



STANDARD OPERATING PROCEDURE

AIXTRON Black Magic Pro 6-Inch

CORAL Name: Graphene Black Magic

Model Number: Black Magic Pro

Location: 39-428a TRL

What it does: Mono-Layer and Multi-Layer Graphene Deposition



Figure 1. The TRL Black Magic Reactor

INTRODUCTION:

This tool accepts substrate pieces as large as 6-inch wafers, and **is gold-contaminated!** This system is designed to grow mono-layer (using Hydrogen and Methane) and multi-layer graphene (using Hydrogen and Acetylene). The growth occurs at 25 mbar and it uses a metal-catalyzed

substrate that can be either conductive or dielectric in nature. The tool can use either DC or square-wave AC bias voltages to produce plasma. Typically, the catalyst metals are Nickel or Copper. Representative catalyst thicknesses for graphene growth are 400-500 nm. The primary process gases are Methane, Hydrogen, Argon, Acetylene and Ammonia.

SAFETY:

Note the location of the red, **EMO** button near the monitor (Figure 2). Pushing this button disables the power to the entire system (but not the vacuum pumps). Use this button in emergency situations!!! Notify the Staff immediately so the system can be checked!!!



Figure 2. EMO Button

Due to the toxic nature of the processing gases, only qualified Staff is allowed to perform the tasks of turning gases on and off and changing of gas cylinders. Keep your hands away from all moving parts. Do not try to defeat any of the system interlocks.

If you encounter any problems while operating this machine, report them immediately in CORAL. The Staff in charge will address them in a timely fashion.

The reactor is made of very delicate parts. After a certain amount of cumulative deposition, the reactor chamber will require a disassembly and a thorough cleaning before further use. If the reactor is dirty, i.e., the chamber cannot reach a good vacuum level, the tool cannot ignite/sustain plasma, and/or, continuous arcs occur when plasma is ON, you should stop your process and immediately and notify the Staff in CORAL.

PROCEDURE:

Preliminaries:

- 1- Check reservations in CORAL to insure that you reserved the correct machine in the correct facility for the correct date. Please make only the reservations that you need, when you know you are ready to use them. *As a reference, a typical sample can be completed within 2 hours, including cooling.*



Figure 3. Process Gases are closed

- 2- Check that the process gasses are open. If they are closed (Figure 3), please call the staff in charge of the tool (Bob Bicchieri and Donal Jamieson), or call the Chemical Cell phone to get the gases open.

Log on:

- 3- 'Engage' the Black Magic in CORAL **before** you start the operation. Insure that the correct facility is set (ICL, TRL, etc.) and that your lot name and number of wafers are entered correctly. The CORAL switchbox must indicate "ON" in order to load the chamber!!!
- 4- Verify that the chiller (under the table, by the oven) is ON and the display reads 15 °C.
- 5- Power on the Monitor if necessary. The computer has two Windows accounts, one for Staff and one for users. Left click on the *PECVD* account. The screen should now look like in Figure 4:



Figure 4. Initial Screen



Figure 5. Log on Window

- 6- Using the mouse pad on the keyboard, double click on the “Black Magic PECVD” icon with the left button. A Log on window should appear (Figure 5).
- 7- Select your username from the username list using the arrows on the left of the username field. *If you don't have an account, please ask the Staff to create an account for you.*
- 8- Left click on *Password* and type your password.
- 9- Left click *Log On*

