LC Technologies Thermal Evaporator

Standard Operating Procedure

Faculty Supervisor: Prof. Robert White, Mechanical Engineering (x72210)
Safety Office: Peter Nowak x73246 (Just dial this directly on any campus phone.)
(617)627-3246 (From off-campus or from a cell phone)
Tufts Emergency Medical Services are at x66911.

Revised: February 25, 2020

Warnings:

The tool uses high power and low vacuum during deposition. Do not attempt to open the chamber until the deposition cycle is complete.

Required Checkout:

You must be formally tested by the faculty supervisor before you may use this tool unsupervised. When you are first learning procedures, work with experienced lab users (students, post docs, faculty or staff) to become familiar with the tool before requesting a formal checkout procedure.

Notes:

- ***The evaporator will be left on (under vacuum). When you arrive at the tool look for a note and check in the log book or chamber to determine if someone else is pumping down and preparing to deposit before you vent.***
- ***If you arrive at the tool and it is off and at atmosphere please contact lab staff***
- ***High temperatures (over 1000 C potentially) occur during deposition. If you ramp up too fast you will crack the boats. If the boats are old and alloy with the deposition metal you will crack the boat. This is ok – nothing terrible happens – you just won't be able to deposit and will have to vent.***
- ***You must stay at the tool for the entire deposition in case a boat breaks you need to bring the power back down.***

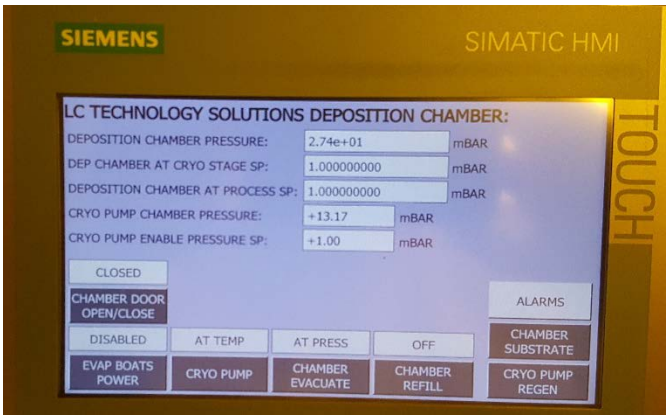
1.0 Material Requirements:

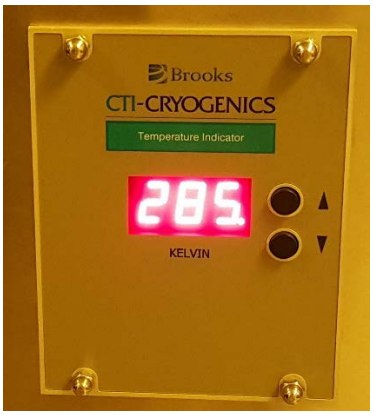
1.1 Equipment: substrate, wafer tweezers, metal targets, evaporation boats
1.2 Personal Protective Equipment: nitrile gloves, safety glasses
1.3 Chemicals: none
1.4 Facilities:
- **1.4.1** N_2 tank with pressure regulator set to 80 psi. This provides compressed nitrogen to run pneumatic elements in the tool including opening the door – with low N_2 pressure the door will not open.
- **1.4.2** VWR-1175PD recirculating chiller located below the desk behind the evaporator. Provides cooling for the cryo compressor and runs constantly.
- **1.4.3** Cryo compressor located to the left of the evaporator. Provides compression for the helium cycle in the cryo pump and runs constantly
- **1.4.4** Small recirculating chiller located atop the cryo compressor. This should be powered up before beginning deposition and powered down when the run is completed.
- **1.4.5** Electrical. The entire tool runs off 208 Volt /40 Amp /3 Phase, 5wire from a twist lock plug.

2.0 Procedure

Vent the tool (if you arrive and there is no sample in the chamber):

1. Check the front panel of the Siemens controller. Read off the base pressure. It should be in the 10^{-7} mbar range or below. Also check the cryo pump temperature. It should read below 20 Kelvin. If pressure or cryo temperature are high, contact lab staff and do not try to run.





