



STANDARD OPERATING PROCEDURE

AIXTRON (*formerly* NanoInstruments) Carbon Nanotube Deposition System

CORAL Name: CCNT

Model Number: Black Magic Pro

Location: 39-428a TRL

What it does: Plasma Enhanced Chemical Vapor Deposition (PECVD) of Carbon Nanotubes (CNTs)



Figure 1. The TRL PECVD CNT Reactor (CCNT)

INTRODUCTION:

This system is designed to grow multi-walled CNTs (MWNTs) and single-walled CNTs (SWNTs) that are perpendicular to the substrate. The CNTs are attached to the substrate, so they can be grown in specific locations and be part of devices. The growth occurs at 2 – 4 Torr 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, Iron or Cobalt. Representative catalyst thicknesses for MWNT growth are 1 – 15 nm, and 1 – 3 Å for SWNT growth. The primary process gases are Acetylene, Ammonia and Argon, but Methane and Hydrogen are available too. Currently only recipes that use the primary process gasses have been developed. This tool accepts substrate pieces as large as 6-inch wafers, and it has segregated reactor quartzware and tweezers for gold-contaminated or CMOS-compatible processes. However, **the default state of the tool is gold-contaminated**, and a request to PTC would be needed well in advance to obtain an approval to convert the reactor to CMOS-compatible.

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 technical 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 CNT deposition, the reactor chamber will require a thorough cleaning before further use. **Users are not allowed to perform cleaning procedures on the tool.** 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 run can be completed within 1.5 hours, including cooling.* The tool is in “mission critical” status. **Therefore, the tool can only be reserved from 8 AM to 2 PM.** The tool will be dedicated for its mission critical use after 2 PM. This restriction will be in effect until further notice.



Figure 3. Process Gasses 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 gasses open.

Log on:

- 3- ‘Engage’ the CCNT 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 *user* account. The login password of this account is *user*. The screen should now look like in Figure 4:

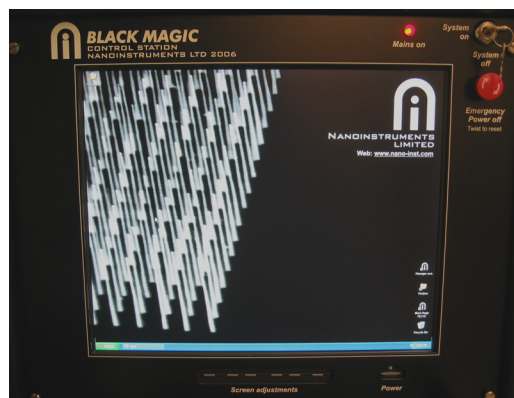


Figure 4. Initial Screen



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 train you and to create an account for you.*
- 8- Left click on *Password* and type your password.
- 9- Left click *Log On*

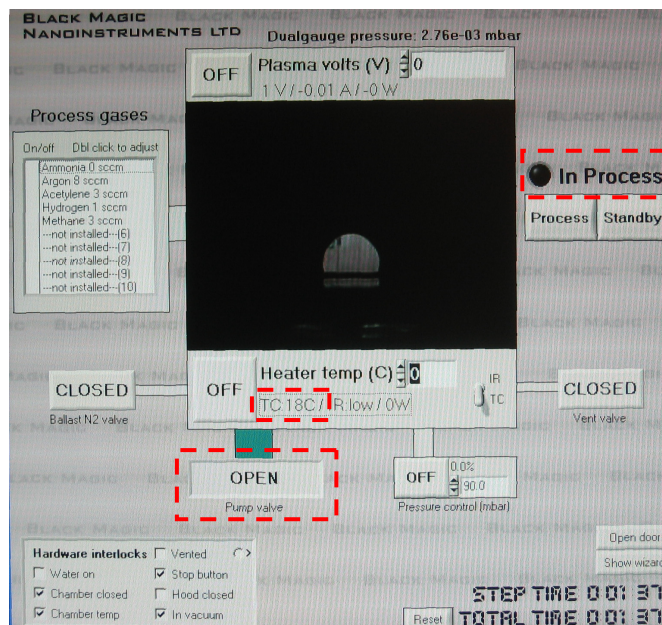


Figure 6. Initial Screen within running tool

- 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 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.**

Venting and Loading:

11- Left Click on the *Pump Valve* to close it (Figure 7, left).

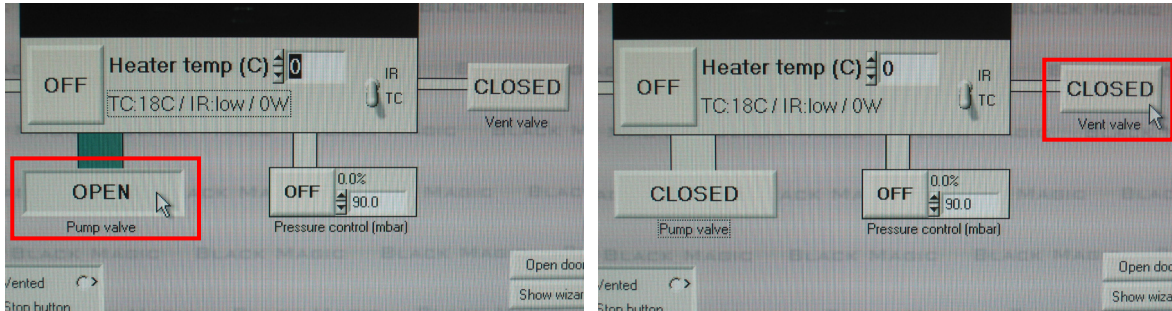


Figure 7. Close pump valve (left), then open vent valve (right).