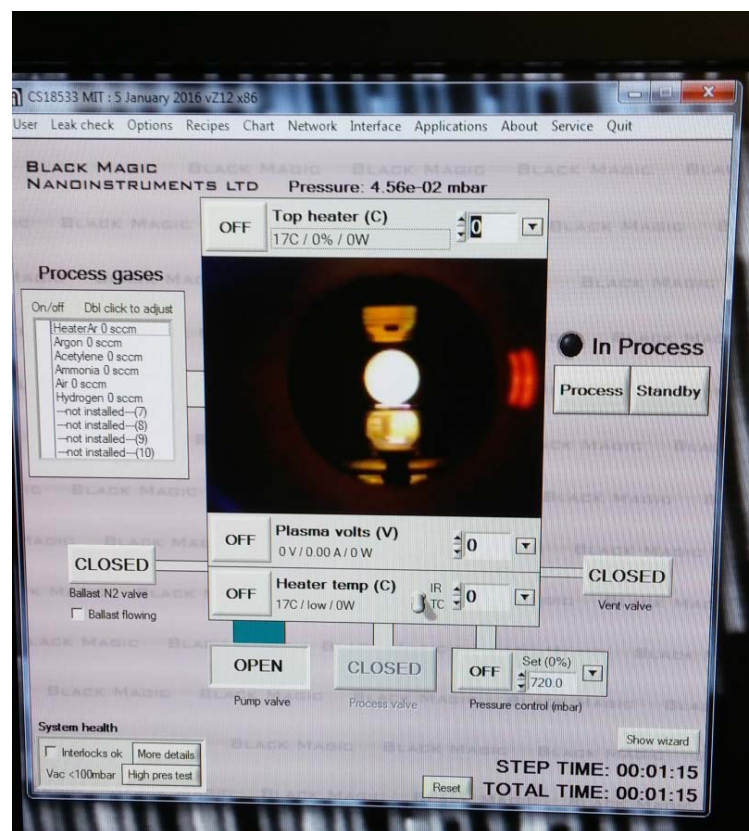


Figure 6. Initial Screen within running tool

- 10- A Black Magic Kernel Window will open and display various parameters initializing. Then a Black Magic 2006 Pro Window will open and display the status of the tool.

Before you proceed, there are several things you need to check (Figure 6). There are system interlocks that need to be satisfied before you can vent the tool:

- a- The pump valve is open
- b- The TC temperature is less than 200 °C.
- c- The tool is in **Standby** mode, i.e., there should be a **Black dot** next to **In Process**.
- d- The Foreline Vacuum valve behind the tool is open. Walk to the rear of the tool from the right side. Near the floor is an orange-handled valve. **Rotate it so the handle is parallel with the vacuum line, i.e., open.** See Figure 7.



Figure 7. Foreline Vacuum Valve in Open Position

Hydrogen Cleaning:

This is periodically necessary—if not, then proceed to Venting and Loading....

11- NOTE: This will significantly affect your total reservation time (extra 2 hours) as the tool will have to cool before loading your actual sample! To Proceed with H2 cleaning skip to **Step 22 and follow to Step 32**. Use the following recipe located in the H2 Cleaning Folder: **AixLtd_base_6inch_H2_Clean_1000C_20min**.

Venting and Loading:

12- Left Click on the *Pump Valve* to close it (Figure 8, left).

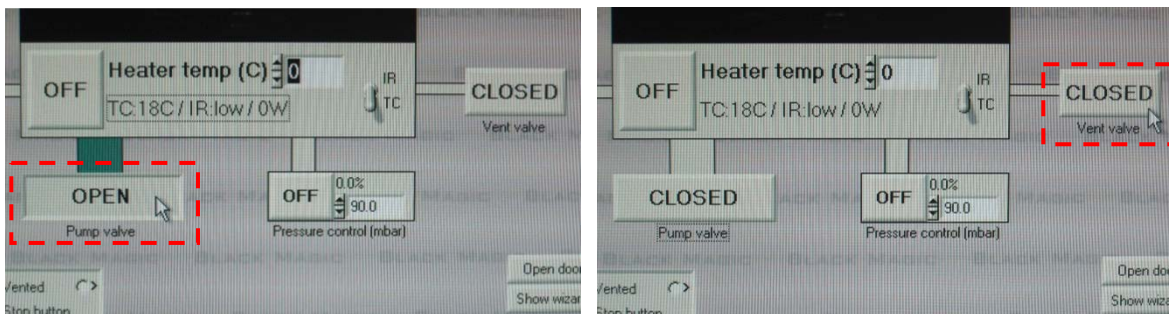


Figure 8. Close pump valve (left),

then open vent valve (right).

13- Left click *Vent Valve* to open it (Figure 8, right).

14- The chamber will slowly vent with Argon gas. Look in the Pressure field; it will indicate *Vented/Unlocked* (Figure 9).

