MMRC PVD Evaporator Instructions

(Updated Aug 2016)

Warnings

- Under PVD conditions, even oils and greases are volatile. WEAR GLOVES, and if you touch your skin or anything else oily or wet, change them. One fingerprint on your sample or the evaporator parts may mean an extra hour or more of pump-down time, depending on the pressure you need to get to. The next user will have to pump off your fingerprint even if you don't have to get a low pressure, so PLEASE be considerate and very careful. Dust will not disturb the vacuum, but may degrade the quality of your evaporated films. Be especially careful of gold dust, which usually coats every surface of our evaporator, at least inside the birdcage.
- Two photographs of the system are shown below. Familiarize yourself with the location of the valves and pressure gages.
- The diffusion pump valve is the hand wheel on the right side of the evaporator clockwise. It requires 15 rotations to go from fully open to close. When closing you need to give it an extra push to completely seal it.
- When closing the bell jar always use your gloved hands to rub clean the seal on the bell jar and the region of the metal base where the seal will rest. Pull down the bell jar and hold it against the base lightly when you open the roughing pump valve.

• Startup

1. **Make sure the NESLAB is turned on (water cooler) to the right of the evaporator in the corner (both zones need to be turned on).**
   i. Check that the water level in the circulator is within 3 cm of the top; if not, add DI water.
   ii. Verify that water is flowing at the rotary flow switch behind the evaporator. If water isn't flowing, verify that the four valves connecting the NESLAB to the evaporator are open. If the flow switch doesn't spin, the diffusion pump will not heat up. This is a safety precaution.

2. **Make sure the bell jar thermocouple gauge is turned on**
   i. Located hanging immediately to the left of the bell jar it should read 000. If not, **do not proceed,** contact the GLA.
3. **Initial checks:**
   i. Make sure the bell jar thermocouple gauge is turned on if not contact the GLA
   ii. Make sure the mechanical pump behind the PVD is turned on, if not contact the GLA.
   iii. Close the roughing valve, C (Fully Clockwise).
   iv. Make sure vent valve, D is closed.

v. Close the diffusion pump Valve, A (Use the hand wheel on the right side of the evaporator clockwise (15 rotations) until it stops. Give it an extra push to seal it).

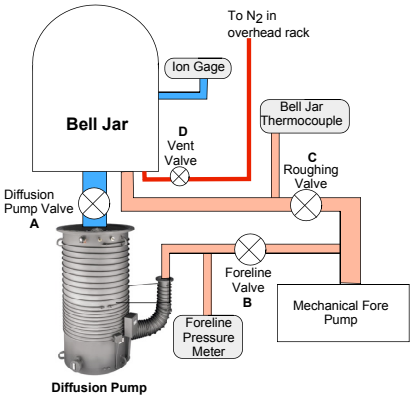
vi. Open the foreline valve, B (CCW).

vii. Wait till the foreline pressure gauge reads < 10 mTorr (microns of Hg)

4. **Turn on the diffusion pump heating element**
   i. Flip the switch on the right-side panel of the evaporator marked "diffusion pump" to "ON." Make sure that current flow is indicated on the topmost panel meter.
   ii. You can load the chamber while waiting for the diffusion pump to heat up, or you can proceed with step 3 of "Evacuation" immediately, if you want to pre-desorb stuff from the walls.

Loading

1. **Prepare your source and sample. Make them as free of volatiles as possible.**
   i. WEAR GLOVES, and if you touch your skin or anything else oily or wet, change them. See warnings above.
   ii. Use the right source for the metal you want to put down, and know what conditions you'll have to reach to make it evaporate. Use the right-side filament for hard-to-evaporate metals. You may also wish to use smaller sources to help you heat high-melting metals (Pd, Pt, and Ni especially). Do not mix different metals or differing purities on the same filament. Assume any filament is used for 4-9's (99.9999% by mass) purity or better.



2. **Make sure the diffusion pump valve, A, is closed. [fully ccw]**
3. **Make sure the roughing valve, C. is CLOSED. [fully cw].** The chamber MUST be fully isolated from the pumps before it can be safely opened.
4. **Open the valve(s) to the N_2 on the overhead rack. Then open the vent valve, D, until the bell jar lifts off of the base, then close it**
   i. The vent valve has a round black handle and is at the front of the evaporator. Open it all the way; it takes almost a minute to pressurize the bell jar. Be sure to close the vent valve tightly once the bell jar pops free. Torque it firmly, but not abusively.
5. **Close the vent valve and the valve to the N_2 on the overhead rack.**
6. **Load your source and sample; use the birdcage if at all possible**

i. Screw your source firmly into the elevated clamps, rotating a coil such that any eccentricity along the long axis is as low as possible. Make sure the shutter blocks the source when in place, and does not do so when moved away.

ii. Place your sample underneath the source; directly underneath if directionality is a concern, not directly underneath if you are worried you may lose a drop of your metal when it first melts. Use the elevated table to save metal and time, if film uniformity is not very critical and heating of your sample is not a problem. (Photoresist usually does fine.)

