CORAL NAME: GB_Metals_Evaporator, GB_Organics_Evaporator

Location: Lab 2U, 12-3111
Purpose: Deposition
Contact: Gary Riggott

Contents

Introduction 1
Safety 1
User Qualifications and Responsibilities 2
Operating Procedures 3
Appendices 6

Introduction

The GB_Metals_Evaporator and GB_Organics_Evaporator are located in an inert nitrogen glovebox that provides ambient conditions with less than 3 ppm levels of water (moisture) and 0.1 ppm oxygen (O_2). The glovebox is intended as an environment to enable the processing of oxygen and moisture-sensitive samples. The glovebox contains equipment to enable thin film deposition of moisture or air-sensitive samples including:

- a Thermal Evaporator for Metals (left chamber)
- a Thermal Evaporator for Organic Materials (right chamber)

Thermal evaporation uses a resistive heater around the source “boat” to heat a target metal to a high enough temperature so that it evaporates and deposits on the sample.The two chambers can each hold four boats: one of them has a 4kVA heater for higher temperatures while the other three have 1kVA heaters. The two chambers share a power supply and therefore can’t be used at the same time.
Only anhydrous materials are allowed in the glovebox; this includes both solid materials and liquids. Samples are introduced into the glovebox through an antechamber which is cycle purged multiple times to remove any residual moisture and to maintain a pristine processing environment.

Safety

The glovebox is not intended as personal protective equipment, its primary intent is to protect the materials and samples inside from exposure to oxygen or moisture. The doors to the antechambers and the evaporator source load doors may be pinch points, so use caution when opening and closing. Handling of materials and equipment through the gloves may be awkward, so work slowly and carefully to avoid knocking over or damaging equipment and samples. Forceps are available inside the glovebox to help grasp out-of-reach items.

The glovebox gloves provide an interface to handling items inside the environment, they are not intended as personal protective equipment (PPE).

- For all processing, standard PPE (nitrile gloves) must be worn over the glovebox gloves and safety glasses must be worn at all time. Some chemicals may react with the butyl glovebox gloves causing them to dissolve or soften, the disposable nitrile PPE protects them, extends their lifetime and prevents the spreading of contamination to other samples.

- Careful handling and proper disposal of sharps, including samples, is required. If the gloves are torn or punctured, seal with electrical tape and report in CORAL.
- General mechanical hazards such as squeezing, stretching, moving parts, sharp corners and edges exist when using this system. Be aware of those hazards and work carefully.

All personal chemicals, including solid source targets must be reviewed and approved by MIT.nano Safety Staff. Submit a Personal Chemical Request on the linked form and staff will follow up. All chemicals and samples must be clearly labeled with a name and date, unlabeled items will be disposed of.

The glovebox has additional electrical and thermal hazards from the equipment inside.

- Do not attempt to modify any electrical wiring in the system or to defeat any safety interlocks.
- Do not unplug any electrical cords inside the glovebox.
- Thermal evaporators heat targets to high temperatures during deposition. Use caution when loading and unloading samples and deposition materials as they may be hot.
- Identify the Emergency Off (EMO) on the front of the Control Panel. If the system exhibits dangerous behavior such as thermal runaway that may damage the equipment or the building, then you may need to press the EMO.
- Certain materials may require the use of a respirator for loading and unloading; these materials must be run with staff assistance and will require additional charges for staff time.

When loading and unloading samples, a vacuum is pulled on the antechamber. Make sure any containers or vessels used for transfer are vacuum compatible to avoid breaking glass during this cycle purge sequence.

Safety Interlocks:
Three interlocks must be met in order to perform an evaporation with this tool:

- The vacuum level in the process chamber must be < 3.75e-5 Torr. Often chamber pressure rises during deposition-the pressure must not climb > 3.75e-4 Torr, or the power will cut out.
- The yellow doors at the back of the evaporator glove box must be properly and securely closed. These doors protect the user from exposure to high voltage electrical components.
- The chiller must be on, this prevents the system from overheating during deposition.
- When these interlock conditions are met, the Volts and Current displays become active (they illuminate). If they remain off, one of the above 3 conditions are not met.