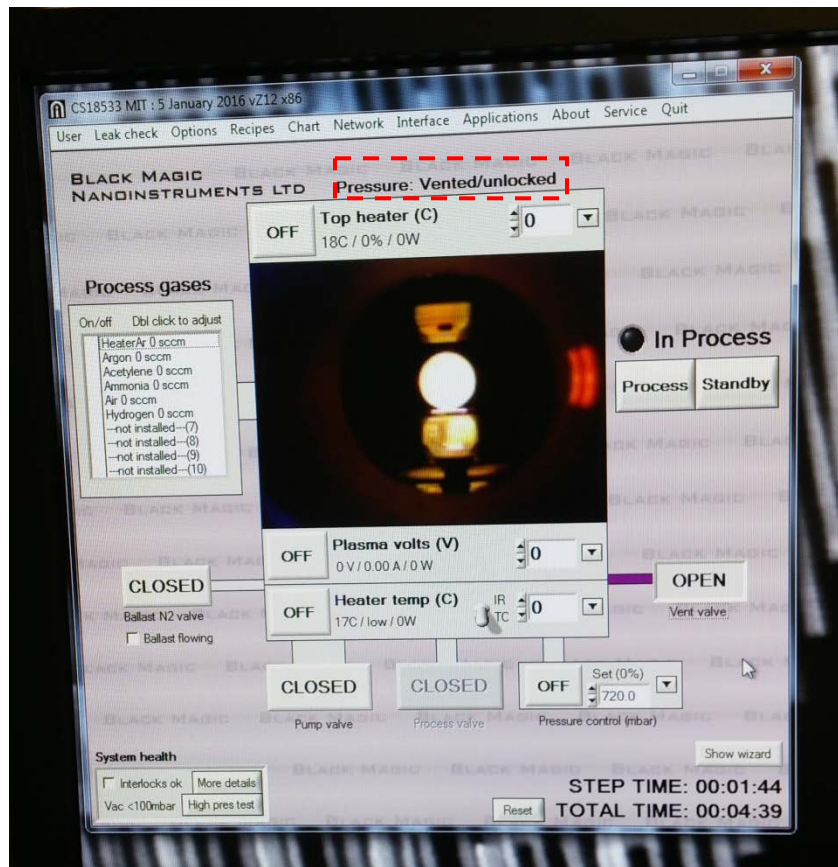


Figure 9. The reactor chamber is vented

15- Manually raise the enclosure door. Loosen the Chamber Lock and tilt the mechanism toward you, see Figure 10. Carefully lift the chamber lid (it is heavy).

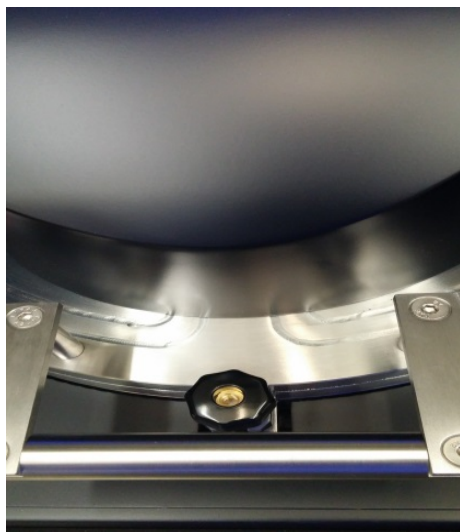
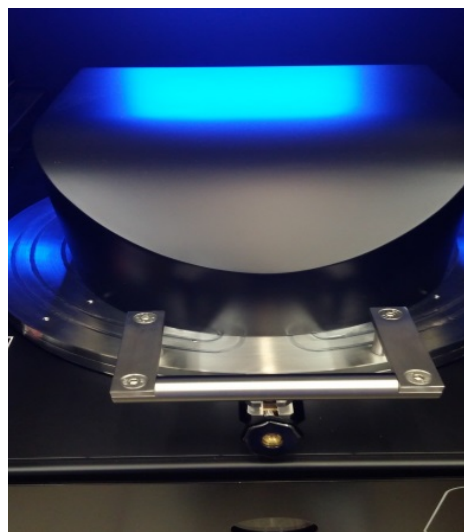


Figure 10. Locked Chamber Lid, Left:



Unlocked Chamber Lid, Right

- 16- Place your substrate, centering it with respect to the chuck. **NOTE:** If you ran the clean or another sample previously then the chamber may be **HOT!** Carefully close the chamber lid and tighten the locking mechanism.
- 17- Slowly lower the enclosure door. **The chamber enclosure door must be closed.**
- 18- Shut the vent line by left clicking on *Vent Valve*
- 19- Then, open the Pump valve by left clicking on *Open*. **Wait 5 minutes.**
- 20- While you're waiting you can change the process gases, if necessary (typically replacing Acetylene with Methane). Default Gases are shown on left. (Figure 11) Double left click *Acetylene* to open window shown on right.

