

Glovebox Usage Manual v1.4

Logbook

When you enter the lab walk straight to the log book. Enter all information including N₂ and O₂/H₂O levels.

Nitrogen tank

Alert Daeri Tenery or Andre Gesquiere if the pressure at the nitrogen cylinder is at 200 psi (for weekdays) and 500 psi (for the weekend)



Figure 1: Read the N₂ cylinder pressure (dial indicated by yellow arrow) and record in logbook at start and end of glovebox use.

MBRAUN controller

The glovebox electronics are controlled by a touch screen display. The touch screen implies touching, not hitting or poking. When you come in the lab the controller will be in the home screen, see Figure 2. If not touch *End* at current panel.

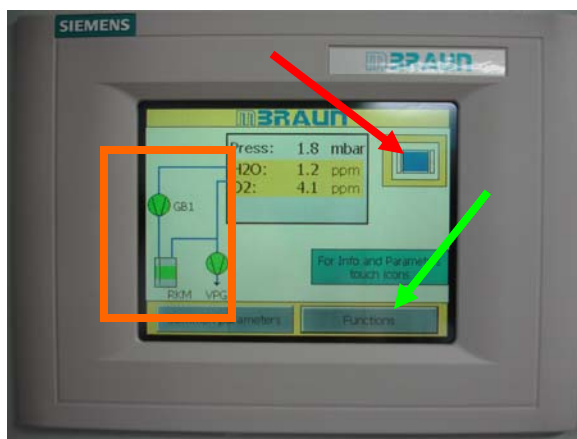


Figure 2: MBRAUN controller home screen. The red arrow points to the antechamber/evaporator menu icon, the green arrow points to the functions menu icon. Box pressure and O₂/H₂O levels are reported in the center of the screen.

The orange box in Figure 3 contains 3 green icons related to the pumping and circulation status of the glovebox. If any of these are red contact Andre or Daeri. To turn on the glovebox light or restart circulation (see below!) touch *Functions*. This will take you to the panel shown in Figure 3.



Figure 3: In the functions menu the *Circulation Purifier 1*, *Analyzer*, and *Box Light* buttons are of importance.

To turn on or off the glovebox light touch the *Box Light* button.

Sometimes the box circulation can shut down during operation when the user exceeds the upper box pressure limit (for example two people put hands in at the same time, put hands to fast etc...) In that case you will no longer hear the blower noise, the analyzers will be off (i.e. the system is not reading O2 and H2O levels), and the circulation and analyzer buttons in the Functions menu will be red instead of green. You can restore the system circulation by pressing *Circulation Purifier 1* followed by *Analyzer*. Both should now appear green as in Figure 3. If that doesn't work ask for help, don't mess around with the system.

NEVER TOUCH *REGENERATION* UNDER ANY CIRCUMSTANCE WHATSOEVER.

Loading samples from the smaller chamber to the glove box

1. The smaller chamber is operated *manually*.
2. When you first come in, the chambers should be in *static vacuum*.(this means the monitor reads -30 in. Hg and the manual evacuation/refill valve is closed, see Figure 4)



Figure 4: Mini-antechamber under static vacuum.

3. Refill the chamber slowly (i.e. don't turn the manual evacuation/refill valve too fast) → close the manual evacuation/refill valve → turn the knob on chamber lid counterclockwise and open by rotating lid → place a sample **Note: no Chem wipes, paper towels, or other materials that absorb air or moisture**
Take off the caps from containers---this goes for anything that has caps or lids (i.e. Vials, slide container etc...)
After loading materials close the chamber door by rotating lid and turn knob clockwise until knob is finger tight → evacuate to -30 and refill 3 times → close the manual evacuation/refill valve. **The chamber is now filled with nitrogen.**
4. Wear glove on the left hand (**NOTE: NO rings or watches**) and stick your hand in the glovebox → Turn the knob counterclockwise to open the chamber door, open by rotating lid → unload the sample → close the door by rotating lid and finger tight the knob
5. Evacuate chamber to -30 → close the valve
6. When you leave, the chamber should be on *static vacuum*. (this means the monitor reads -30 and the manual evacuation/refill valve is closed, see Figure 4)