7. Determine and enter the tooling factor for your setup (if not already entered)

i. Turn on the thickness monitor (Inficon) by flipping the switch in the back, top center. The display should flash "P Fail." Press the "STOP" button. If any sort of failure is still indicated after you press "STOP," please get the GLA to help you out. Something is wrong.

ii. Measure the distance (a) from the center of the source to the bottom of the thickness monitor oscillator crystal above it. This should be very close to 24 cm. Measure the distance (b) from your source to your sample. This should be roughly 11 cm with the table in, 24.5 cm with the table out. Now calculate the tooling factor (TF):

$$TF = \frac{\square^2}{\square} \times 100\%$$

Typical values are $\square$ = 23 cm, $\square$ = 12 cm (table in) or 25 cm (table out)
TF = 367.4 % (table in) or 84.6% (table out)

iii. Enter the tooling factor on the thickness monitor by pressing the "TOOLING FACTOR" button followed by the "INCREASE" or "DECREASE" buttons as appropriate.

iv. Check the oscillator crystal life is above 85% by pressing the "TEST" button. The two rightmost digits on the rate display give the percent of crystal life remaining.

Evacuation

1. Pump down the bell jar with the mechanical pump

i. **Close the foreline valve, B, (cw)**

ii. Using your gloved hands, rub clean the seal on the bell jar and the region of the metal base where the seal will rest. Pull down the bell jar and hold it against the base lightly.

iii. **Open the roughing valve, C, (ccw)** Open the valve all the. It may sound funny.

iv. (You can now let go of the bell jar.) Wait until the bell jar pressure drops below 20 mTorr. This takes about 5 minutes.

2. **Feed the thickness monitor the input data it requires**

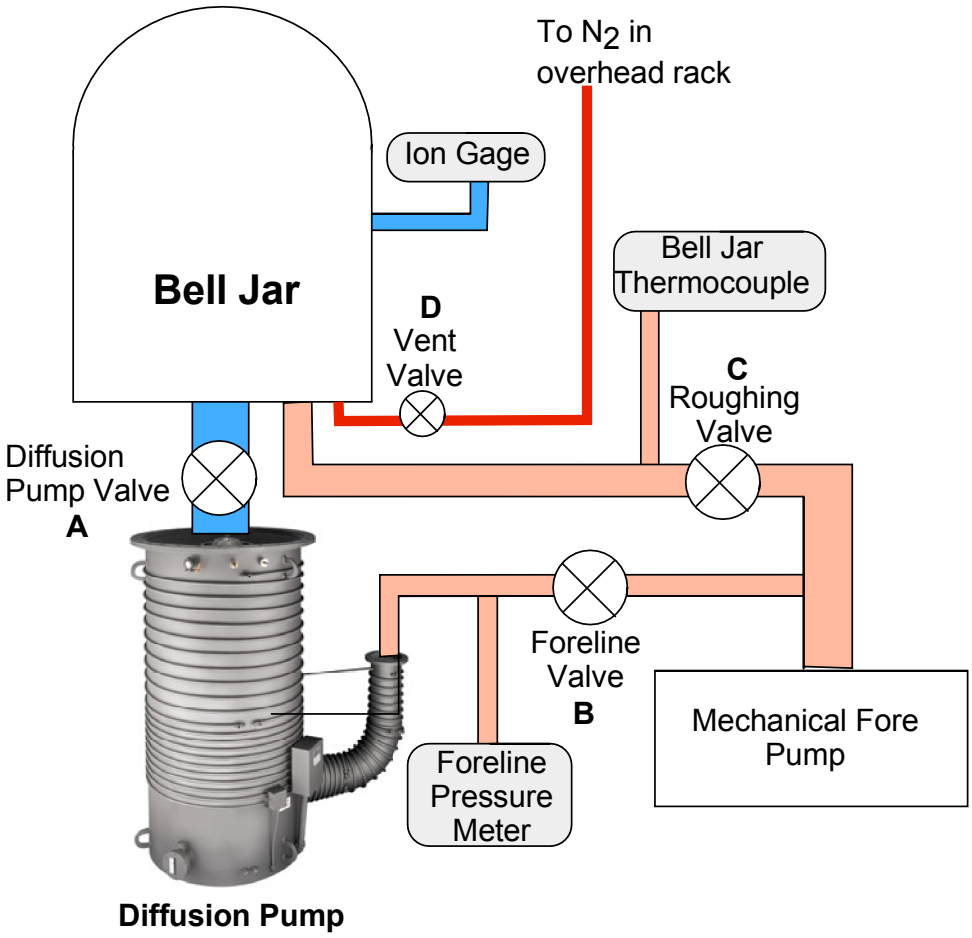
i. Enter the appropriate material density, most likely one of those listed in the table below.