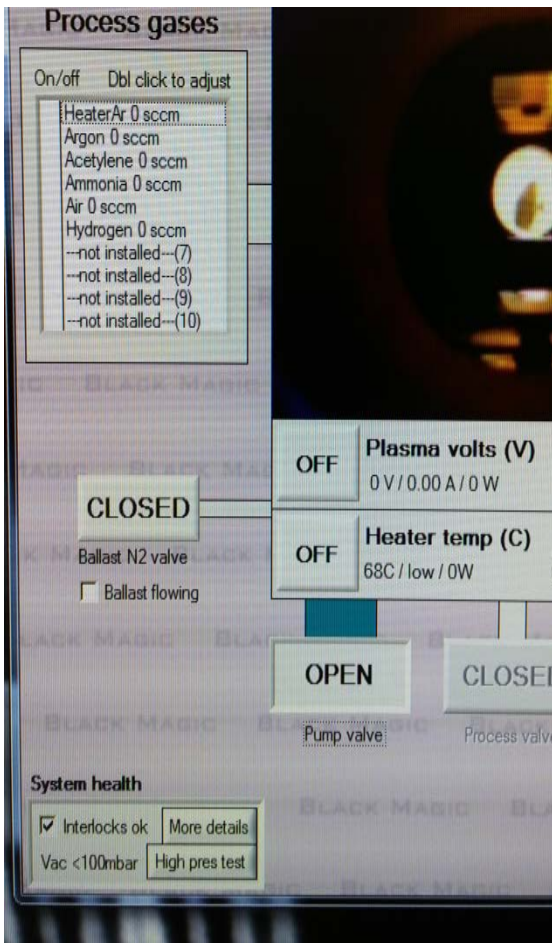
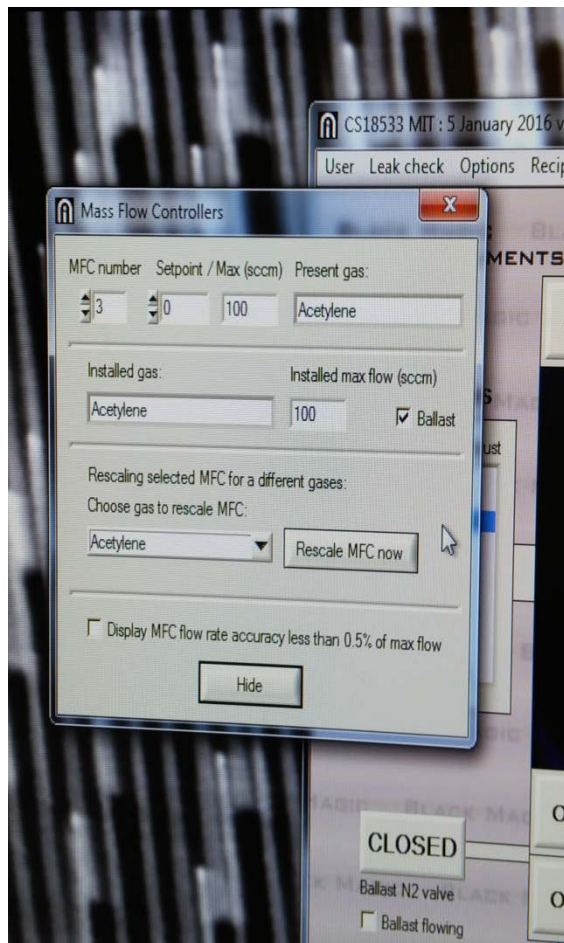


Figure 11. Default Gases, Left



After Selecting Acetylene, Right

- 21- Left Click *Acetylene* to open a pull-down menu and select *Methane* (Figure 12, left). After selection, left click *Rescale MFC Now* to complete replacement (Figure 12, right.) Then Left Click *Hide*.