User Qualifications and Responsibilities

To maintain the low oxygen and low moisture environment, it is the Users' responsibility to observe the following rules and best practices. Failure to do so may result in disqualification and retraining will be required.

- All users must have an approved PTC process and all Personal Chemicals must have approval from MIT.nano Safety Staff.
- Users must receive training and qualification on glovebox use from MIT.nano Staff. Users must be engaged on the glovebox for the duration of their work.
- Abnormal conditions must be reported immediately in CORAL. This includes high O2 or moisture levels, issues with the antechamber cycle purge process and issues with the gloves.
- Users must be familiar with the above Safety requirements and must follow all safe working practices in the lab.

- Do not remove parts/supplies from the lab, they are part of the shared system and required for everyone to operate the tools.
- Clean all the residuals from your experiment and do not leave any materials and samples unattended.
- Users must pass a quiz on glovebox safety and best practices.
- At the end of each run both antechambers must be left under vacuum.
- Users must record the O2 and moisture levels at the start and end of each run in the log book.

Materials: Only **anhydrous** materials are allowed inside the glovebox. This rule applies to solids and solvents.
Porous materials such as fab wipes or cardboard (for boxed gloves, foil rolls) are prohibited as they introduce large amounts of moisture into the system.

Storage: Approved personal chemicals may be stored inside the glovebox for the typical 6-month duration of personal chemical requests, extensions may be granted for longer duration. All samples and chemicals must be labeled with the username, the date, and the chemical name. Abreviations are acceptable if the chemical is added to the "key" which provides full details and chemical names and is posted on the side of the glovebox. Materials dissolved in non-hydrocarbon solvents must be stored in **Teflon-capped vials with electrical tape wrapped around the rim.** Materials stored in any other way (unwrapped vials, centrifuge tubes, syringes, etc.) will be removed from the glovebox. When a user has finished with a chemical it should be removed from the glovebox and properly disposed of (MIT.nano Staff can assist).

Operating Procedures

Sample Loading



Vent the chamber (Controller-Turbo off)
Pump Controller (Edwards) for Metal Evaporation Chamber is on the left (**1**), Organic Evaporation Chamber is on the right (**2**). Please note that chambers should always be kept under vacuum when not in use.

1. Turn turbo and backing pumps off by pressing System Control (**B**) button twice:
    a. First press opens the System Control menu.
    b. Second press switches "SYSTEM ON" to "SYSTEM OFF."
2. Press Menu button (**A**) to return to the main menu.
3. Chamber will begin venting procedure, which takes about 15 min.
    a. Note: begin this process immediately at the start of your reservation to save time.
    b. Pressure will rise to 7.5E+2, then chamber will be fully vented in an additional 3-5 minutes after this point is reached.
    c. If the chamber doesn't vent, verify you are engaged in CORAL; chamber vent valve is interlocked.

Load Samples into Glovebox

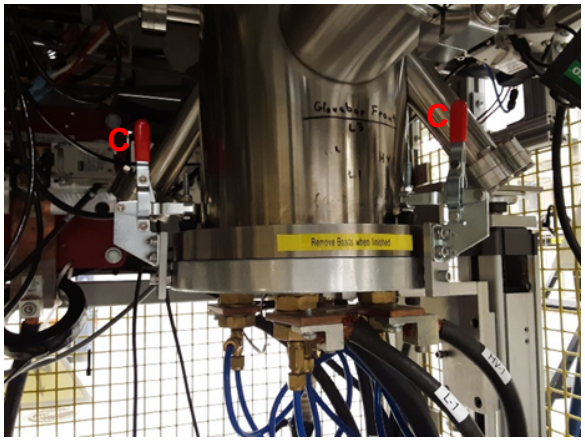
1. Reserve tool time in advance on CORAL. Engage the appropriate process chamber in CORAL before starting your process.
2. Open the Small Antechamber and load your samples, then close the door. Initiate the cycle purging sequence to minimize moisture coming into the glovebox: run 3 cycles x 5 minute pump-down (15 minutes total).
3. Use the manual "Refill/Evacuate" valve on the front of the antechamber to move back and forth between pumping and backfilling with nitrogen.