Unloading the samples from the glove box to the smaller chamber

1. The smaller chamber is operated *manually*.
2. When you first come in, the chambers should be in *static vacuum*.(this means the monitor reads -30 and the manual evacuation/refill valve is closed)
3. Refill the chamber slowly (i.e. don't turn the manual evacuation/refill valve too fast) → evacuate to -30 and refill 3 times → close the manual evacuation/refill valve. **The chamber is now filled with nitrogen.**
4. Wear glove on the left hand (**NOTE: NO rings or watches**) and stick your hand in the glovebox → Turn the knob counterclockwise to open the chamber door,

open by rotating lid → load the sample → close the door by rotating lid and finger tight the knob

Turn the knob on chamber lid outside the glovebox counterclockwise and open by rotating lid → take samples

After taking materials close the chamber door by rotating lid and turn knob clockwise until knob is finger tight

5. Evacuate chamber to -30 → close the valve
6. When you leave, the chamber should be on *static vacuum*. (this means the monitor reads -30 and the manual evacuation/refill valve is closed, see Figure 4)

Usage policies

Strictly follow the above procedures. They are written up like this for a reason: the procedures work. Don't reinvent procedures.

Use the signup sheet to reserve time on the thermal evaporator. In the event you will not use the system after signing up you must cancel reservations 24 hours or more before the time of your reservation. **Don't waste other people's time by signing up and not showing up.**

You must sign in before using the glovebox (your hands touch the log book before they touch the glovebox)

Keep glovebox environment clean, do not leave aluminum foil, vials etc. lying around.

Do not spill chemicals inside glovebox or on the gloves, and clean up spills thoroughly. You can wear nitrile gloves over the ones you wear to use the glovebox.

You must consult with Andre before loading new materials, including chemicals and user supplies or equipment

Do not leave the system unattended at any time when using antechambers or the evaporator. While in use you must remain in the room to supervise the operation of the glovebox in case an issue comes up.

Do not change settings on the instrument, especially the ones you don't know what they mean. You can only change parameters related to your particular process (e.g. thermal evaporator film parameters)

The following process and film assignments are in place for the evaporator:

Paul process and films 1-5

Andre process and films 6-10

Daeri process and films 11-15

Zhongjian process and films 16-20

Lynn process and films 21-25

You are required to follow the following assignments of materials to pockets for the thermal evaporator:

aluminum: OUT1 pocket1

aluminum: OUT1 pocket2

chromium: OUT2 pocket1

gold: OUT2 pocket2

The evaporator will only be operated from inside the glovebox.

Place boats under the washers on the posts. Only finger tighten the screws. You don't need tools.

Do not exceed 50% power on the evaporator system. If you reach this power level and nothing happens something is out of sorts that needs to be corrected.

Only use sensor 2 for deposition, if the crystal fails contact Andre.

Additional policies established by the instrumentation committee apply to external users.

If you fail to follow user policies your account will be subject to suspension.

I have read these usage policies and the user manual, and will follow the procedures explained therein.

Name of user:

Email address of user:

Signature of user:

Date:

The usage policy and user manual are subject to revision.

Date revised: 03-22-08

Revision history

V1.4, 03-22-08: release to instrumentation committee

Thermal Evaporator

Follow the procedures described in the Glovebox section for loading and unloading samples and materials, and pay specific attention to filling out the logbook and confirming N₂ levels.

Logbook

When you enter the lab walk straight to the log book. Enter all information including N₂ and O₂/H₂O levels.

Venting and preparing evaporator

The evaporator is kept under vacuum 24/7. When you enter the system will be under vacuum and pumping. To vent the system touch the *Antechamber/Evaporator* menu icon (Figure 2). You will be taken to the menu shown in Figure 5.



Figure 5: Antechamber 1 menu. The *Evaporator* button is located in the bottom left corner.