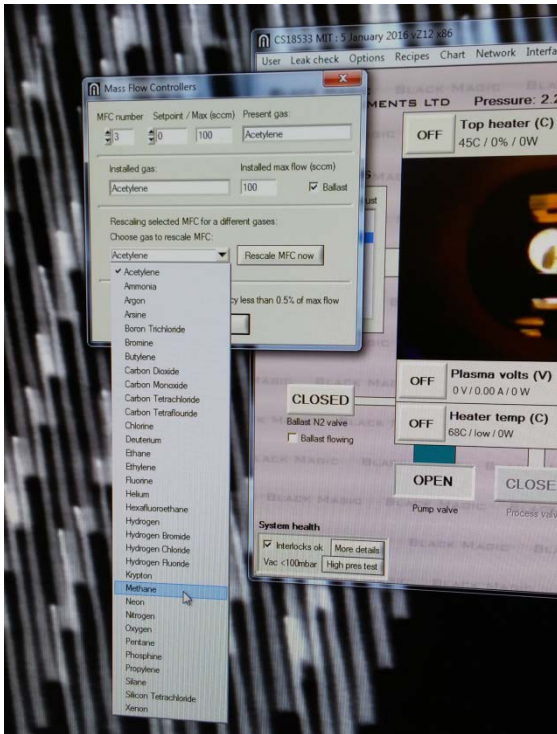
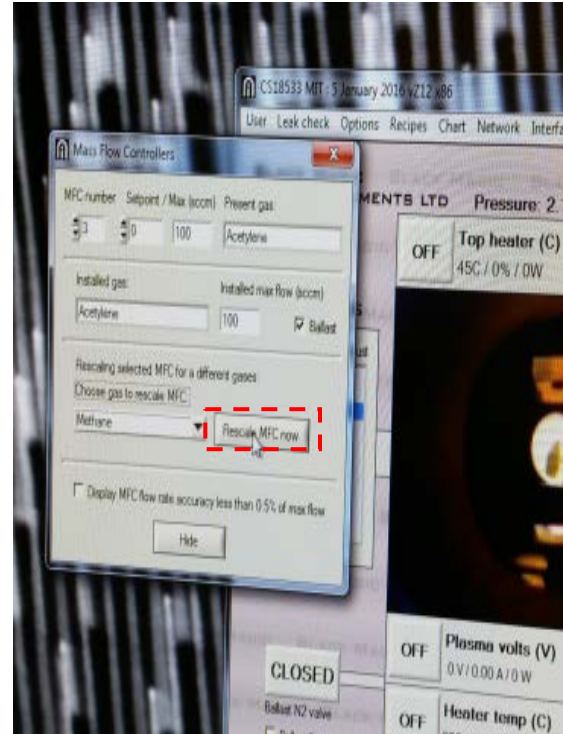


Figure 12. Select Methane from menu, Left



Click Rescale MFC Now, Right

- 22- On the upper Toolbar, left click *Leak Check*, Prompted to do Low Pressure Leak Check? Then, a small window will open. (Figure 13, Left).
- 23- Click *Start* and wait until it passes leak check (Figure 13, Right). When it passes, Click *Hide*. **System must pass for you to proceed! If not, return to Step 12 and try again. If it fails a second time, STOP and shutdown the tool in CORAL!!**

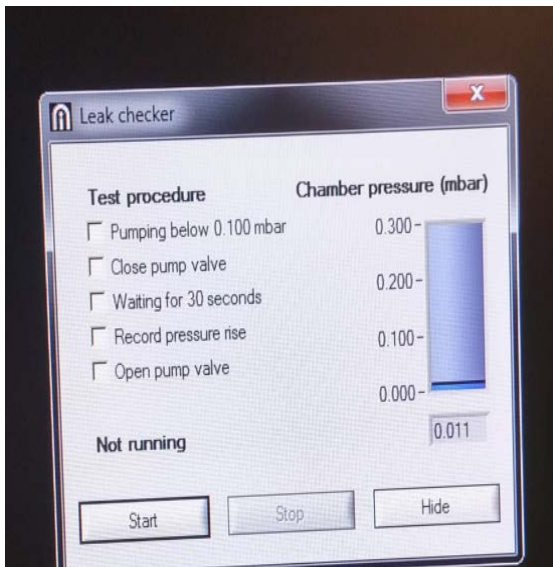
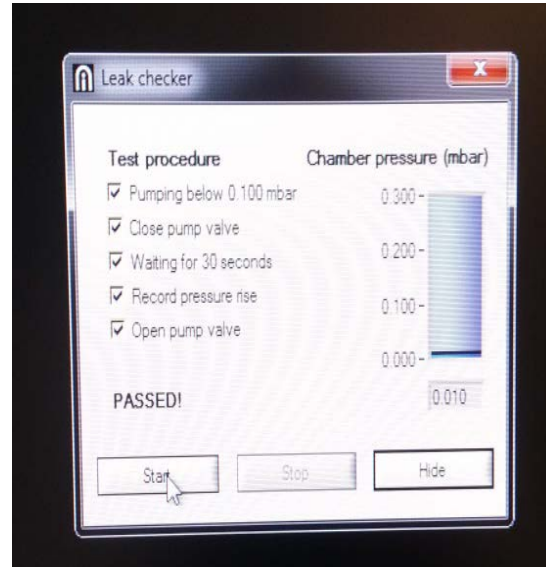


Figure 13 Leak Check, Left



Passed Leak Check, Right

- 24- Click *PROCESS*. A red dot appears above In Process. Also a red PROCESS STOP button appears (Figure 14). For the system to go into Process mode:
- The pump valve must be open.
 - The chamber pressure must be less than 100 mbar.
 - The chamber door and chamber enclosure door must be closed.
 - The chiller must be working.

