: Select ***Evaporation Tab***

1. Click on the source shutter button to open the shutter that covers the hearth, this will allow the chamber light to fully illuminate the hearth.

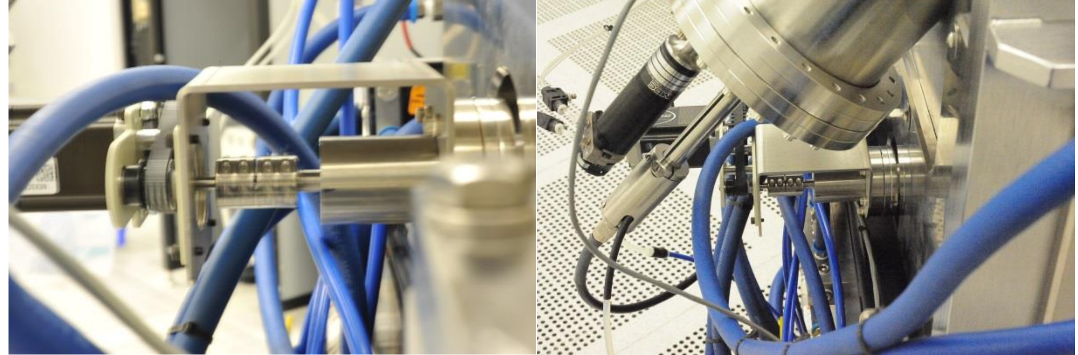


2. Turn on the chamber light so that the hearth can be seen more easily.

3. Click Indexer “Mode” button so it indicates manual.



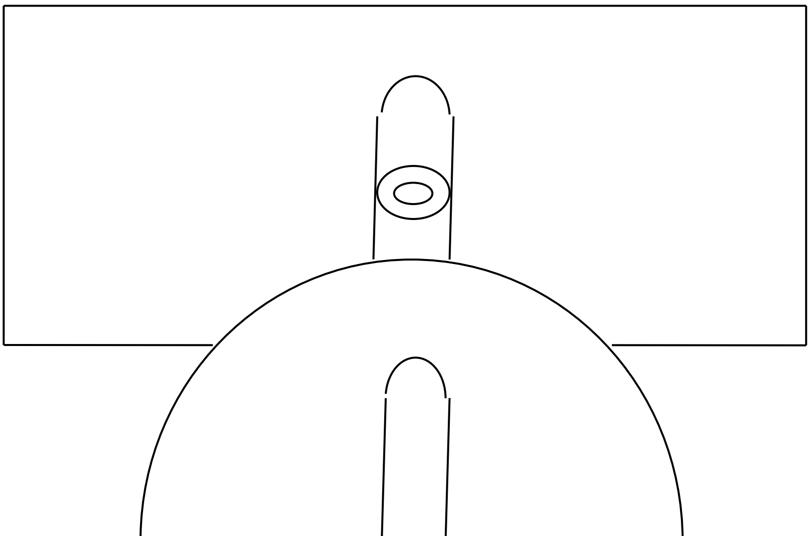
4. Click the “4” button and watch the motor on the left of the machine drive the hearth feed through to ensure it rotates completely.



It should rotate once for each pocket, check the rotary hearth in the chamber and confirm that it is moving from pocket to pocket. If it does not rotate properly then repeat several times alternating from #1 to #4.

⚠ If it fails after several attempts call staff for assistance. Watch the crucibles in the hearth rotate as well, if there is any doubt about whether the indexing is correct call staff.

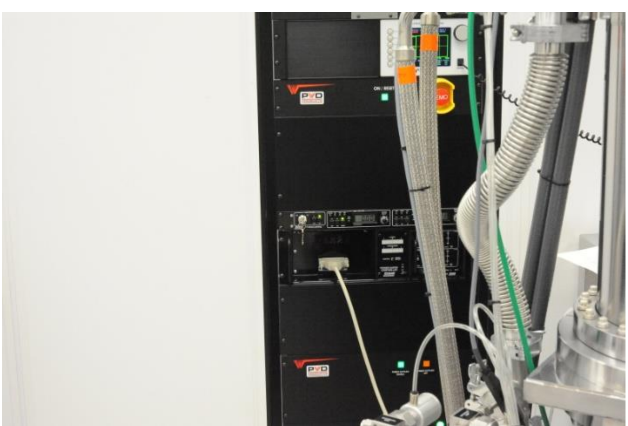
Diagram of Hearth Configuration



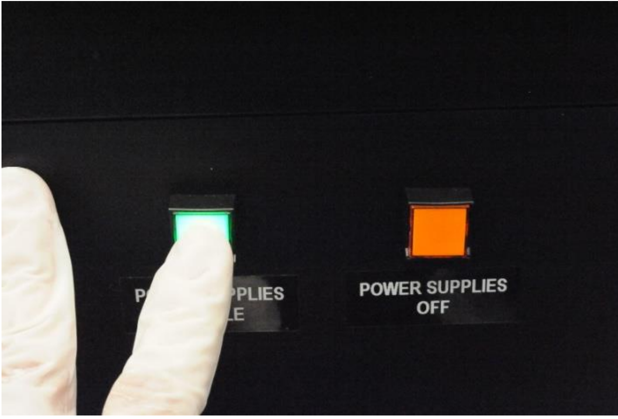
5. Change the Indexer mode back to "Auto" mode, the hearth will automatically return to the last pocket that was selected by the program.
6. Leave source open for XTak monitoring of film conditioning.