4. Open Refill valve to fill the antechamber with nitrogen until it reaches atmospheric pressure
5. Open Evacuate valve to pump down the antechamber for 5 minutes (use a timer)
6. Repeat the above steps three times
7. When not in use, the antechamber should be pumped down and isolated.
8. Make sure all containers are vacuum-compatible: don't introduce anything with trapped air into the glovebox (capped vials, syringes/needles in their wrappers, transfer tubes capped in air, etc.) Thin-walled glass or plastic containers can potentially explode when placed into a vacuum.

Mounting the substrates/Loading into Process Chamber

1. Place substrates into substrate holder with the prism-shaped bumpers facing down, toward the bottom of the chamber.
2. Select and assemble mask holder, keeping the mask and holder stars aligned.
3. Insert substrate holder/mask holder into evaporation chamber carefully. Try not to wiggle substrate holder around so as not to scratch the active layer near the devices.
4. Make sure substrate holder is properly in place atop mask holder before securely closing chamber lid.

Loading the Materials





1. Open the shielding doors at the back of the evaporator Glovebox and remove them.
2. Open the toggle clamps (**C**) used to keep the chamber closed.
3. While holding loading chamber up with the wheel, unscrew the set screw on the back of the rails. While holding onto both the wheel and the bottom of the loading chamber, gently lower the chamber by allowing the wheel to spin (controllably).
4. Unscrew boat set screws and remove boat.
5. Place a new boat into clamps. Center the boat in the clamps. Tighten boat clamps.
6. Add the material to the boat. Pellets should be standing up and be clustered near the middle of the boat.
7. Check the crystal monitor life (**D**). The *Inficon* controller located in the front of the glove box. If the crystal life is less than **87%**, please follow the crystal monitor replacement procedure in the Appendix.
8. Lift loading chamber by cranking the wheel. Support the bottom of the loading chamber and do not release the wheel. As the loading chamber gets close to the chamber housing, guide it so that the boat apparatus fits neatly inside of/does not bump the housing and so that the lip of the chamber aligns with the lip of the housing in the front and back (there will be some overhang on one side).
9. When the door is in place and aligned, tighten the set screw in the back of the rails.
10. Close and secure the toggle clamps (**C**) to secure the chamber for pump-down.
11. Turn turbo and backing pumps back on by pressing **System Control** button twice. Wait until pressure reaches at least ~1e-6 Torr before starting evaporation procedure (currently 30-60 min).

12. Close the shielding doors at the back of the evaporator Glovebox. Make sure that the doors contact each other to complete the safety circuit. If the doors are closed properly, the green interlock light (**E**) will be on continuously. If the doors are not closed properly, this light will flash.

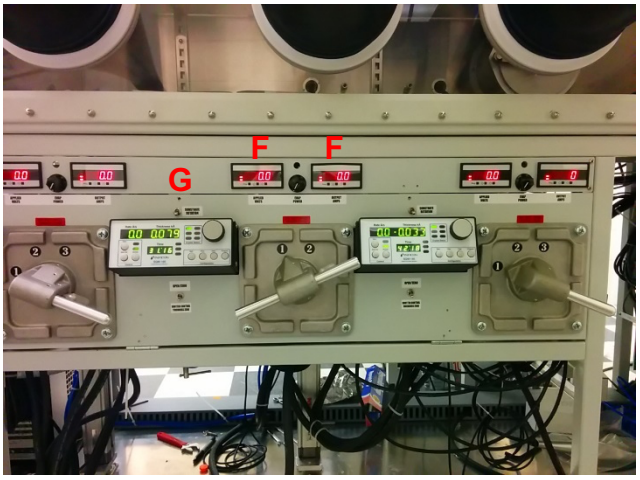


Evaporating

1. Record your run in the logbook.
2. On the Inficon Controller, choose the program. To see program parameters (density, tooling factor, etc.), press **PROGRAM**, scroll to program number using large knob, and press **NEXT** to cycle through parameters. Confirm density, tooling and Z-factor with known values.