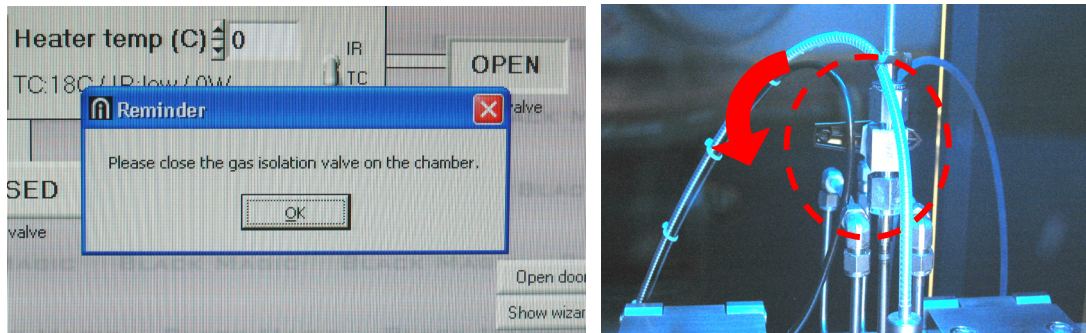


Figure 8. Close gas isolation valve request (left), and isolation valve (right)

- 12- Left click *Vent Valve* to open it (Figure 7, right). A Reminder Window will open asking you to close the gas isolation valve (Figure 8, left). This is a manual ball valve located inside the enclosure and above the chamber. To close the gas isolation valve:
 - a. Open the chamber enclosure door and verify that the valve is shut, i.e., perpendicular to the gas line (Figure 8, right). Close the chamber enclosure door.
 - b. THEN, left click on *OK*.
- 13- The chamber will slowly vent with Nitrogen gas. Look for the *Vented!* indicator in the Dualgauge Pressure field (Figure 9).
- 14- Left click on *Open Door* (Figure 10, left)—a Door Controls Window will open. The *Door Fully Closed* red indicator must be present (Figure 10, center). To open the door, make sure the *Open* direction is selected, and then left click on *Start*. The lid will open automatically. **CORAL must be engaged in order to raise the chamber lid. Also, the chamber enclosure door must be closed.** The *Door Fully Opened* red indicator must appear at the end of the step (Figure 10 right). If not, check again the direction of movement of the door is *Open* and push *Start* again. Don't open the chamber enclosure door UNTIL the *Door Fully Opened* red indicator appears.