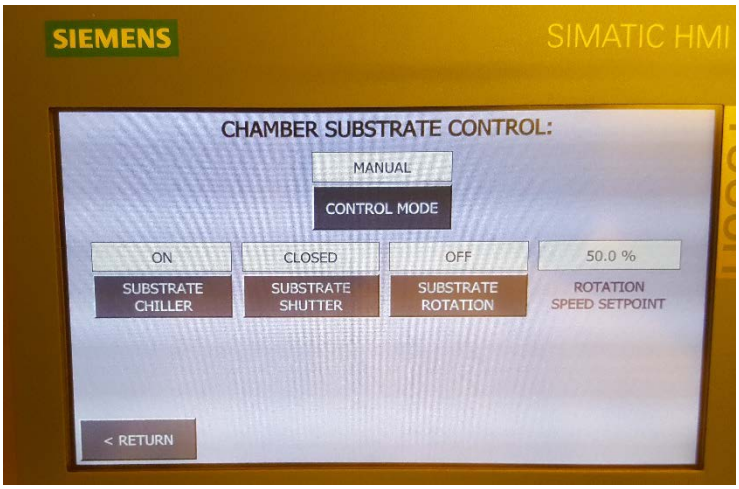
2. Push “chamber evacuate” on the touch screen. Readout changes from “at press” to “off”. Chamber pressure will rise to about $5 \cdot 10^{-5}$ mbar in about 30 seconds.
3. Push “chamber refill” on the touch screen. Readout changes from “off” to “on”. You will hear the sound of air flowing into the chamber. The pressure will rapidly rise to 1000 mbar.
4. Wait for a minute or two until the sound of flowing air completely stops.
5. Push “chamber refill” on the touch screen. Readout changes from “on” to “off”.
6. Make sure the “evap boats power” reads “disabled”. If it reads “enabled”, push the “evap boats power” button to disable the boat power. The door will only open if boat power is disable for safety.
7. Push “chamber door open/close”. The readout changes to “open”. The door should move away from the seal.
8. Slide the door to the left.

Check crystal:

1. There are two crystal thickness sensors in the tool, sensor 1 on the left and sensor 2 on the right.
2. To check the lifetime on the crystals, push “Sensor info…” on the Inficon controller.
3. If the crystal life for either sensor is below 50%, replace the crystal by twist/pulling the cylindrical housing out, popping out the old crystal, and popping in a new one from the box. Don’t touch the front surface of the crystal during this operation, to keep it clean.

Load samples and material:

1. Your samples can now be loaded onto the platen at the top of the chamber.
    a. Push “chamber/substrate” on the right side of the touch screen to bring up the chamber/substrate controls, which you see below.
    b. Push “substrate shutter” to open the substrate shutter.

