

BI Metal Physical Vapor Deposition Evaporator Instructions

(Updated 07/26/2010 by MGW)

1. Make sure the neslab to the right of the evaporator in the corner is turned on (water cooler).

- Check that the water level in the circulator is within 3 cm of the top; if not, add DI water.
- 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 an important safety precaution.**

2. Make sure the bell jar thermocouple gauge is turned on

- Located hanging immediately to the left of the bell jar, the digital readout should be reading 000 microns. If it is not turned on, please **do not** proceed and contact the GLA.

3. Close the diffusion pump valve

- If the system hasn't just been used, the foreline valve should be open and the diffusion pump should be off. If so, turn the hand wheel on the right side of the evaporator, toward the front, clockwise until it stops. This may take up to 16 turns. When the valve stops, give it an extra push to seal it, as the diffusion pump valve makes use of an O-ring compression seal.

4. Verify diffusion pump can be safely heated

- Diffusion pump oil **oxidizes** if exposed to air when hot, and it sprays out and covers *everything* if the diffusion pump sees a differential pressure.
- Make sure the ROUGHING VALVE is CLOSED [Fully Clockwise]
- Make sure the FORELINE VALVE is OPEN [CCW] and the MECHANICAL PUMP is ON
- Make sure the DIFFUSION PUMP VALVE is CLOSED [Fully Clockwise]
- Make sure the FORELINE PRESSURE gauge reads < 10 millitorr (microns of Hg)

5. Turn on the diffusion pump heating element

- 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.
- 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.

- Under these 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.
- 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 is CLOSED. [FULLY CLOCKWISE] Make sure the ROUGHING VALVE is CLOSED. [FULLY CLOCKWISE]

- The chamber MUST be fully isolated from the pumps before it can be safely opened.

3. Open the valve(s) on the N₂ tank to the right of the evaporator

- Don't turn the pressure regulator dial; open the valve on the gas cylinder itself, then rotate the selector switch beyond the regulator such that it points to the *left*.
- Make sure that the tank contains an appreciable quantity of N₂, so you don't evacuate the nitrogen tank.

4. Open the vent valve until the bell jar lifts off of the base, then close it

- The vent valve has a metallic green 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 valve(s) on the N₂ tank.

6. Load your source and sample; use the birdcage if at all possible

- 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.
- 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.)

Loading, continued...

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

- 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 Rob to help you out. Something is wrong.
- 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):

$$TF = \left(\frac{\alpha}{\beta} \right)^2 \times 100\%$$

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

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

- **CLOSE THE FORELINE VALVE (CW)** [lower round handle on back right side]
- 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.
- **OPEN THE ROUGHING VALVE (CCW)** [upper round handle on back right side] Open the valve all the way in order to maximize pumping speed. It will sound funny.
- (You can now let go of the bell jar.) Wait until the bell jar pressure drops below 20 millitorr. This takes about 5 minutes.

2. Feed the thickness monitor the input data it requires

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

Evacuation, continued...

3. Fill the diffusion pump trap and baffle system with liquid nitrogen

- Slowly open the liquid valve on the low pressure N₂ dewar next to the evaporator. After the whistling stops, open the valve completely until liquid N₂ starts spraying out of the reservoir.
- Fill the trap using a portable dewar and pouring LN₂ into the trap with the help of the metal funnel.

Note: The baffle and trap system prevent diffusion pump oil vapors from escaping. Try to keep LN₂ in the trap whenever the diffusion pump is hot and the diffusion pump valve is open.