Touch the *Evaporator* button to move on to the evaporator screen (Figure 6).

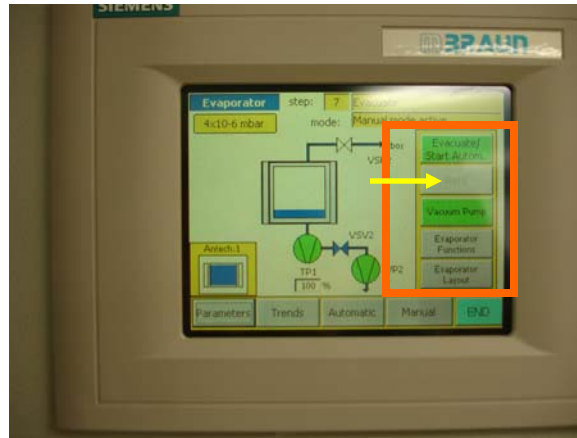


Figure 6: Evaporator screen. The chamber pressure is displayed in the top left corner. An evaporator schematic is shown in the center and shows pumping status of the roughing vacuum pump (VP2) and the turbomolecular pump (TP1). Green means the pump is on. The relative rotation speed of the turbomolecular pump is displayed as well (bottom center of screen). The buttons in the right hand column control the evaporator pumping cycle and shutters (orange box). Green means the pumps are activated. The refill button (yellow arrow) is grayed out during pumping. The *END* button returns you to the home screen.

Touch the *Evacuate/Start Autom.* button to shut down the turbomolecular pump. The button will turn red, and TP1 will turn red also in the evaporator schematic. Now you have to wait until the TP1 relative speed reaches 89% or lower. This is to protect the turbomolecular pump upon refilling the vacuum chamber.

Once the system is safe to refill with N₂ the refill button will change from grayed out to red. Touch the refill button. It will turn green and the vacuum chamber will start filling with N₂. When the chamber pressure reads “atmosphere” touch the refill button again. Now the refill button is colored red again.

Unlock the evaporator door by pressing the “inner door release”, shown in Figure 7.



Figure 7: The evaporator door is operated from the front of the glovebox. Press “inner door release” to lock or unlock the vacuum chamber door.

Load your boats, targets and substrates into the chamber. Do not over tighten the screws that hold the boats in the posts. In principle you do not need tools, just your fingers to tighten the screws.

Lock the evaporator door by pressing the “inner door release button”, shown in Figure 7.

Pumping down the vacuum chamber

When the vacuum chamber door is sealed, touch the *Evacuate/Start Autom.* button to engage the turbomolecular pump. The button will turn green, and TP1 will turn green also in the evaporator schematic. Once the pressure reaches 2×10^{-6} mbar or lower (~ 20 minutes) you are ready to evaporate your materials.

Creating an evaporation process

The evaporation process is controlled from the Sigma SQC-310 deposition controller. Once the vacuum chamber pressure reaches 2×10^{-6} mbar or lower turn on the deposition controller. After the software has loaded you will be presented with the screen shown in Figure 8.

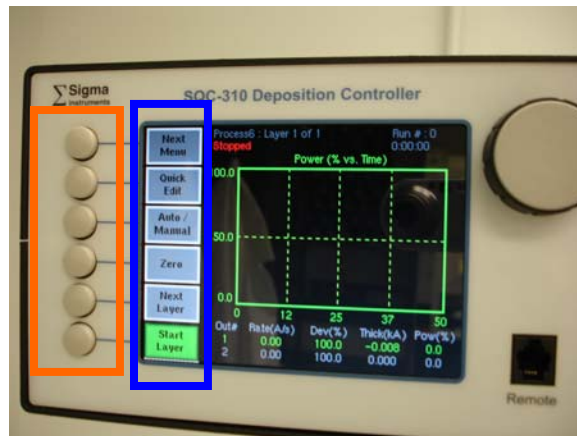


Figure 8: The default screen shown by the deposition controller after startup. We'll call this screen Menu1 for convenience.