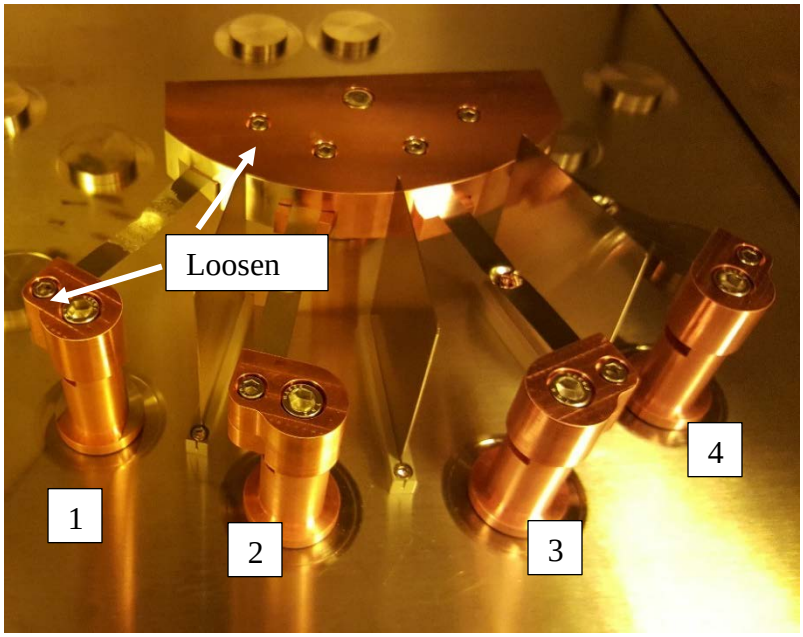


    c. If necessary, push “substrate rotation” to turn on/off substrate rotation to rotate the platen so you can easily access the holder.
    d. Loosen or remove the four thumb screws to remove the aluminum holder.
    e. Load your samples into the holder – currently there are three holders; one for 100 mm diameter wafers, one for 25 mm x 25 mm glass cover slips (Fisher part # 12540C), and one for standard 75 mm x 25 mm x 1.7 mm glass slides. Put the samples in so the face you want to sputter on is pointing down. The sample holders will hold your parts against the water cooled platen to keep them cool during deposition.

    f. Close the substrate shutter.

2. Now, it is time to load the materials for deposition. There are four sources in the tool at the bottom of the chamber, numbered #1 to #4 from left to right.
    a. If necessary, loosen the smaller screw using a hex key and replace the boat. The boats are 4” long, ½” wide metal boats. Typically these might be made of tungsten or molybdenum (see chart below). Note that boats can be re-used for multiple runs, but that

after some number of runs the boat may fail due to alloying with the deposited material. This is a bigger problem for high temperature deposition materials such as titanium. For Ti, I recommend using a new boat each run.



b. Add material to the boat. Typically we use 1/8" diameter, 1/8" long pellets. One pellet is typically enough material to deposit about 250 Å of material on the wafer (give or take), so plan accordingly. Do not over-fill the boat so the material can fall out. Try to lay the pellets down nicely so they have as much contact area as possible with the boat.

Source #	Material	Recommended Boat	Recommended Material
1	*Aluminum*	TUNGSTEN BOAT SOURCE, 5/PKG, 4" L X 1/2" W X 1 1/2"-1/8" DEEP. Lesker Part # EVS20A010W or Tungsten boat, thick (.015") trough boat, Lesker part # EVS20A015W	ALUMINUM PELLETS, Al, 99.99% PURE, 1/4" DIAMETER x 1/4" LONG Lesker Part # EVMAL40QXQD
1	*Chromium*	CHROME PLATED TUNGSTEN ROD, 5/PKG, FOR Cr EVAPORATION, 4" LONG X 0.050" DIAMETER ROD, W/ CLEAN/FLUSH ENDS, Lesker Part # EVSCRW2 Or Tungsten boat, thick (.015") trough boat, Lesker part # EVS20A015W	CHROMIUM PIECES, Cr 99.95% PURE, 0.8MM TO 6MM PIECES, 25 GRAMS, Lesker part # EVMCR35A
2	*Titanium*	TUNGSTEN BOAT SOURCE, DIMPLE, 4" L X 1/2"W X 1/8" DEEP-7/16" thick. Lesker Part # EVS9A015W Or Tungsten boat, thick (.015") trough boat, Lesker part # EVS20A015W	TITANIUM PELLETS, Ti, 99.995% PURE, 1/8" DIAMETER X 1/8" LONG, Lesker Part # EVMTI45EXE-A
3	*Copper*	MOYBDENUM BOAT SOURCE, 4"L X 1/2"W X 7/16"-1/8" DEEP, 0.005" THK MATERIAL. Lesker Part # EVS9A005MO Or Moly boat, medium thick (.010") trough boat, Lesker part # EVS20A010MO	COPPER PELLETS, Cu, 99.99% PURE, 1/8" DIAMETER X 1/8" LONG, Lesker Part # EVMCU40EXEA
4	*Gold*	Users must supply their own boat and material. TUNGSTEN DIMPLE BOAT 4" L X 1/2"W X 1/8" DEEP-7/16" thick. Lesker # EVS9B010W You may have some issues with the Au and W alloying but this is the downside of thermal evaporation. Alumina coated boats are **not recommended**, they lead to significant process issues, avoid them for evaporating Au.	Users must supply their own boat and material Lesker is a good source for Au pellets, recommend the 1/8" size EVMAUXX50G