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

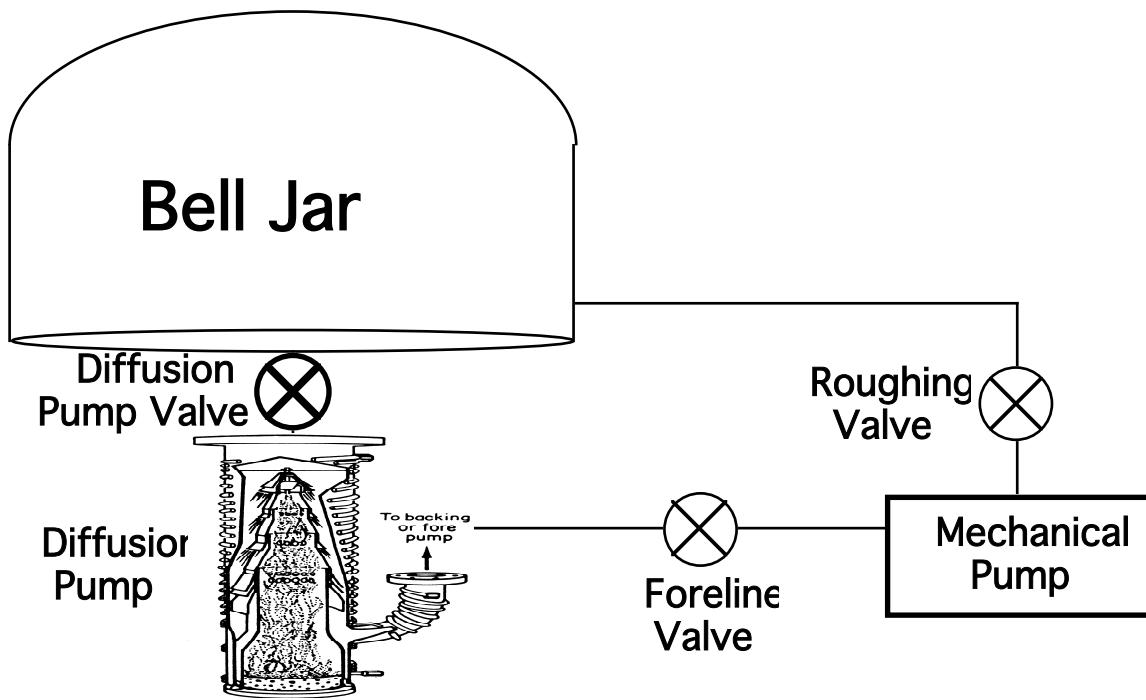
- Wait until the bell jar thermocouple gauge reads less than 30 millitorr.
- **CLOSE THE ROUGHING VALVE. OPEN THE FORELINE VALVE.**
- *Slowly* open the diffusion pump valve [the hand wheel closest to you on the right side of the evaporator]. *Watch* the foreline pressure gauge. If the foreline pressure 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. Turn on and activate the ion gauge controller

- 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.

6. Allow the pressure to drop until it is low enough for your evaporation

- To make the ion gauge reading accurate, you will need to activate the "DEGAS" feature for about 3 minutes, 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.
- The lowest pressure I've been able to get out of the present system is in the low-10⁻⁷ millitorr range, and the pressure climbs quickly when a filament is energized.
- If you can not get down to the pressure you need, there are a few tricks. Please talk to me if you are having trouble; most of any solution will require patience on your part.



Metal Deposition

1. If possible, pre-wet the filament without evaporating any metal.

- Turn on the filament(s) you are going to use at the right-side panel of the evaporator.
- With the shutter closed (in place between the filament and your sample), turn up the appropriate filament power control gradually [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.**
- **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.

2. Record data in the log and deposit your metal once the pressure is low enough

- Fill in the date, metal(s), initial pressure, and your initials in the evaporator logbook.
- Slowly turn up the power to the filament until the thickness monitor indicates an acceptable metal deposition rate for the evaporation you want to do.
- *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 zero. When the rate display drops back to 0.1 Å/sec, press the "STOP" button on the thickness monitor.

3. If you have another filament to run on the same substrates, repeat Step 2.

4. Finish filling in the log; let the system contents and diffusion pump cool down

- Record the final pressure (immediately after metal deposition) and the metal thickness
- **Unless you are doing another evaporation immediately following this one, TURN OFF THE POWER SWITCH FOR THE DIFFUSION PUMP**
- The evaporator contents, in particular the filament(s), are very hot after an evaporation. Because they are in vacuo, 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

- **MAKE SURE THE DIFFUSION PUMP VALVE IS CLOSED. [CW]**
- **MAKE SURE THE ION GUAGE IS TURNED OFF.**
- Open the valve(s) on the N₂ tank immediately to the right of the evaporator
- Make sure that the tank contains an appreciable quantity of N₂.
- Open the vent valve until the bell jar lifts off of the base. The vent valve has a metallic green handle and is at the front of the evaporator. Open it all the way, and be patient. It takes almost a minute to pressurize the bell jar. (That's a mighty big bell jar. Do yourself a favor and make sure the small valve is all the way open, not just a quarter-turn, or you'll wait for an eternity in the next step.)
- Close the vent valve tightly when the bell jar is free. Torque it firmly, but not abusively.
- Close the two valves on the N₂ tank immediately to the left of the evaporator

2. Remove your sample and ready the system for the next user

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

- **CLOSE THE FORELINE VALVE [CW]** [lower round handle on back right side]
- 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.
- **OPEN THE ROUGHING VALVE [CCW]** [upper round handle on back right side]
- (You can now let go of the bell jar.) Wait until the bell jar pressure drops below 20 millitorr. 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

- Wait until the bell jar thermocouple gauge reads less than 30 millitorr.
- **CLOSE THE ROUGHING VALVE. OPEN THE FORELINE VALVE.**
- *Slowly* open the diffusion pump valve [the hand wheel closest to you on the right side of the evaporator]. *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. Leave the bell jar under active vacuum. Turn off the auxiliary systems.

- The N₂ dewar can defrost and the diffusion pump can finish cooling down after you leave. **Just be sure the diffusion pump heat has been OFF for at least 15 minutes before you go on to the next step!**
- **Leave the FORELINE and DIFFUSION PUMP valves open, the ROUGHING and VENT valves both tightly closed.**
- **Turn off** the two thermocouple gauges, thickness monitor, and water cooler.