There are six buttons on the left (orange box Figure 8) that are pressed to select the menu options shown on the screen (blue box Figure 8).

To create an evaporation procedure you need create a film, and then a process. This is covered in the next two sections.

Creating a film

From the startup screen press *Next Menu*. You will go to the screen shown in Figure 9, which we will call Menu2.

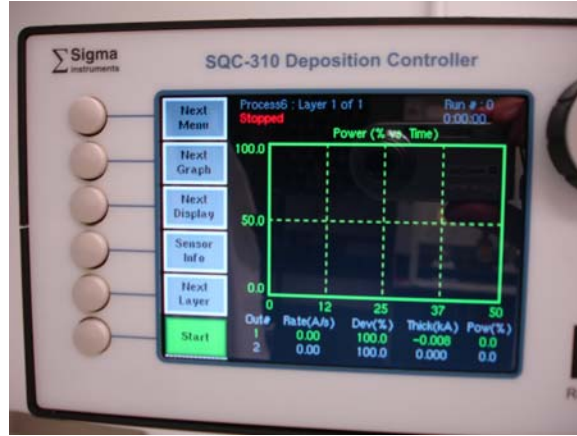


Figure 9: Menu2 is displayed on the SQC-310 controller.

Press *Next Menu* again. You will go to the screen shown in Figure 10, which we will call Menu3.

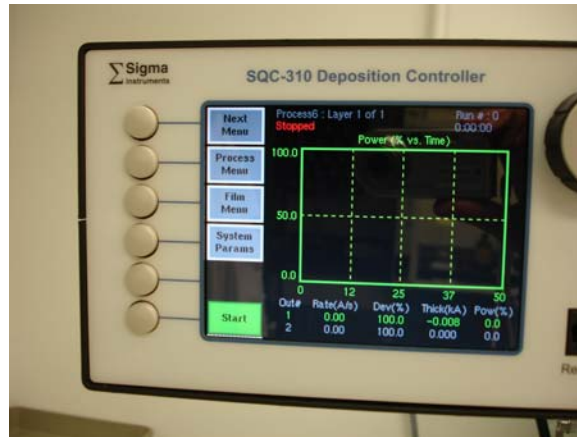


Figure 10: Menu3 is displayed on the SQC-310 controller. From here you can access the film and process menus.

That will take you to the following screen, shown in Figure 11, where you can set up the film for your evaporation. Press *Create* to define your film.



Figure 11: Screen where you create a film by pressing *Create*.

After creating the film you can edit its properties. With the selection box located on your film press *Edit* (Figure 12).

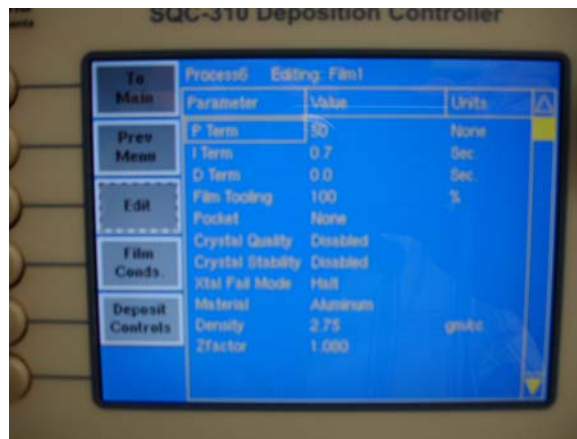


Figure 12: The film editing screen. Here you will adjust the Pocket and the Material.

There are only 2 parameters you need to worry about here, leave the other ones alone. The parameters of interest are:

1. Pocket: choose pocket 1 or 2 depending on the material you will deposit
2. Material: choose the material you will deposit. The corresponding profile already contains the correct parameters for successful deposition.

Use the scroll knob and edit button to make these changes.

Press the *To Main* button to go back to Menu3. Next up is creating a process to run the film.