Pump Down

1. Once everything is loaded, close the door by sliding it all the way to the right.
2. Push "chamber door open/close". The readout changes to "closed". The door should close firmly against the seal.
3. Push "chamber evacuate". The display will show "Rough Evav". ***THERE WILL BE A VERY LOUD NOISE AS THE SCROLL PUMP TURNS ON, THIS IS NORMAL. You may want to warn other users in the lab before you start the pumpdown!***
4. The loud noise of the scroll pump should get quiet within a minute or so, and the chamber pressure should start to drop.
5. By about 5 minutes time the chamber pressure should reach about 1 mbar and the cryo gate valve will open automatically. At this point the readout will change to "cryo evac".
6. Pressure will drop rapidly to the 10^{-5} mbar range (within 30 seconds).
7. The cryo temperature will bump up a little to about 17 K or so, this is normal. It should then drift back down to about 14 K.
8. Within a minute the readout should read "at press" and the chamber pressure should be in the 10^{-6} mbar range.
9. Allow the chamber to continue pumping to reach the desired base pressure (mid 10^{-7} mbar range is easily achievable).
10. If pumpdown does not proceed as described above – particularly if the cryo temperature is high (> 20 K) or the pressure does not get down easily into the 10^{-6} or 10^{-7} range, inform lab staff and do not run your deposition.

Note : If you plan to leave the lab for an hour or so while the tool pumps down, that's fine. It might be a good idea to leave a note with your name and phone/email telling people that you are pumping down and when you expect to finish your deposition, in case someone comes while you are not there and vents the tool!

Run Automatic Deposition

1. **Power up the small recirculating chiller, this cools internal evaporator components.**
2. *Make sure the Siemans control panel has the control mode set to "auto" mode under chamber/substrate. This means that the Inficon can control the substrate shutter and substrate rotation.*
3. In the Inficon controller, select the desired Process.
4. *In the Inficon controller, make sure the manual/auto menu item is toggled so that "auto" is at the top – it should read "auto/manual" **NOT** "manual/auto"!!*
5. Select "Quick edit…" on the Inficon, and set the film thickness to be the desired thickness you want to deposit. DO NOT EDIT ANY OTHER PARAMETERS.
6. Return to the main menu.
7. Verify that the name of the process (e.g. Ti2, Cu3, …) appears at the top of the Inficon controller.
8. Use the "next display" and "next graph" buttons to get to your favorite display – I like the one that shows power vs. time on the graph, and shows the power level, dep rate, and total thickness for each source in the table below.
9. You should see that the source you plan to use is colorful and bright in the table and all other sources are grayed out.
10. Note that source 1 and 2 should use sensor 1 (this is the crystal on the left) and sources 3 and 4 should use sensor 2(this is the crystal on the right). This should already be programmed into the process so there is nothing to change.
11. In addition, the tooling factor should already have been measured by lab staff and programmed into the material data, so there is nothing to set there either.
12. *On the Siemans control panel, toggle the "evap boats power" to "enabled".*
13. Once everything looks good, push "start process" and "start layer".
14. The Inficon will now take over control of the tool. You should see an initial ramp up to a first soak, then a second ramp up to a second soak.