Powering up the machine:

<u>AT THE 'ELECTRONIC CONTROL RACK'</u>



Step 1: Press the “Power Supplies Enable” button (turns green).

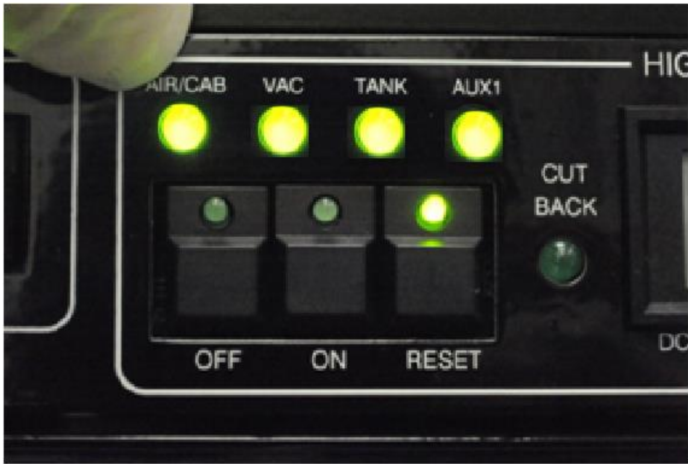


Step 2: Turn the “Main Power” key to on, then press the “On” button.

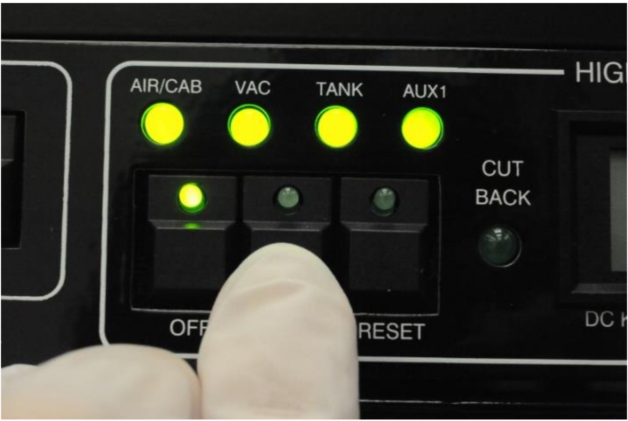
Step 3: In the “High Voltage” section press the “Reset” button.

Step 4: Check that the VAC/Tank/Aux 1 lights are lit. The Air/Cab interlock light will light after approximately 5 minutes. Wait for this to happen. You can work on sample prep while waiting.

(See page edge tab for this section)



Step 5: Once the Air/Cab light is on, press the "On" button. Do not press lightly; press firmly with no hesitation.



Step 6: Ensure the DC/Kilovolts readout shows voltage.⚠ Do not change the voltage.

<u>AT ‘SOURCE 1’ SECTION</u>

Step 7: Move Remote/Loc/Hand toggle to Loc.

Step 8: Press the “On” button.

Step 9: Move the Remote/Loc/Hand toggle to Remote.

<u>AT THE ‘POWER SUPPLY CONTROLLER’</u>

Step 10: Set toggle to “On”.

AT THE LAPTOP

Step 11: Check to ensure all the lights, except for the “Cryo High Temp”, on the left of the screen are on.

Start a log entry on the Excel workbook on the desktop.

There should be a single line used for each deposition run, a stack of three metals should use three lines!

Date	time	first name	last name	user number	material	FTF	Xtal life (%) From the Xtal	Set Dep rate (A/sec)	set thickness	indicated thickness	measured thickness
yy/mm/dd	1300	Nivla	Nedgo	4242	Al	100	100	5	10000	10010	15000

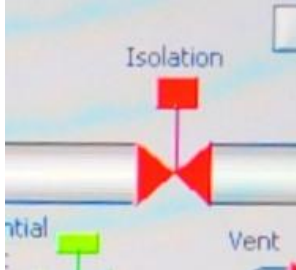
Loading the sample (See sample preparation guide at the end):

Step 1: Ensure that the loader handle is all the way against the right stop.