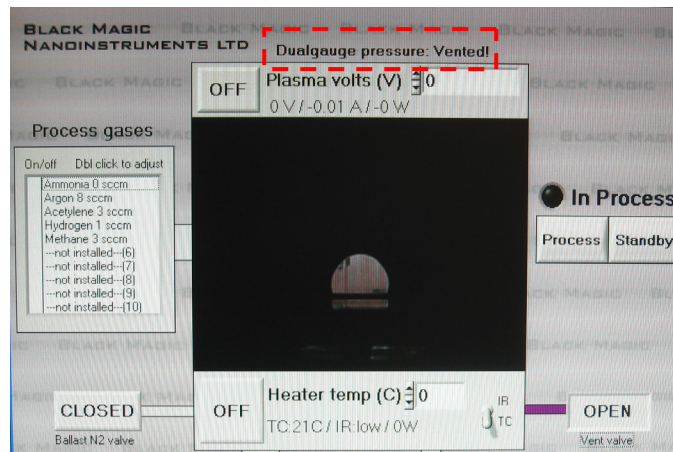


Figure 9. The reactor chamber is vented

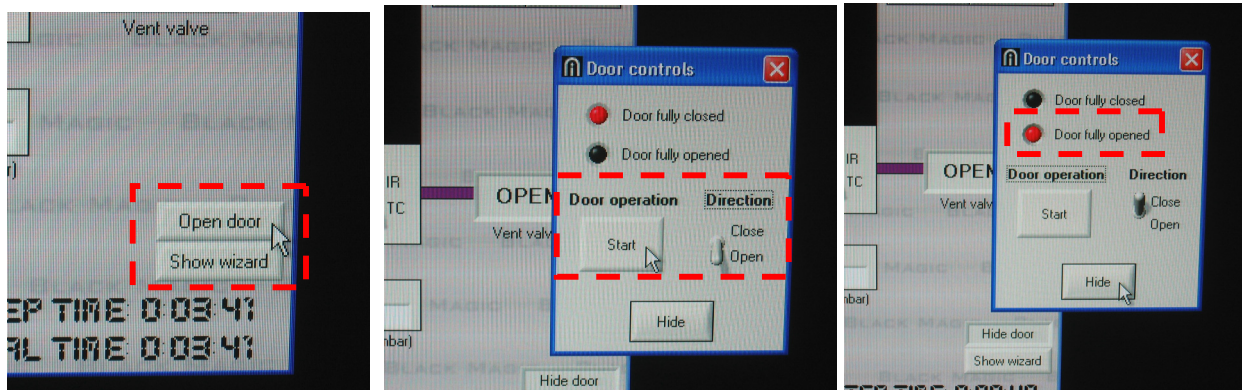


Figure 10. Open door button (left), initial door menu before opening (center), final door menu after opening (right).

15- Open the chamber enclosure door and place your substrate, centering it with respect to the chuck (Figure 11). Close the chamber enclosure door.



Figure 11. Reactor chamber is open (left), reactor chuck without substrate (center), and with substrate (right)

16- To close the door, make sure the *Close* direction is selected, and then left click on *Start* (Figure 12, left). The lid will close automatically. **The chamber enclosure door must be closed.** The *Door Fully Closed* red indicator must appear at the end of the step. If not, check again that the direction of movement for the door is *Close* and push *Start* again.

Don't proceed with your process UNTIL the *Door Fully Closed* red indicator appears.

17- Left click on *Hide* to hide the Door Controls Window.

18- Shut the vent line by left clicking on *Vent Valve* (Figure 12, center)

19- Then, open the Pump valve by left clicking on *Open* (Figure 12, right)

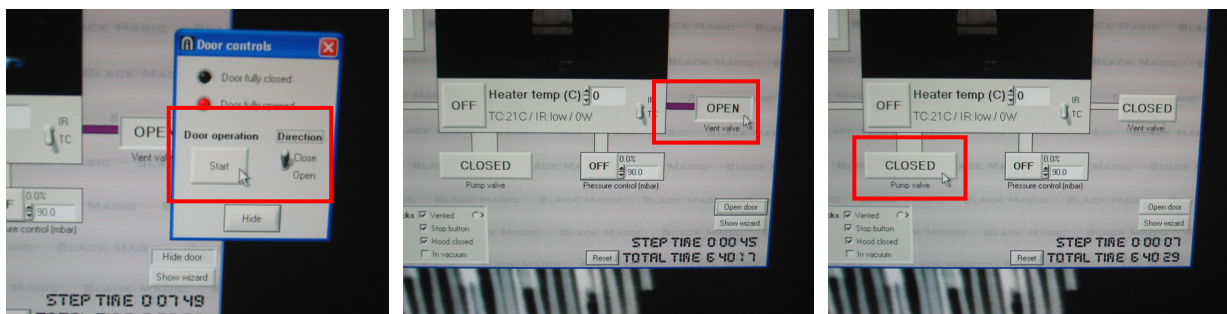


Figure 12. Close Chamber door (left), close vent valve (center), and open pump valve (right)

20- Left click on *Show Wizard* button (Figure 13, left). A window on the right of the screen should appear. Left click on *Load* (Figure 13, center). There are four recipes in the system (Figure 13, right):

- a. *Nano_Dots_AC*, for catalyst pads smaller than 0.3 μm (Ni) and AC plasma.
- b. *Nano_Dots_DC*, for catalyst pads smaller than 0.3 μm (Ni) and DC plasma.
- c. *Two_Step_AC*, for catalyst pads larger than 0.3 μm (Ni) and AC plasma.
- d. *Two_Step_DC*, for catalyst pads larger than 0.3 μm (Ni) and AC plasma.

The key difference between the nanodots recipes and the two-step recipes results from the fact that the catalyst can move around during annealing. Therefore, the annealing step in the nanodot recipes is a lot shorter. The AC recipes are intended for dielectric substrates, using a physical principle analogous to the principle used in DRIE footing kits to discharge insulating layers at the bottom of the etching. **Select the recipe that you want to use.**

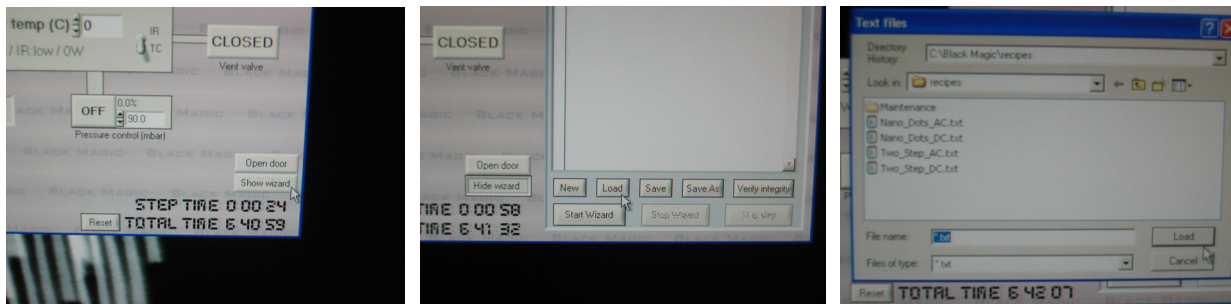


Figure 13. Open sequencer (left), load recipe (center), and select recipe from menu (right)