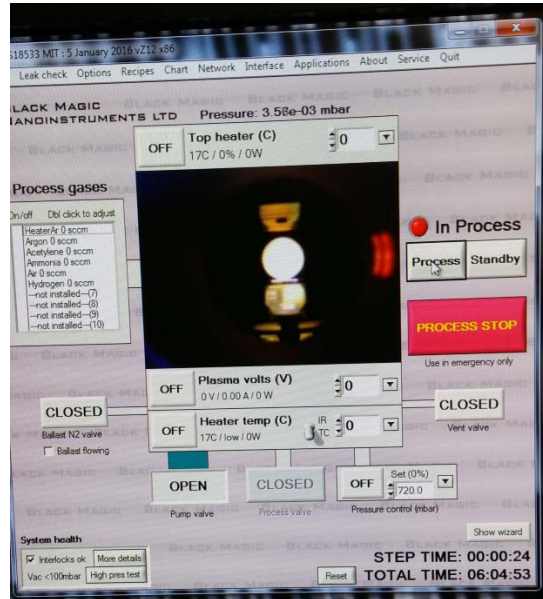


Figure 14

- 25- Left click on *Show Wizard* button (Figure 15, left). A window on the right of the screen should appear. Left click on *Load* (Figure 15, center). Select the folder and recipe that you want to use. (Figure 15, right)

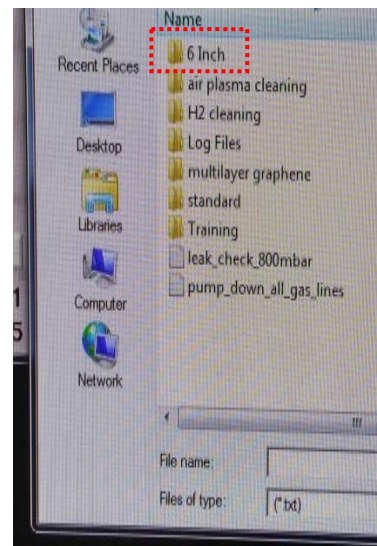
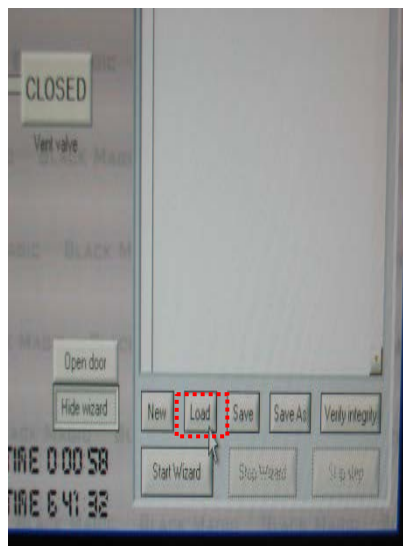
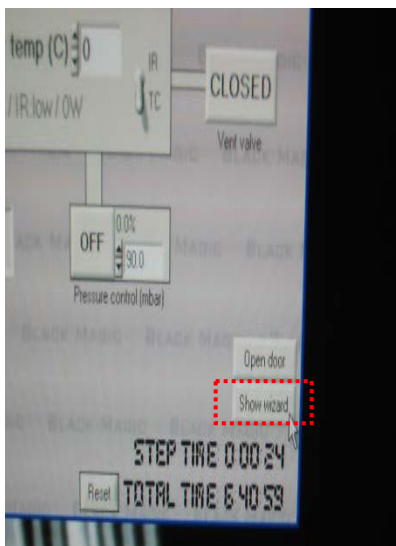


Figure 15. Open sequencer (left),

load recipe (center),

and select recipe from menu (right)

26- The recipe is uploaded in the sequencer. Left click the *Verify Integrity* button (Figure 16, left), A Recipe Compiler window opens (Figure 16, right). Make sure that the tool agrees with every single step of the process. Left click the *Hide* button. If there are any problems with the verification, report the problem in CORAL immediately and don't continue with the process.