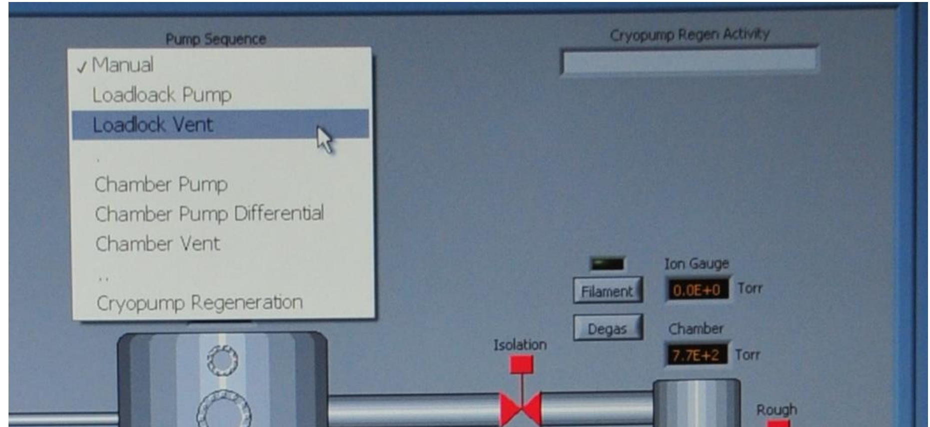
Look in the chamber and make sure that there is no sample or sample holder loaded on the substrate Z stage. If there is then it will have to be unloaded first. (see removing the sample on pg 22)

Step 2: Select the ***Chamber Tab***.

1. Ensure that the isolation valve is closed.



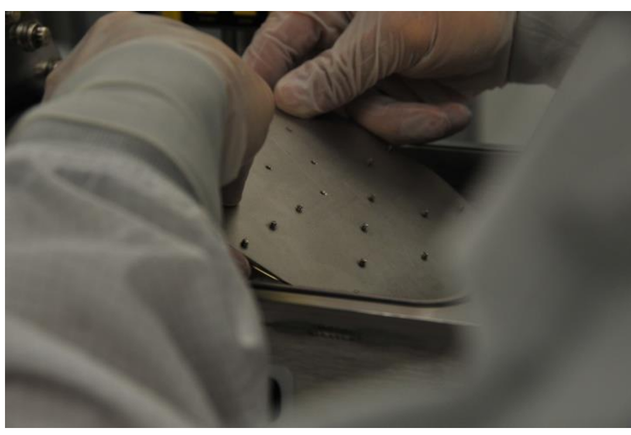
2. Select the "Pump Sequence" drop down menu and select "LoadLock Vent"



The loadlock is vented when the load lock chamber reads ~ 7.5 E+2 Torr.

Step 3: Once vented, manually open the loadlock door. Be careful, the door is heavy. DO NOT TOUCH THE O-RING.

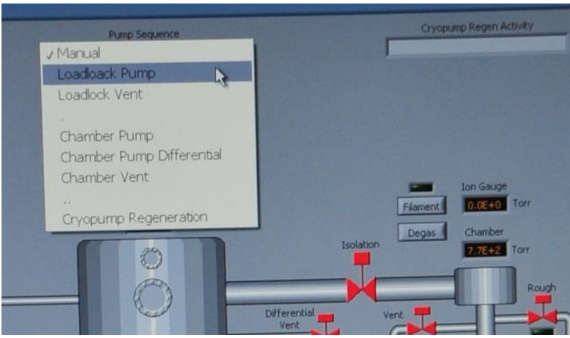
Step 4: Place the properly prepared (see sample prep section) sample holder on the loader arm; the posts on the loader arm (one on the right ant two on the left) should correspond with the slots on the sample carrier. Check to ensure there is there is no debris on the o-ring; if there is debris; clean the o-ring an isopropyl alcohol moistened wiper.



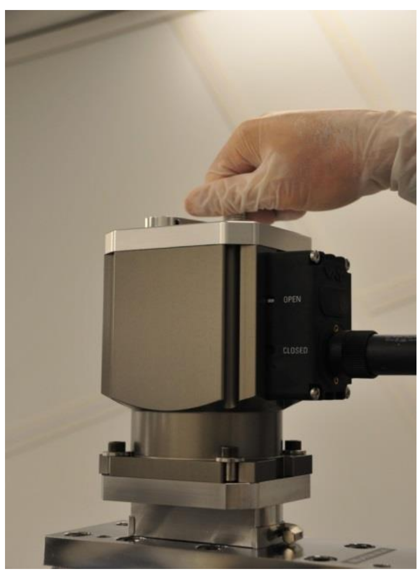
Step 5: Close lid gently.

Select loadlock pumpdown in “Pump Sequence Window” and wait for the load lock chamber ion gauge to reach ≤8.0 x 10^{-6} Torr. Caution (lower if the material is reactive with Oxygen)

(Reset rotations by turning on & off.)