21- While the sequence is uploaded into the system, the computer will ask you for the annealing time (Figure 14, left –only in two-step recipes) and the growth time (Figure 14, right). These are the only parameters that can be varied from the recipes. **The recipes must not be overwritten.** The annealing and growth time must be declared in seconds. The annealing time can vary from 1 second and 10 minutes, while the growth time can vary from 1 second to 30 minutes. A typical annealing time is 5 minutes. The growth is linear in the range provided by the sequencer. As a guidance, there is a set of SEMs and a calibration for 7 nm thick Ni on top of silicon, annealed for 5 minutes. In this case, the growth is about 0.1145 $\mu\text{m}/\text{min}$. Thicker catalyst films result in taller (although slightly fatter) CNTs, and other catalysts such as iron are more active, i.e., they can grow taller CNTs in the same amount of time. Each catalyst stack and process flow is distinctive enough that you will need to determine the particular growth rate of your process.

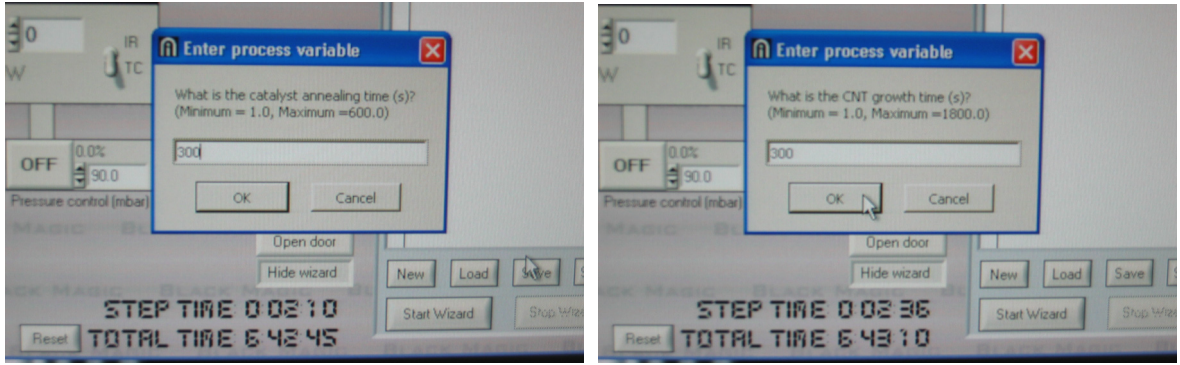


Figure 14. Input annealing time (left), and growth time (right)

22- The recipe is uploaded in the sequencer. Left click the *Verify Integrity* button (Figure 15, left), and make sure that the tool agrees with every single step of the process. Left click the *Hide* button (Figure 15, right). If there are any problems with the verification, report the problem in CORAL immediately and don't continue with the process.

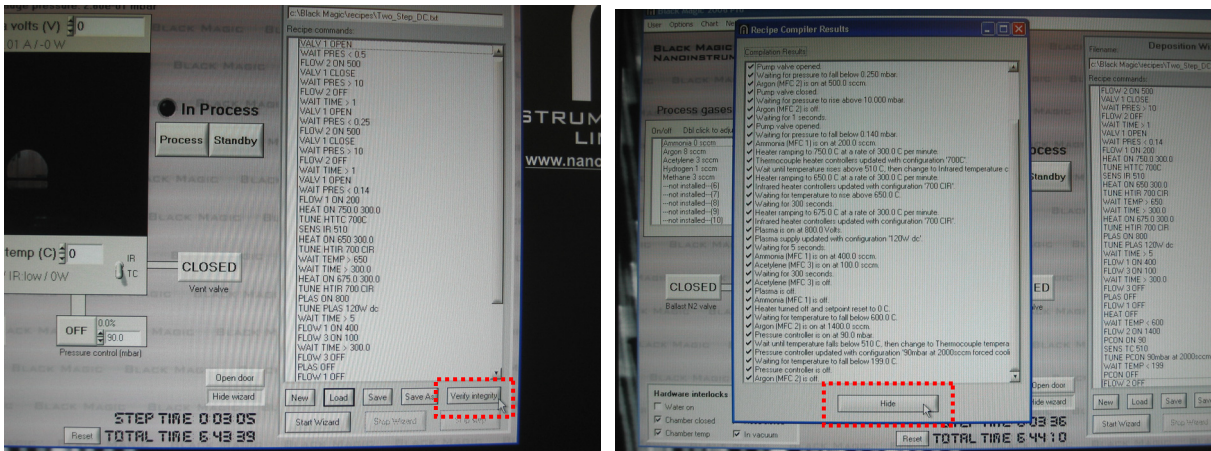


Figure 15. Verify recipe (right), close verification menu (right)