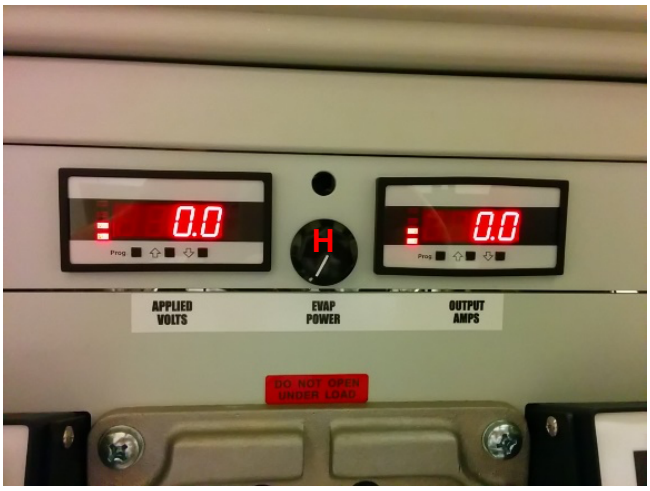


3. Enter Final Thickness in kA.
4. In order for evaporation to begin, the three Safety Interlocks described in the Safety section must be met:
    a. The chamber pressure must be less than 3.75E-5 Torr.
    b. The yellow shielding doors at the back of the glovebox must be properly closed.
    c. Chiller must be on and flowing (red flow switch lights will be off).
    d. When these conditions are met, the red Voltage and Current displays (**F**) illuminate.



5. Turn substrate rotation (**G**) on using the switch above *Inficon* growth rate/thickness controller.
    a. Speed controller for substrate rotation is a knob located under chamber Pressure Controller. Standard rotation speed is 1/4 turn/second.
6. Close shutter: Press the **SHUTTER** button on the Inficon controller so that CLOSED illuminates.
7. At the appropriate voltage source slowly begin turning current knob (**H**) clockwise. Increase to 1.0 volts and hold for 30 sec.
    a. Left selector: 1kVA source for metal evaporator

   b. Middle selector: 4kVA source (HV1-for the metal evaporator and HV2 for organic evaporator)
   c. Right selector: 1kVA source for organic evaporator



8. Watch the applied current (A) and system pressure (Torr) while ramping up the deposition rate to 1 Å/s for metals and 0.1 Å/s for organics :
   a. Always turn Current Knob (**H**) slowly and in small increments, allowing the current reading/pressure stabilize in between.
   b. At some point during the ramping, materials will start to degas, causing a spike in pressure.
   c. Maintain pressure below 2e-5 at all times. Stabilize pressure just below 2E-6 Torr.
9. When appropriate growth rate has stabilized, begin evaporation by flipping **Shutter Control** switch located below growth rate/thickness controller. Flip the switch from the down position to up **Open/Zero**. This will open the shutter and zero the crystal monitor.
10. During deposition, monitor the pressure, current, and deposition rate. Dep rate sometimes fluctuates a little. Adjust **Applied Volts** accordingly so as to maintain a steady growth rate.
    a. The **Applied Volts** knob is highly sensitive and has slow response. Make small adjustments then wait patiently for a change in current and dep rate.
11. The shutter will automatically close once Final Thickness (Step 3 above) is reached. Deposition is now complete.
12. Turn current knob (**H**) slowly to zero current over 30 seconds.
13. Turn the **Shutter Control** switch under rate/thickness monitor off to **Shutter Control Thickness Zero** position.
14. Turn off substrate rotation switch (**G**) above rate/thickness monitor.
15. Initiate venting the chamber by pressing System Control (**B**) button twice, followed by the Menu (**A**) button.
16. Venting takes about 15 min. Once the screen displays 7.50E+2 Torr, it will take an additional 3-5 min to be able to open the chamber and remove your substrates and loaded materials.
17. Turn chiller off after venting is complete.
18. Put the system under vacuum by pressing the System Control (**B**) button twice (ensure that both Turbo and Backing pumps turn on), followed by the Menu (**A**) button. Verify that the pump down begins and pressure decreases.
19. Complete the logsheet. Include measured thickness (if applicable) and any comments/observations on film appearance.