Step 6: Open the manual isolation valve completely by turning the crank handle counterclockwise, slight resistance is felt followed by the sound of the gate snapping open, continue to turn the crank until the white bar appears in the “Open” Window.

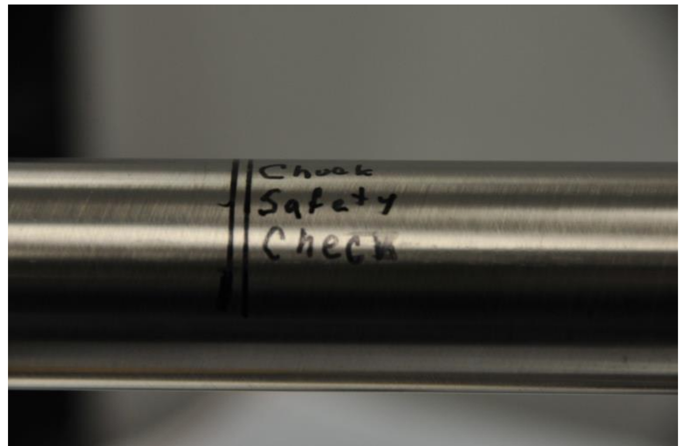


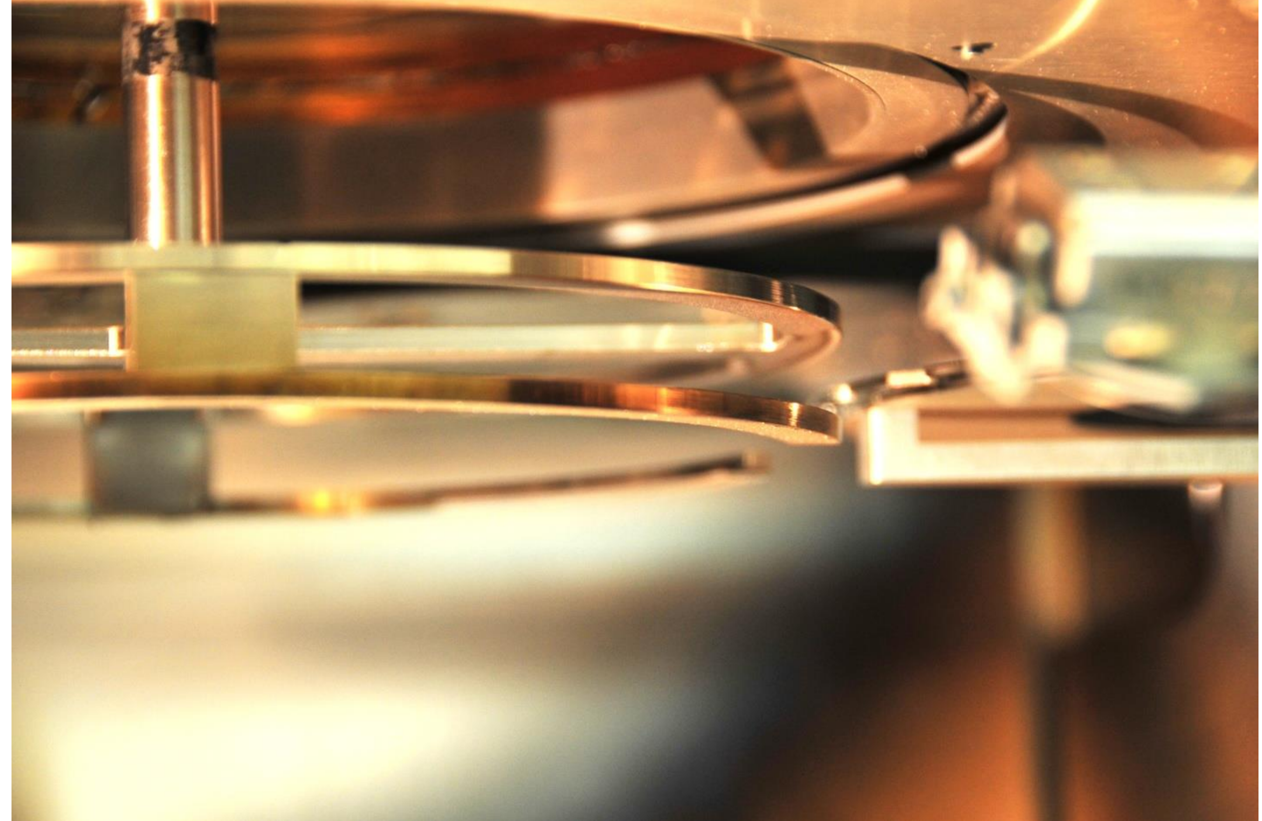
Step 7: Turn on chamber light if not already on.