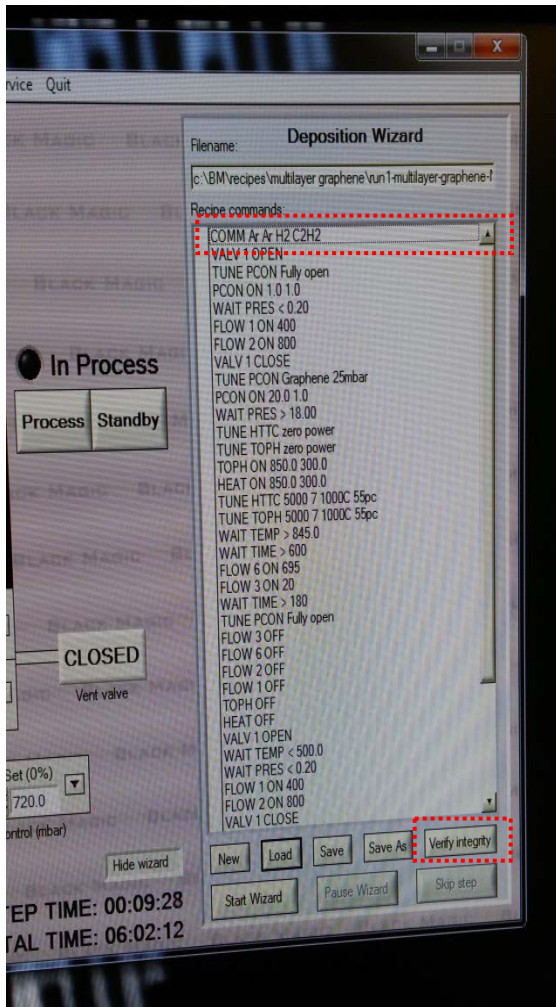
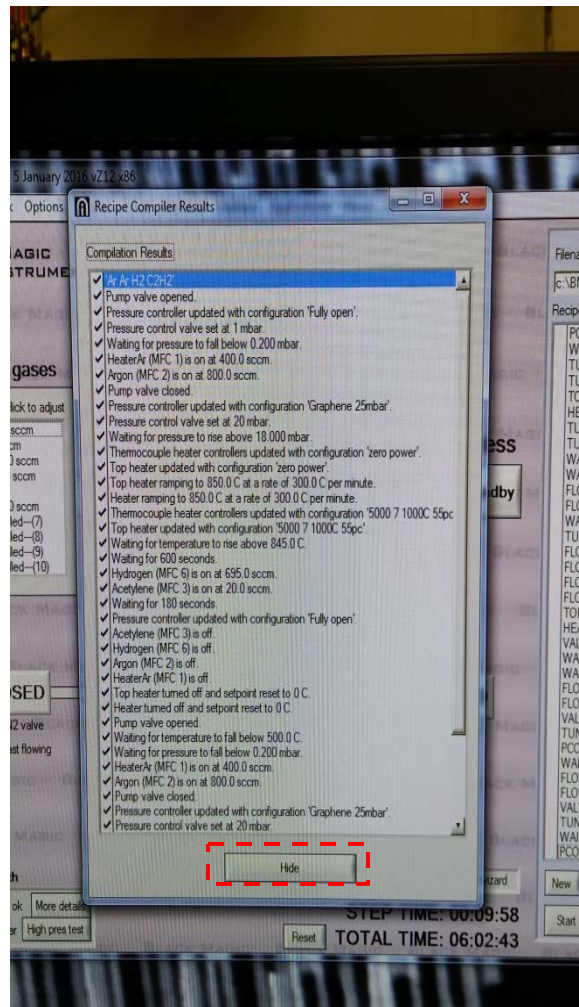


Figure 16. Verify recipe (left),



Close verification menu (right)

27- Also in Figure 16 left, notice the Comment in step 1, telling what gases are required for this recipe. Go to the gas box on the right side of the tool and make sure the appropriate ball valves are open for the recipe gases. See Figure 17