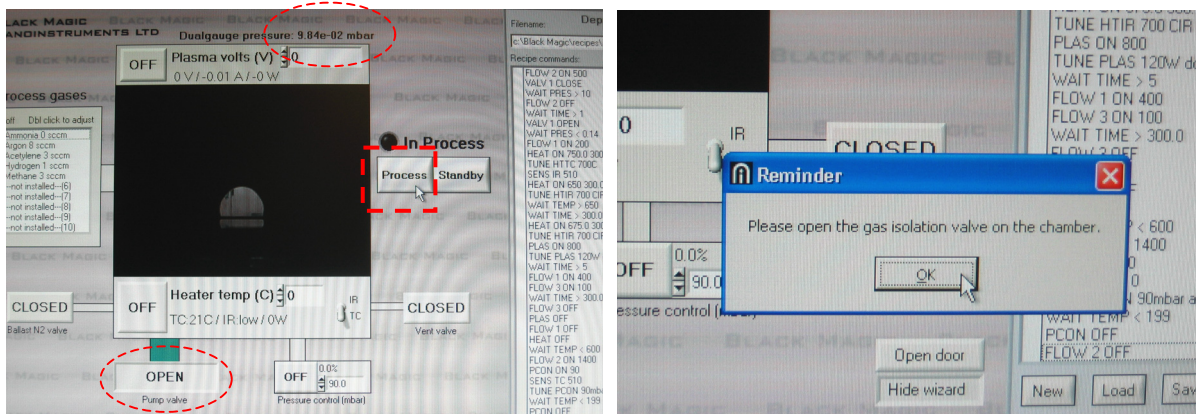


Figure 16. Left click Process button, (left), and request of opening the gas isolation valve (right)

23- Once the recipe is verified, you are ready to go. Left click the *Process* button (Figure 16, left). For the system to go into Process mode:

- The pump valve must be open.
- The chamber pressure must be less than 100 mbar.

- c. The chamber door and chamber enclosure door must be closed.
 - d. The chiller must be working.
- 24- A Reminder Window will appear, asking you to open the gas isolation valve (Figure 16, right). To open the gas isolation valve, first open the chamber enclosure door. Next, open the valve on top of the reactor chamber (Figure 17, left). Finally, close the enclosure door. THEN, left click OK on the Reminder Window. The indicator for the *In Process* status should turn red, and the screen should also include the *Panic Button* (Figure 17, right).

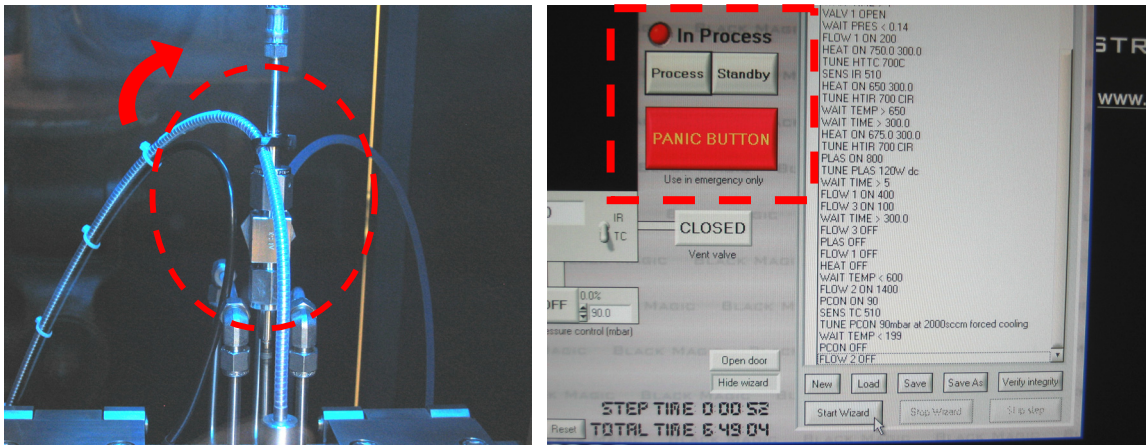


Figure 17. Open gas isolation valve (left), and *In Process* status (right)

25- Left click the button *Start Wizard* (Figure 18, left). The tool will ask you if you want to save the run data. You should say YES, and name the following format: “your login name” “month day year” “growth time” “DC/AC”.txt (Figure 18, right). For example, *John January 1 2008_10 min DC.txt* means that the user with CORAL login John ran the tool on January 1st of 2008 for a growth time of 10 minutes using DC plasma. The tool will save in the file the real-time data of the run. Those parameters are very useful for the staff to plan maintenance, and run the tool smoothly. From time to time, the HDD of the tool will be cleaned as maintenance. 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.**