ii. Check that the acoustic impedance is set to 20, unless thickness accuracy is critical, in which case you should enter the appropriate value from the table below. (The acoustic impedance has a minimal impact [<10%] on the thickness reading, so we rarely bother changing it.)

Material	Density (g/cm^3)	Acoustic Impedance	Melting Point (°C)	Approximate Maximum Pressure (Torr) & Comments
Au	19.30	23.18	1062	10^{-4}; usually poor adhesion
Ag	10.50	16.69	961	10^{-4}; evaporates very well
Cr	7.20	28.95	1890	sublimes 10^{-4}; adheres very well

Cu	8.93	20.21	1083	10^{-4}; usually poor adhesion
Co	8.71	25.74	1495	10^{-5}; alloys with W
Fe	7.86	25.30	1535	10^{-4}; attacks W filaments
Ni	8.91	26.68	1435	10^{-4}; alloys with W
Pd	12.00	24.73	1550	10^{-4}; alloys W, evaporate fast
Pt	21.40	36.04	1769	10^{-7}; alloys W, poor adhesion
Ti	4.50	14.06	1675	10^{-6}; alloys with W

3. **Fill the diffusion pump trap and baffle system with liquid nitrogen**
   i. Hold a small portable Dewar under the liquid tube of a low pressure N_2 Dewar. Slowly open the liquid valve on a low pressure N_2 Dewar. After the whistling stops, open the valve completely until liquid N_2 starts spraying out of the small Dewar.
   ii. Fill the trap from the portable Dewar and pouring LN_2 into the trap with the help of the metal funnel or the LN_2 hose if available.
   iii. **Note:** The baffle and trap system prevent diffusion pump oil vapors from escaping. Try to keep LN_2 in the trap whenever the diffusion pump is hot and the diffusion pump valve is open.
4. **Pumping the bell jar to low pressure.**
   i. Wait until the bell jar thermocouple gauge reads less than 30 mTorr.
   ii. Close the roughing valve, C. Open the foreline valve, B and wait till foreline pressure <10 mTorr
   iii. *Slowly* open the diffusion pump valve, A,. ***Watch*** the foreline pressure gauge. If the foreline pressure approaches 20 mTorr, close the diffusion pump valve until it drops again. Open the diffusion valve all the way once this does not raise the foreline pressure. It takes about 16 turns to do so.
5. **Wait 30 minutes.**
6. **Turn on and activate the ion gauge controller**
   i. Press the "PWR" button on the digital ion gauge controller [on the shelf next to the bell jar thermocouple gauge controller]. Press the "ON" button to activate the ion tube. If the pressure is above 9×10^{-5} torr, turn off the controller and wait 15 more min.
7. **Allow the pressure to drop until it is low enough for your evaporation**
   i. To make the ion gauge reading accurate, you need to activate the "DEGAS" feature for about 3 minutes, and then let the gauge cool again. When the "DEGAS" button is pressed, a heating element in the ion tube heats the glass walls, forcing off adsorbed junk. This junk normally comes off the ion tube continually but slowly, artificially raising the ion gauge pressure reading. Once the tube is cool again, such junk no longer comes off and the pressure reading is accurate.
   ii. The lowest of the present system is in the low-10^{-7} Torr range, Normal pressures for deposition are in the low 10^{-6} Torr range, and the pressure climbs quickly when a filament is energized.
   iii. If you cannot get down to the pressure you need, there are a few tricks. Please talk to the GLA if you are having trouble; most of any solution will require patience on your part.