Step 8: Click the ***Main Tab***.

Step 9: Visually check to see if the substrate stage is in the load position. If not, then lower the substrate Z stage by clicking the “Down” button.

Step 10: Slide the loader handle to the “Chuck Safety Check” line.


Safety
Check

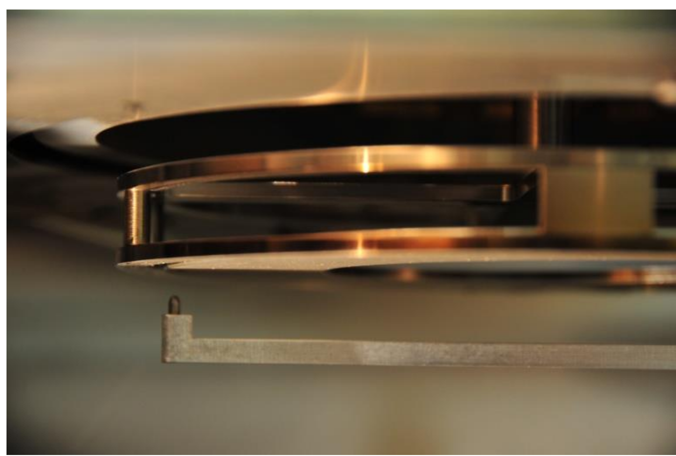


Step 11: Ensure that the sample holder will go between the substrate Z upper and lower rings as in the photo above. If it does not,

adjust the Substrate Z (in the Main tab) until proper alignment is obtained by inputting 2mm into the jog box and clicking the "Jog Up". Repeat if necessary until the proper alignment is reached. Be aware that more precision is required for the clamping small sample holder and or thicker samples. It may be that some samples may only be loaded manually.⚠ **During this operation, no substrate Z motion is permitted with the loader arm past the safety line.**

Step 12: Slowly insert the sample holder all the way against the stop. If you feel resistance, visually inspect the sample holder. If further adjustment is needed, return the loader arm to the "Safe" position and make the proper adjustments. DO NOT FORCE ANYTHING, IT SHOULD BE EASY AND FRICTION FREE MOTION.⚠ **If there is any mis-alignment or obstructions preventing a safe and proper load then stop return to safety line and call staff.** ⚠⚠⚠⚠ **If there is ANY resistace to the remoal of the arm, stop and call staff!!!**

Step 13: Press the "Up" button on the Substrate Z holder in the window and watch for the substrate holder to seat properly and clear the arm after picking up the sample holder. One can jog up as well I that is more comfortable. ⚠ Any time that the Z is in motion the cursor should be hovering over the STOP button so that motion can be stopped instantly if there is a problem.

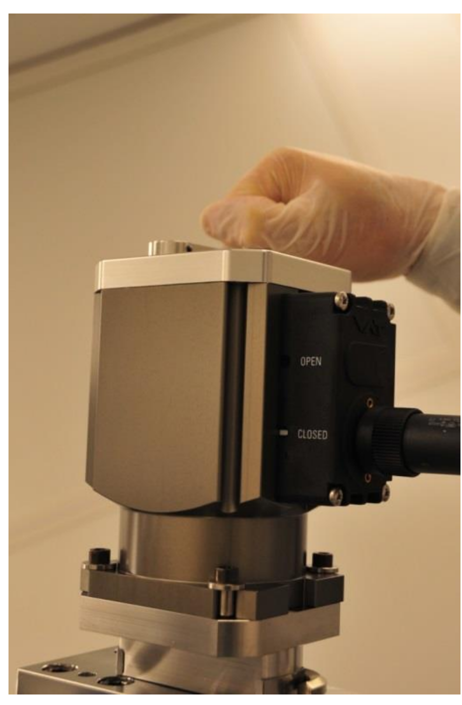


Step 14: Click ***Main Tab***

1. Ensure that "Z" is up (light will be on).

Step 15: Once clear, move the loader arm to the far right stop.

Step 16: Close the manual isolation valve by turning the crank clockwise. When nearly closed a slight resistance is felt during crank rotation followed by the sound of the gate snapping closed, a few more turns to resistance is all that is needed (The white bar appears in the "Closed" Window)



Running a Process:

Go to the chamber tab

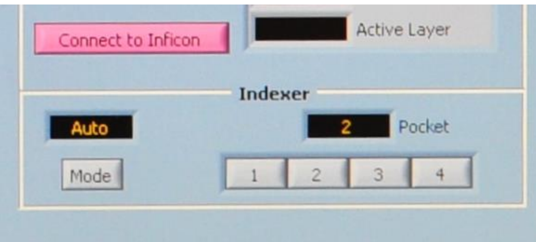
Step 1: Check that the substrate shutter is closed, and the source shutter is closed. Do so by checking for the light on the computer as well as visually through the chamber port hole. If the light is off, the shutter is closed.