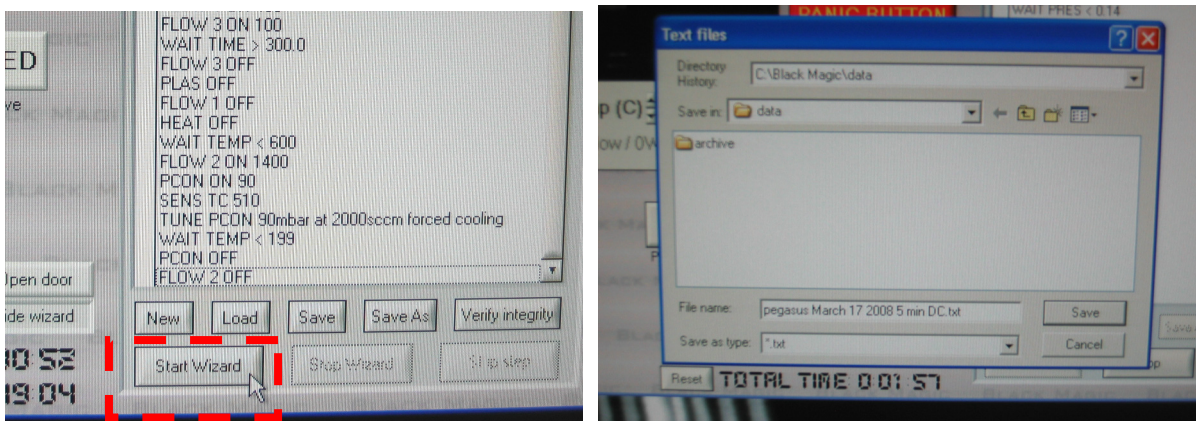


Figure 18. Start sequencer (left), and save run data (right)

26- The sequencer will start by making two purges, then reach a base pressure (Figure 19, left), and then perform the annealing (Figure 19, right), and growth (Figure 20, left). As the sequencer advances, the tool will tell you what steps have been completed. The only option that you have at this moment, is to stop the sequencer by left clicking the *Stop Wizard*, or left click the *Panic* 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.*

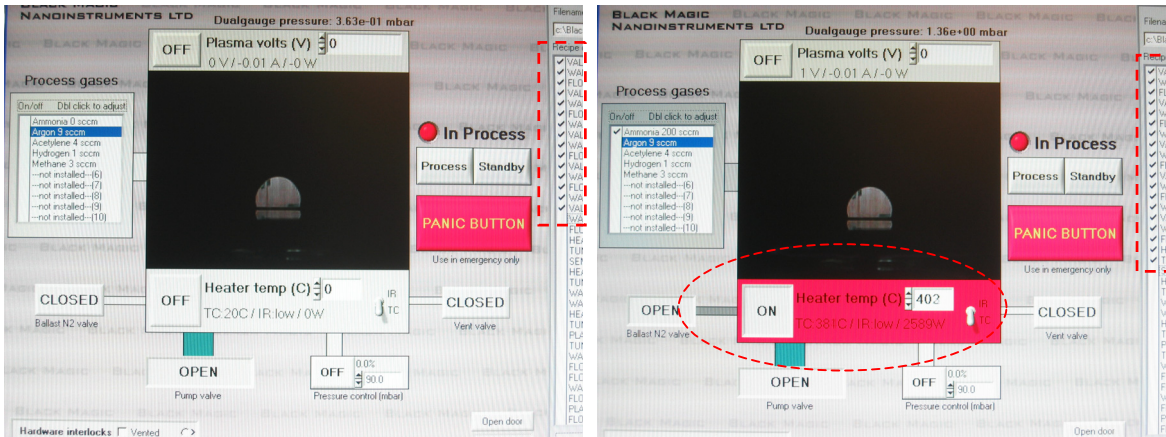


Figure 19. Reaching base vacuum pressure (left), and producing annealing to the substrate (right)

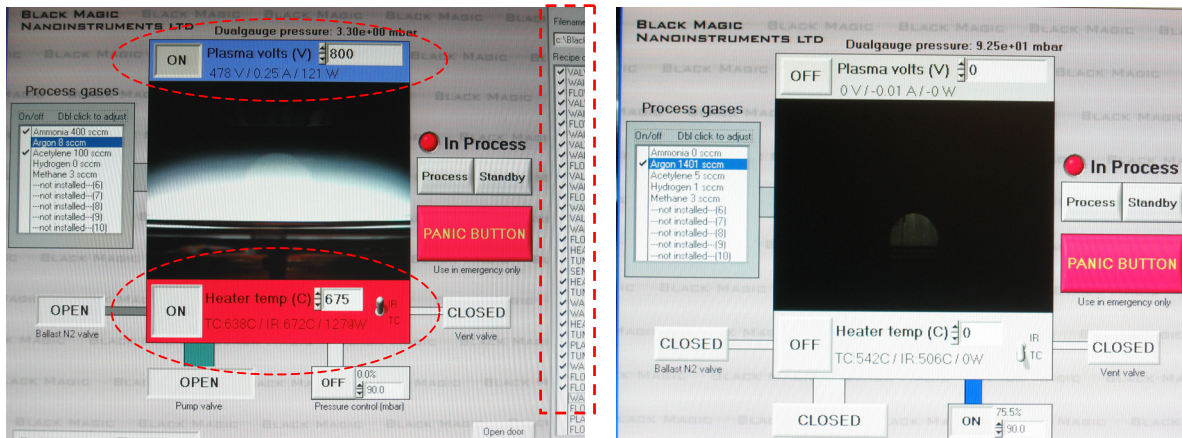


Figure 20. PECVD CNT growth (left), and substrate cooling down (right)