Appendices

Tips and Tricks

- Do **not** attempt to open the antechamber doors while it is under vacuum!
- Users requiring very low base pressure can load the tool and pump through the night and perform the deposition the next day. Users must remain engaged on equipment for the duration of their run, including pumpdown time.
- Never put porous materials or materials with adhesive, such as general lab tape, into vacuum tools that get hot inside. This can result in contamination of the equipment.

- Check log book for recent depositions of the same material by other users. Deposition conditions, such as the current and voltage are expected to be similar (for example, deposition rate increase will begin at the same current).

Crystal Monitor Replacement

1. Prepare a work area in the hood with a clean aluminum foil.
2. Gently pull crystal monitor sensing head from the chamber (crystal monitor located inside the evaporation chamber, on the top left side, near the substrates).
3. Poke the crystal out with one gloved finger and replace it with a new one.
4. Gently put the new crystal monitor inside the chamber.
5. Verify that the new crystal is working properly by reading the lifetime.

Loading Air Sensitive Samples into Process Chamber:

Air sensitive samples follow the same steps and procedures as above, however there are two important changes:

- The materials are loaded into the process chamber before the samples.
- The process chamber is partially pumped down to remove air, then vented with N2 before the samples are loaded.

1. Follow the instructions for **Loading the Materials**
2. Pump the process chamber down to remove air from the chamber.
3. Press the System Control (**B**) button so that the pumps turn on. Start a timer.
4. Press the System Control (**B**) button again after 60 seconds. This will turn the pumps off and vent the chamber (about 5 minutes).
5. The air has now been pumped out of the process chamber and replaced with N2.
6. Now load your samples as described above in “Mounting the Substrates/Loading into Process Chamber”.

Metals Evaporator Materials

The materials shown in the table below have been previously approved for deposition in the Metals Evaporator chamber. The table lists the relevant deposition parameters. New materials that have been approved by the MIT.nano Safety Staff will require assistance from staff to set up new parameters.

Material	Program	Density	Tooling Factor	Z-Factor	Comment
Silver (Ag)	90	10.49	49	0.53	
Aluminum (Al)	85	2.73	71	1.08	
Gold (Au)	81	19.3	59	0.38	
Calcium (Ca)	87	1.55	70	2.62	
Chromium (Cr)	34	7.2	70	0.31	Rod source
Molybdenum Oxide (MoO3)	82	4.75	97	1	

Organics Evaporator Materials

The materials shown in the table below have been previously approved for deposition in the Organics Evaporator Chamber. The table lists the relevant deposition parameters. New materials that have been approved by the MIT.nano Safety Staff will require assistance from staff to set up new parameters.

Material	Program	Density	Tooling Factor	Z-Factor	Comment
Aluminum (Al)	86	2.73	100	1.08	
Gold (Au)	95	19.3	59	0.38	
Bismuth (Bi)	55	9.8	45	0.79	
Bismuth Teluride (Bi2Te3)	56	7.7	60	1.00	Heeger materials, 2-3mm flakes
C60	74	1.1	52	8.83	
Copper (CuPc)	75	1.1	52	8.83	
Indium (In)	35	7.3	60	0.84	
Lead (Pb)	94	11.34	60	1.13	Staff-Run Only
Tungsten Oxide (WO3)	84	7.16	100	1	
Pentacine					
Phthalocyanine					
Zinc (Zn)	36	7.14	97	0.514	Requires Ti adhesion layer for witness piece