Step 2: If rotation is not required, skip to Step 3. If you desire substrate rotation, go to the “Main” tab and enter the RPM set point you prefer (i.e. 10) and click “Rotate”.

If substrate heating is required now is when the heater would be started; please note that heating is Staff programmed and a request must be made at least 24 hours in advance. See the substrate heating section at the end of this document.

Step 3: Select the ***Evaporation Tab***.

1. Ensure that the “Inficon SQC-310” mode box is set to Auto.



2. Select proper process number (See Process Assignment Board)
3. Click on “Connect to Inficon” button if it is not already connected.
4. The ‘Rate through active layer boxes’ should be populated. Check that the frequency box and the % life box have data not ”0” or characters. ⚠ If there is no data in these boxes, then the crystal monitor is not functioning properly and the process will not run. Call staff for assistance.

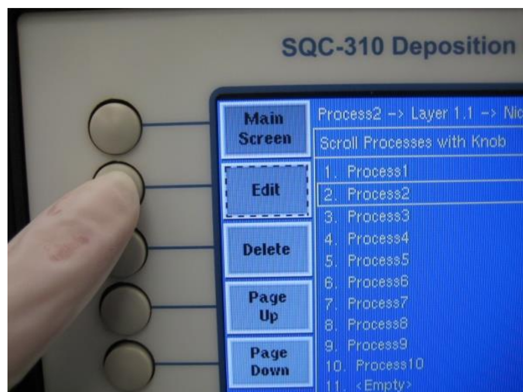
Step 4: Set the desired thickness as follows.

1. Go to ‘Electronics Control Rack’

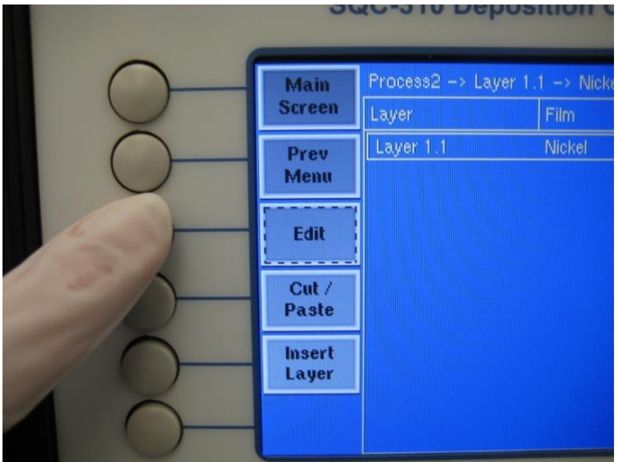
2. Go to SQC-310 deposition controller.

3. Press the “Next Menu” button until you see ‘Process Menu’ on the list.
4. Press the “Process Menu” button

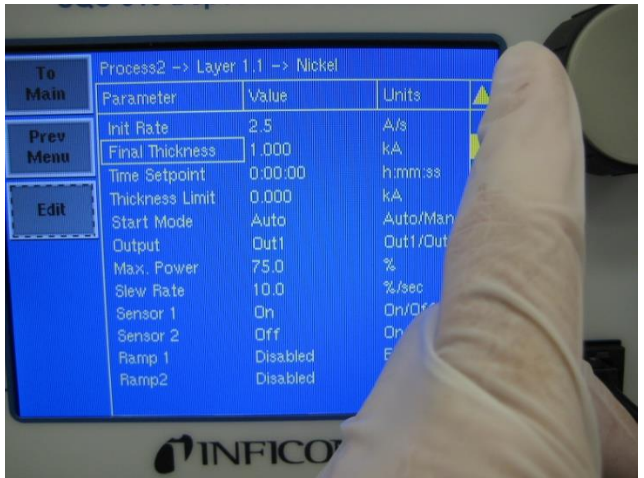
5. Rotate the selector knob until your process is selected.
6. Press the “Edit” button



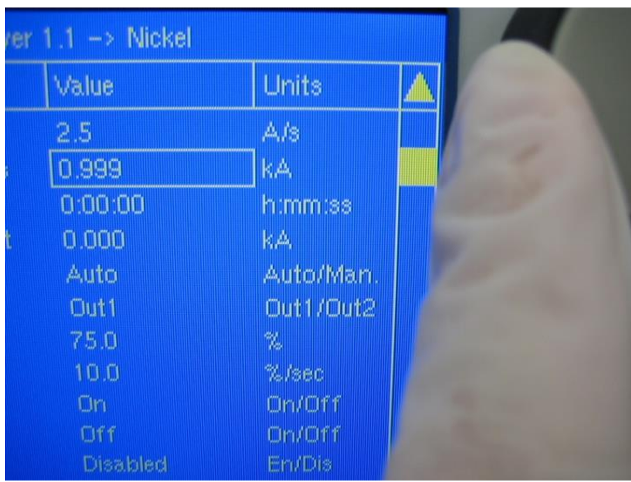
7. Check that the layer contains the requested deposition material on the display.
8. Select the layer with the knob.
9. Select “Edit”.