To N_2 in overhead rack
Ion Gage
Bell Jar
Bell Jar Thermocouple
D
Vent Valve
C
Roughing Valve
Diffusion Pump Valve
A
Foreline Valve
B
Mechanical Fore Pump
Foreline Pressure Meter
Diffusion Pump

Metal Deposition

1. **Check the LN_2 Dewar and refill.**
2. **If possible, pre-wet the filament without evaporating any metal.**
   i. Turn on the filament(s) you are going to use at the right-side panel of the evaporator.
   ii. With the shutter closed (in place between the filament and your sample), turn up the appropriate filament power control gradually ~1 tick every 2 min. [there is a significant lag between turning up the power and the filament reaching a steady temperature!], until the filament begins to glow and the metal on it starts to melt and absorb into (wet) the filament fibers. Once all of the new metal has melted, turn the filament power down to zero. **Do not do this with pre-made sources, like the Cr on W rods we get from R.D. Mathes.**
   iii. **Do not do this with metals that alloy with tungsten.** Pre-wetting is not essential, but it reduces the risk of a liquid metal drop falling from the filament and destroying your sample during the evaporation process.
3. **Record data in the log and deposit your metal once the pressure is low enough**
   i. Fill in the date, metal(s), initial pressure, and your initials in the evaporator logbook.
   ii. Slowly turn up the power to the filament [~1 tick/ 2 min] until the thickness monitor indicates an acceptable metal deposition rate for the evaporation you want to do.
   iii. *Simultaneously* turn the shutter handle under your filament to "open" and press the "START" button on the thickness monitor. When the thickness of metal deposited comes to within about 8 Å of the total thickness you would like, turn the filament variac quickly back to zeroWhen the rate display drops back to 0.1 Å/sec, press the "STOP" button on the thickness monitor.
4. **If you have another filament to run on the same substrates, repeat Step 3.**
5. **Finish filling in the log; let the system contents and diffusion pump cool down**
   i. Record the final pressure (immediately after metal deposition) and the metal thickness
   ii. **Unless you are doing another evaporation immediately following this one, turn off the power switch for the diffusion pump.**
   iii. **Turn off the Filament power**
   iv. The evaporator contents, in particular the filament(s), are very hot after an evaporation. Because they are in vacuum, they cool down very slowly, mostly by conduction. **Allow a minimum of 20 minutes for the system to cool before you continue.**

Unloading & Shut-Down

1. **Repressurize the bell jar after isolating all pressure-sensitive systems**
   i. Close the diffusion pump valve, A, [cw], check that the roughing valve, D, is closed.
   ii. Make sure the ion guage is turned off.
   iii. Open the valve(s) to the N_2 on the overhead rack.
   iv. Open the vent valve, D, until the bell jar lifts off of the base. Open it all the way, and be patient. It takes almost a minute to pressurize the bell jar.
   v. Close the vent valve tightly when the bell jar is free. Torque it firmly, but not abusively.
   vi. Close the valve on the overhead rack to the N_2
2. **Remove your sample and ready the system for the next user**

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i. **As always, use gloves while handling anything inside the bell jar**

ii. Clean or replace the glass windows on the birdcage. Either clean them with a Kimwipe (works with metals that don't adhere well) or throw them away and put in new ones.

iii. You can leave the filament in if it still has plenty of metal and is a commonly used source. If you take it out, put the filament in the proper box in the "Evaporation sources" drawer.

iv. If you need to do another evaporation, go back to the "Loading" section from here.

3. Pump down the bell jar with the mechanical pump

i. Close the foreline valve, B [cw]

ii. Using your gloved hands, rub clean the seal on the bell jar and the region of the metal base where the seal will rest. Pull down the bell jar and hold it against the base lightly.

iii. Open the roughing valve, C [ccw]

iv. Wait until the bell jar pressure drops below 20 mTorr. This takes about 5 minutes unless you got fingerprints inside the evaporator.

4. Switch over to the diffusion pump once the bell jar pressure is ≤ 30 mTorr

i. Wait until the bell jar thermocouple gauge reads less than 30 mTorr.

ii. Close the roughing valve,C. Open the foreline valve, B, wait till foreline pressure < 10 mTorr**.**

iii. *Slowly* open the diffusion pump valve. If the foreline pressure approaches 20 mTorr, close the diffusion pump valve until it drops again. Open the diffusion valve all the way.

5. Leave the bell jar under active vacuum. Turn off the diffusion pump and auxiliary systems, however, leave the Neslab running.

i. The N_2 Dewar can defrost and the diffusion pump can finish cooling down after you leave. **Be sure the diffusion pump has been OFF for at least 15 minutes before you go on to the next step!**

ii. Leave the foreline, B and diffusion pump, C valves open, and the roughing, C and vent, D valves both tightly closed.

iii. Turn off the two thermocouple gauges, thickness monitor, and water cooler.

iv. Fill out the log book