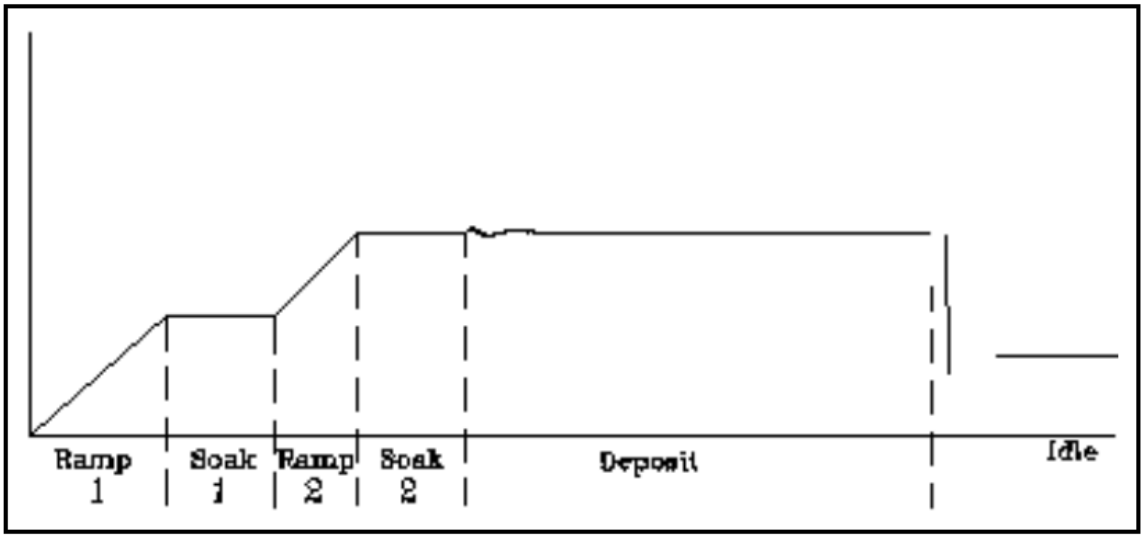
15. At this point it will start controlling the power in a closed loop fashion to achieve the target dep rate. The substrate rotation should turn on and substrate shutter should open automatically.

See the table and graph below for details of the different approved processes.

IMPORTANT NOTE: if the boat breaks you will hear a "ping" sound. Or, if you run out of material, the dep rate will drop to zero and the Inficon will try to raise the power to compensate, but it will not have enough material! (Note that each 1/8" pellet gives about 25 nm of material). In either case:

1. Switch to manual control mode on the Siemans controller
2. Close the substrate shutter.
3. Switch to manual control on the Inficon (push auto/manual on the Inficon). Ramp the power back down to 0 at about 10%/minute.

Process Name	**Source #**	**Material**	**Dep Rate**	**Ramp 1**	**Soak 1**	**Ramp 2**	**Soak 2**	**Tooling Factor**
	1	*Aluminum*						
Ti2	**2**	*Titanium*	1 Å/sec	2 min to 10 %	2 min	5 min to 55%	1 min	100%
Cu3	**3**	*Copper*	3 Å/sec	2 min to 10 %	2 min	3 min to 30%	30 sec	82%
	4	*Gold*						



16. Once your desired thickness is reached, the system should automatically close the substrate shutter and ramp the power back down to zero..
17. You are done – go on to a second film, or use the vent procedure above to vent and remove your samples. Then pump the chamber back down, fill out the log book, and walk away.
18. Turn the small chiller off

Run Manual Deposition

Alternatively, you can run the process manually. For some difficult to deposit materials such as Titanium it may be easier to control the process manually since the Titanium dep rate can be somewhat variable. Still follow a similar procedure to the above process in terms of soaks and slow ramps.