Figure 17

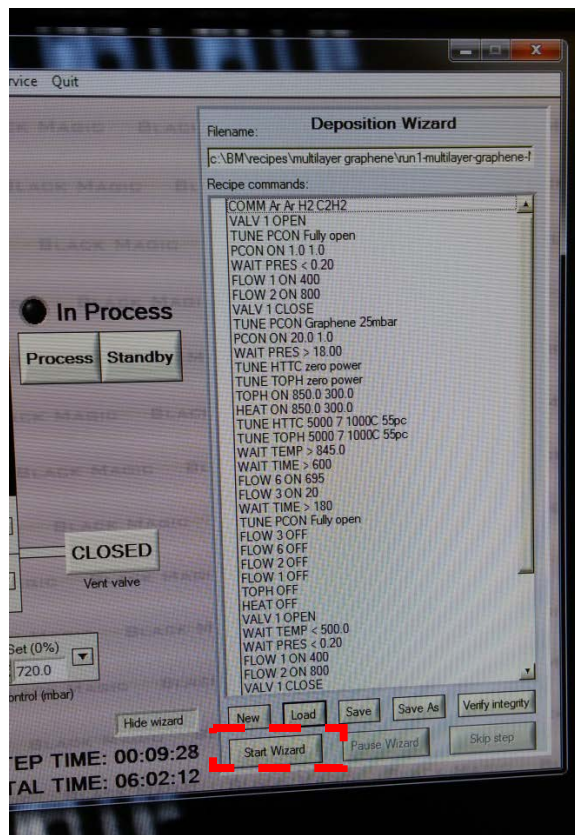


Figure 18

- 28- Left click the button *Start Wizard* (Figure 18).
- 29- A Data Recording Window will open and the tool will ask you if you want to save the run data. You should say YES, and name the following format: “Date (yyyy-mo-day)_run #_Processname_Gas_YourInitials. (i.e. **2018-06-15_run2_Monolayer950_CH4_RB**) The tool will save the real-time data of the run in the Log File. Those parameters are very useful for the Staff to plan maintenance, and ensure that the tool runs smoothly. The computer of the tool is not part of any network nor does it have a wireless connection available. If you need the data of the run, you will need to contact the Staff to retrieve it for you. **You must not attempt to take the data yourself.**
- 30- **You MUST remain with the Tool while the Heater is ON!!!!** The sequencer will start and the tool will show you what steps have been completed. The only option that you have at this point is to stop the sequencer by left clicking the *Stop Wizard*, or left click the *Process Stop* button, which will bring the tool to Standby. If the sequencer is stuck at any step, for example, it does not produce plasma, or the base vacuum level is not reached, notify the Staff immediately using CORAL. These conditions indicate a problem with the tool or the cleanliness of the reactor. *In either case, the reactor will require several hours to cool down before any inspection and diagnosis can be made. The tool will be down while the problem is assessed.*
- 31- After the growth is completed, the tool will conduct a cool down using Argon. The sequencer will stop when the TC thermocouple registers 200 °C. You can leave the tool while the reactor is cooling. A typical cooling time is 45-60 min.
- 32- After the cooling down is completed, left click the *Standby* button.

- 33- Then, left click on the *Pump Valve* button to close the access to the pump.
- 34- The chamber can be vented if:
 - e. The tool is in Standby mode.
 - f. The reading in the TC thermocouple is less than 200 °C
 - g. The pump valve is closed.
- 35- After that, left click on the *Vent Valve* button.
- 36- The chamber will slowly vent with Argon gas. Look in the Pressure field, it will indicate *Vented/Unlocked*
- 37- Manually raise the enclosure door. Loosen the Chamber Lock and tilt the mechanism toward you. Carefully lift the chamber lid (it is heavy).
- 38- Remove your substrate; caution, chamber is still likely hot! Carefully close the chamber lid and tighten the locking mechanism.
- 39- Slowly lower the enclosure door. **The chamber enclosure door must be closed.**
- 40- Shut the vent line by left clicking on *Vent Valve*
- 41- Then, open the Pump valve by left clicking on *Open*. Wait 5 minutes.
- 42- Left click on the *Quit* button located on the upper right of the main menu. Left click *Quit to Windows*
- 43- Turn off the Monitor.
- 44- **Fill in the log book with your run information.**
- 45- Close the orange valve behind the tool.
- 46- Close the process gas box valves—Leave the Argon valves as you found them!!
- 47- Go to the CORAL terminal and fill-in the information of your run. Include in the Comment field a detailed description of the catalyst and substrate you used. “Disengage” the machine in CORAL.
- 48- Notify Staff to shut off the gases in the cabinets.
- 49- **NOTE:** The chiller remains on until the chamber is cool (usually overnight).

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