Creating a process

On Menu3 press the *Process Menu* button. This will take you to the screen shown in .



Figure 13: Creating a process on the SQC-310 deposition controller.

Move the selection box to the appropriate slot using the scroll knob and press *Create*. In Figure 14 Process1 was created.



Figure 14: Process1 was created by pressing *Create*. That process is highlighted with the selection box and can be modified by pressing *Select*.

Select the process by highlighting it with the selection box and pressing *Select*. After the process is selected the *Select* button changes to an *Edit* button. Press the Edit button (not shown). This will take you to the screen where you can modify parameters related to the process of depositing your film.

First we need to connect your film with the actual deposition process. After pressing the *Edit* button we can complete this task on the next screen by pressing *Insert Layer*.



Figure 15: Connect your film to the process by pressing *Insert Layer*.

Choose the film using the scroll knob and press *Insert Layer*.



Figure 16: Add your film to the process you're building. Highlight the film of choice with the box and press *Insert Layer*.

Note: by repeating this process multiple layers can be added to a single process for e.g. co-deposition or sequential deposition.

The result is shown in Figure 17.

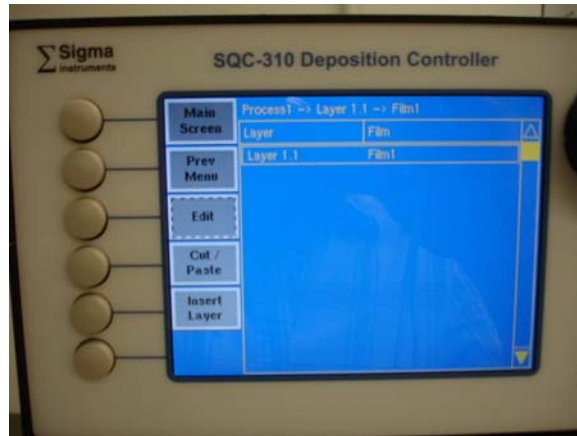


Figure 17: Press *Edit* to change the process parameters for each layer you have added.

Now highlight the inserted layer and press *Edit*. This will take you to the screen where we will set up a few more parameters (Figure 18).



Figure 18: Define process parameters by highlighting with the scroll knob and pressing edit.

In this screen you only need to edit Final Thickness, Output, Sensor1, and Sensor2.

1. Final Thickness: set your desired film thickness in kÅ
2. Output: choose 1 or 2 depending on the material you will deposit
3. Sensor1: Off
4. Sensor2: On

Also verify that the Start Mode is set to Manual.

Use the scroll knob and edit button to make these changes.

Press the *To Main* button to go back to Menu3. Next up is running the deposition.

Running deposition

Make sure the shutter over your source is open, and the shutter at the samples is closed.

Press *Next Menu* to go to Menu1. On Menu, change to manual mode by pressing the Auto/Manual button. Verify that Manual now appears first on that button.

To start the deposition process press *Start*. A *Start Layer* button will appear, press it. You will see a message at the top of the display that either says “Rotate Pocket” or “Manual Power”. Either mode works, the only difference is that you do not get a power vs. time graph in “Rotate Pocket” mode. If you decide you need to see this graph you can abort the process and press Start Layer again. You will now be in “Manual Power” mode.

Use the scroll knob to adjust power. Start with 5% increments every 1 minute until you reach 15% power. Now keep an eye on the chamber pressure and increase power with 2.5% increments. An increase in pressure means boat and material are outgassing. Wait a few minutes before increasing pressure further.

Do not exceed 50% power.

Once the desired and stable rate is achieved open the sample shutter.

To manually stop the deposition press *Abort* on Menu2.

Unloading samples and boats

Let the evaporator cool for 1 hour under vacuum

After 1 hour, start the venting process. See section “Venting and preparing evaporator”.

Remove boat(s) and samples/sample holders from chamber

Leave the system under atmosphere for the next user is scheduled within 3 hours after yourself, otherwise close the system and pump down. For instructions see section “Pumping down the vacuum chamber”.