1. **Power up the small recirculating chiller, this cools internal evaporator components.**

2. Make sure the Siemans control panel has the control mode set to "manual" mode under chamber/substrate. This means that the user has control of the substrate shutter and substrate rotation.
3. Close the substrate shutter.
4. Turn on the substrate chiller.
5. Turn on substrate rotation.
6. In the Inficon controller, select the desired Process.
7. *In the Inficon controller, make sure the manual/auto menu item is toggled so that "manual" is at the top – it should read "manual/auto"* ***NOT*** *"auto/manual"!!*
8. Edit the process, edit the layer, and set the film thickness to be something higher than the thickness you actually want to deposit.
9. Return to the main menu.
10. Verify that the name of the process (e.g. Ti2, Cu3, …) appears at the top of the Inficon controller.
11. Use the "next table" and "next graph" buttons to get to your favorite display – I like the one that shows power vs. time on the graph, and shows the power level, dep rate, and total thickness for each source in the table below.
12. You should see that the source you plan to use is colorful and bright in the table and all other sources are grayed out.
13. Note that source 1 and 2 should use sensor 1 (this is the crystal on the left) and sources 3 and 4 should use sensor 2(this is the crystal on the right). This should already be programmed into the process so there is nothing to change.
14. In addition, the tooling factor should already have been measured by lab staff and programmed into the material data, so there is nothing to set there either.
15. *On the Siemans control panel, toggle the "evap boats power" to "enabled".*
16. Once everything looks good, push "start process" and "start layer".
17. You now have direct control of the power level using the wheel on the Inficon controller.
18. Ramp up to 10% power in about 2 minutes, and hold at 10% power for 2 minutes.
19. Now, ramp up to the power level listed in the table below at approximately 10%/minute. Don't go to fast or you will break the boat! You should start to see a bright glow in the chamber at about 10% power.
20. Once you reach the specified power in the table below, soak for a few minutes. You should see the dep rate start to increase.
21. Once the dep rate gets up above 0.5 A/sec or so, slowly adjust the power level to try to maintain the desired dep rate (see table below). You should be able to maintain dep rate within 5% of the stated power levels below.
22. Once you have the dep rate you want, push "open substrate shutter" on the Siemans control panel.
23. Push "zero" on the Inficon controller.
24. Deposit the desired film thickness, adjusting power slowly as you go to maintain the desired dep rate.

    Note: if the boat breaks you will hear a "ping" sound. In that case, close the substrate shutter and ramp the power back down to 0 at about 10%/minute.

    Note: if you run out of material, the dep rate will drop to zero. At that point you have to stop – don't try to make the material deposit by ramping up the power too high! You don't have any material left! Each 1/8" x 1/8" pellet has enough material for about 250 Å of film thickness. Close the substrate shutter and ramp power back down at 10%/minute.

Source #	**Material**	**Recommended Dep Rate**	**Approximate Power Level for Recommended Dep Rate**
1	*Aluminum*		
2	*Titanium*	1 Å/sec	60%
3	*Copper*	3 Å/sec	35%
4	*Gold*		

25. Once your desired thickness is reached, close the substrate shutter.

26. Ramp power back down to 0 at about 10%/minute.
27. Push “stop layer” on the Inficon.
28. Push “next menu” on the Inficon. Push “reset” on the Inficon.
29. You are done – go on to a second film, or use the vent procedure above to vent and remove your samples. Then pump the chamber back down, fill out the log book, and walk away.
30. Turn off the small recirculating chiller

Normal Shutdown

1. Vent the tool according the procedure at the beginning of this SOP.
2. Remove your samples.
3. Pump the chamber back down.
4. Leave the toolt under vacuum.
5. Remember to enter your job in the logbook.

Emergency Shutdown

If you need to shut down in the middle of a run for any reason, the “stop layer” button on the Inficon controller should be used. Then “reset” the controller (next menu | reset). Inform lab staff.

If something dangerous appears to be happening, you hit the big red emergency off button to shut down all power to the tool. Note that if you shut down all power to the tool the pumps will turn off, which will put significant wear on the tool.

If at any time you feel unsure about how to use the tool, please stop work and contact a qualified user or faculty advisor. Please don’t forge ahead when you are unsure, you may end up damaging the tool.

Report all accidents or tool issues to Prof. White at x72210, r.white@tufts.edu or to Jim Vlahakis at james.vlahakis@tufts.edu.