10. Rotate the process knob until ‘Final Thickness’ is selected.

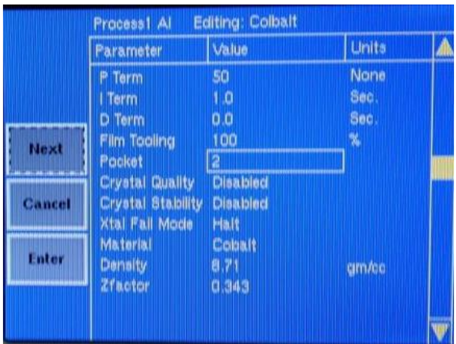


11. Select “Edit”.
12. Set final thickness by rotating selector knob to desired set point.



13. Press “Enter”.

14. If a different deposition rate is desired then it can be changed in this window, Dep rates over 5A/s are not allowed on any material. Usually the slower the rate the better the film.
⚠Discuss any changes to the “default” dep rate with staff prior to processing as there are many factors that affect the rate.
15. Press “To Main” button.
16. Press the main menu button until the process and film menu buttons are displayed, press the film menu then select each material to be deposited and confirm that each loaded material is designated with the correct pocket number as indicated on the load board.

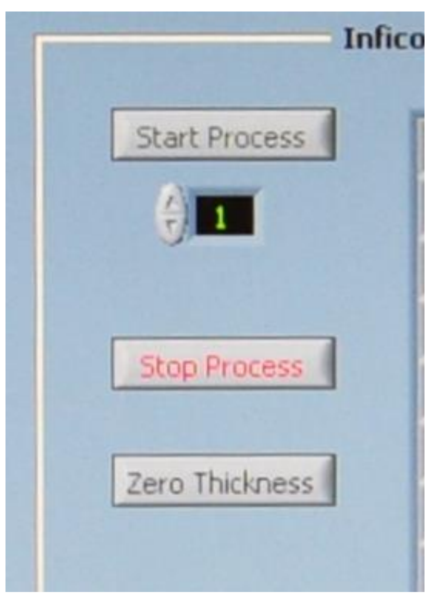


AT LAPTOP

Step 17: Choose the correct process number by pressing the arrows next to the process number window (the correct process is indicated on the load board for each material) then press “Start Process” button. NOTE: The Indexer will move to the proper pocket if not already selected. Then the process will start.

⚠ Turn on the chamber light and open source shutter so that the hearth is well illuminated, Watch the pockets move to the desired material. If I doubt that the proper pocket is not aligned properly in the window, then press “Stop Process” and call or staff assistance.

⚠ **The instrument must be attended at all times during deposition. User is responsible for watching the rate and confirming the beam has not drifted and the crucible material is still present in the crucible liner. Failure to do so could result in the instrument being extensively broken/present a hazard to users.**



Step 18: Press “Zero Thickness” button.

The process will proceed in the following manner. Indexing phase → Film conditioning phase →active evaporation phase → material cool down and be designated using the following shorthand so that one will be able to determine the total process time from beginning to end.

←→↗↘

↗ percent power / time → dwell time ↗ percent power / time → dwell time ↗ evaporation power |

Evaporation Å/sec | ↘percent power / time

IE:

↗ 10 / 5 → 5 ↗ 30/ 10 → 1 ↗ 32 | 5 | ↘ 0 / 5

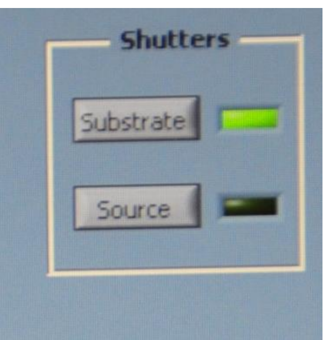
Will increase the power to 10 percent over a 5 minute period and hold it there for five minutes then will proceed to 30 percent power over the next 10 minutes and hold it there for 1 minute then a final ramp to the evaporation power set point at which time the shutters will open and the evaporation controller will vary the power to achieve rate setpoint, when the desired thickness is reached the shutters will close and the cool down will occur over the next 5 minutes.

The phase box will indicate 'Stopped'.

Removing the sample:

Step 1: Once the shutter closes after the deposition, select the ***Evaporation Tab***.

1. Ensure that the source shutter is closed and open the substrate shutter.



Step 2: Select the ***Chamber Tab***.

Step 3: Open the manual isolation valve.

Step 4: Move the loader arm all the way to the left against the stop.