27- After the growth is completed, the tool will conduct a cool down using Argon (Figure 20, right). The sequencer will stop when the TC thermocouple registers 200 °C. **You CANNOT leave the tool unattended from the beginning of your process to the moment when the heater turns off right after the CNT growth is completed.** You can leave the tool unattended while the reactor is cooling after the growth is completed. A typical cooling time is 30 – 45 min.

28- After the cooling down is completed, left click the *Standby* button.

29- Then, left click on the *Pump Valve* button to close the access to the pump.

- 30- After that, left click on the *Vent Valve* button. A Reminder Window will open asking you to close the gas isolation valve. This is a manual ball valve located inside the enclosure and above the chamber. To close the gas isolation valve, first open the chamber enclosure door. Next, close the valve on top of the reactor chamber. Finally, close the enclosure door. THEN, left click OK on the Reminder Window. The chamber can be vented if:
- The tool is in Standby mode.
 - The reading in the TC thermocouple is less than 200 °C
 - The pump valve is closed.

- 31- Once the chamber is vented, left click the *Open Door* button (Figure 21, left). A Door Controls Window will open. The *Door Fully Closed* red indicator must be present (Figure 21, center). To open the door, make sure the *Open* direction is selected, and then left click on *Start*. The lid will open automatically. **CORAL must be engaged in order to raise the chamber lid. Also, the chamber enclosure door must be closed.** The *Door Fully Opened* red indicator must appear at the end of the step (Figure 21 right). If not, check again the direction of movement of the door is *Open* and push again *Start*. Don't open the chamber enclosure door UNTIL the *Door Fully Opened* red indicator appears.

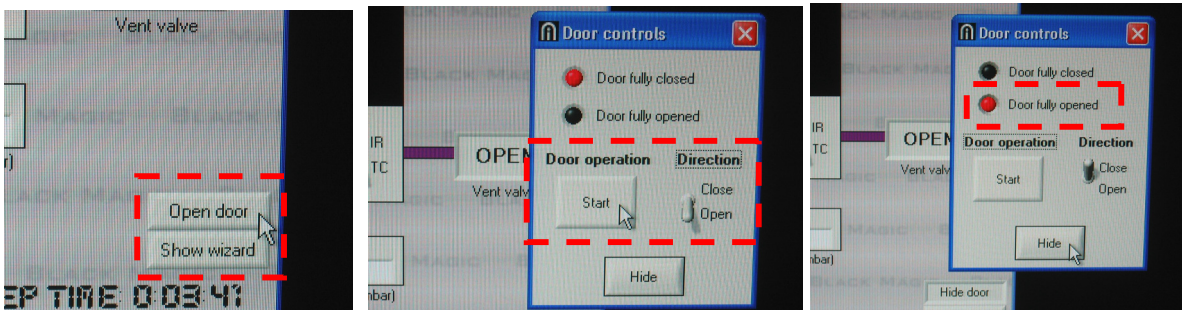


Figure 21. Open door button (left), initial door menu before opening (center), final door menu after opening (right).

- 32- Open the chamber enclosure door and take your substrate out of the reactor. Close the chamber enclosure door.
- 33- To close the chamber door, make sure the *Close* direction is selected, and then left click on *Start* (Figure 21, left). The lid will close automatically. **The chamber enclosure door must be closed.** The *Door Fully Closed* red indicator must appear at the end of the step. If not, check again that the direction of movement for the door is *Close* and push again *Start*. At the end, the *Door Fully Closed* red indicator appears.
- 34- Left click on *Hide* to hide the Door Controls Window.
- 35- Left click on *Vent Valve* to close the vent valve (Figure 21, center)
- 36- Then, open the Pump valve by left clicking on *Open* (Figure 21, right). Wait until the chamber vacuum level displayed in the Dualgauge pressure is 1 mbar.

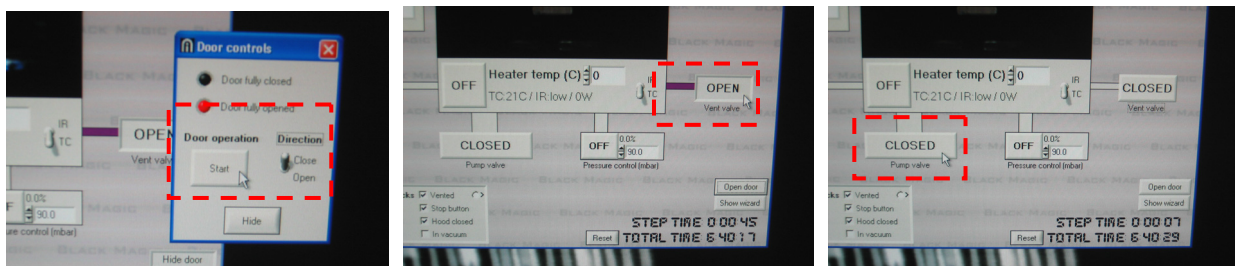
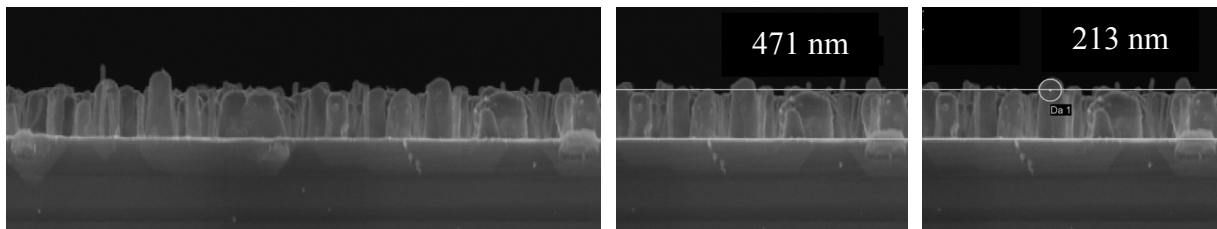


Figure 21. Close Chamber door (left), close vent valve (center), and open pump valve (right)

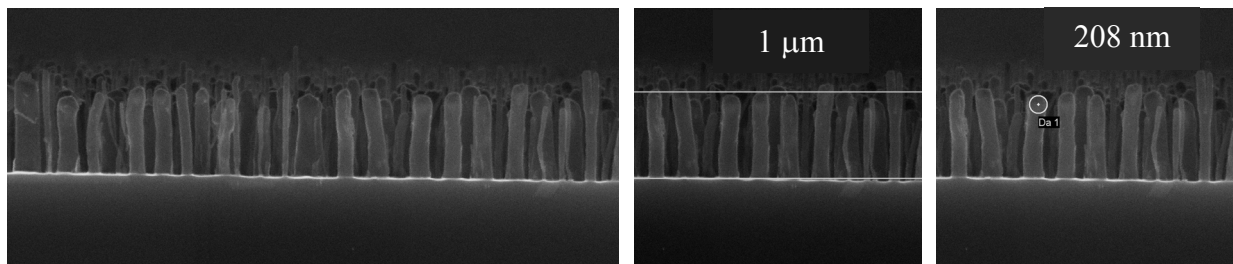
- 37- Once you left the chamber in vacuum, left click on the *Quit* button located on the upper right of the main menu. Left click in *OK, really Quit*. The software should close, and tell you that you can shut down Windows. **Don't shut down windows!**
- 38- Left Click *OK* on the *Turn Black Magic OFF* window.
- 39- Left click on the Start button of Windows. Left Click the *Log off* button, and then left click on *Switch User*. The screen should now show the two Windows accounts that can access the computer.
- 40- Turn off the Monitor.
- 41- **Fill in the log sheet with your run information.**
- 42- Close the orange valve behind the tool.
- 43- 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. **You need to fill-in BOTH the log sheet AND CORAL with the information of your run.**
- 44- "Disengage" the machine in CORAL.

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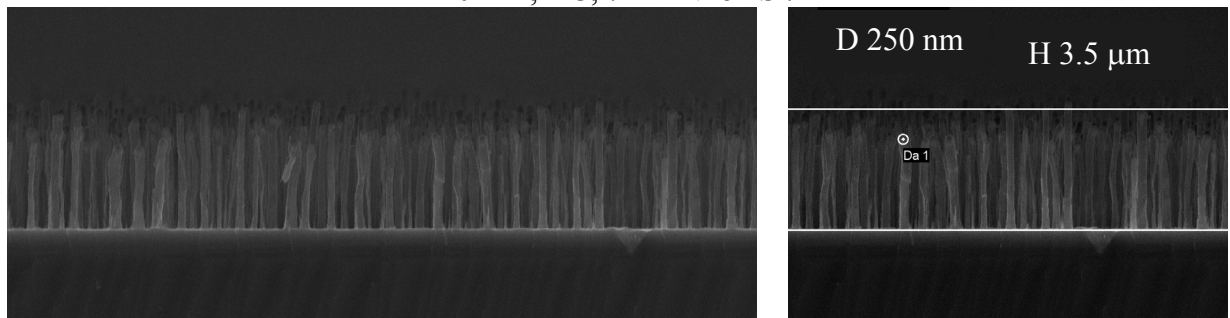
CCNT Baseline



5 min, DC, 7 nm Ni on Si.



10 min, DC, 7 nm Ni on Si.



30 min, DC, 7 nm Ni on Si.

For this set of conditions, $H(\mu\text{m}) = 0.1145 \cdot t(\text{min}), R^2 = 0.995$