Step 5; Select the ***Main Tab***

1. Select the "Down" button in the Substrate Z stage box.
2. Move the cursor over the stop button (Do Not Click Yet) and watch the substrate lower on to the loading arm. Watch to make sure the loader arm pins align and seat will with the substrate holder pin holes. If they do not click the "Stop" button and call for assistance.
3. If there are not issues, click stop once the sample holder is elevated by the loader arm to the center substrate z assembly.

Step 6: slide the loader arm all the way to the right against the stop. If ANY resistance or unusual friction is felt stop and visually check for proper adjustment and use the jog buttons to reposition the z holder BY SMALL STEPS until the sample holder if free of contact while removal.

Step 7: Close the manual isolation valve. (The white bar appears in the "Closed Window")

Step 8: Select the ***Chamber Tab***.

1. In the "Pump Sequence" drop down, select Loadlock Vent.

Step 9: Once the chamber pressure reaches ~7.0E+2, carefully lift the loadlock door and remove sample.

Step 10: Close the loadlock door.

Step 11: In the "Pump Sequence" dropdown, select Loadlock Pumpdown.

Shutting down the machine:

AT THE 'POWER SUPPLY CONTROLLER'

Step 1: Set toggle to "off".

AT THE 'SOURCE 1' SECTION

Step 2: Move Remote/Loc/Hand toggle to Loc.

Step 3: Press the "Off" button.

AT THE 'HIGH VOLTAGE' SECTION

Step 4: Press the "Off" button.

AT THE 'MAIN POWER' SECTION

Step 5: press the "OFF" button, then turn the "Main Power" key to the off position.

Step 6: Press the "Power Supplies Off" button.

Step 6: Log off TUMI, making any notes that you feel are appropriate.

Wafer and sample prep

For whole 4- and 6-inch wafers there are dedicated holders and the wafer will just sit in the depression.

For small pieces there is a holder with multiple screw holes and clamps so that many samples may be loaded on one carrier. A word of caution about the clamps on the small sample carrier; if the deposition is going to be long and intense so that the sample holder heats up or if the substrate heater is used there is the possibility that the clamps will loosen and drop the sample, in this case use multiple clamps of consult with staff. If a sample drops into the crucible and contaminates it then the user will be charged for the entire crucible of material. The small sample holder will not be used if there are any open holes; make sure that uncovered screw holes have a screw in them. For alternate methods of securing samples in the machine consult with staff.

Fresh clear wafers and samples (No pattern clear substrate) should be thoroughly clean with the oxide stripped if appropriate. Samples that are post lithography should be well developed and residue free in the clear areas and, ideally, be undercut if liftoff is planned after deposition. It is wise to stop by the Asher and O2 clean the cleared features just before loading, be careful not to over clean, 10 seconds should do to remove any residue and over cleaning can remove too much PR and change the pattern.

Sample holders can be made from 4-inch wafers using the dicing machine and then loaded on the 4" wafer holder; custom holders must be designed with staff consultation and approval.

Substrate Heating

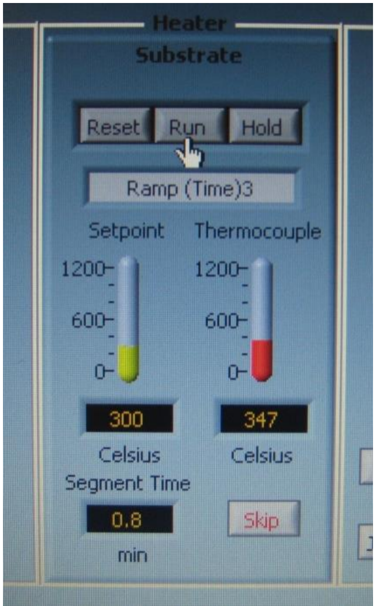
Note: Substrate heating must be requested 24 hours in advance and all samples, materials and mounting methods must be approved by staff. It is a staff programmed procedure that is initiated by the user at the appropriate time. There may be times, depending on the condition of the chamber that staff will require that a heated process be postponed until after a thorough chamber clean.

Substrate heating process:

Step 1: At the electronics rack press the PWR switch on the Heater Controller and allow it to initialize to the Standby mode.

Step 2: Press the Start button so that the **POWER** indicator lights.

Step 3: At the laptop, select the Main tab and click on the run button under Heater Substrate.



The substrate heater will go through a preprogrammed heater ramping routine to the requested temperature. Wait for the temperature to stabilize at set point before proceeding with the deposit (return to Step 3)

Step 4: After the deposition is finished and the shutter is closed return to the Heater Substrate window and press Skip; the heater will go through a pre-programmed cool down.

Step 5: The Heated sample will not be removed until it has cooled down to less than 50 C. the progress of the cooling can be monitored on the laptop or the electronic rack.

Step 6: After cooldown go to the "Removing Sample" section