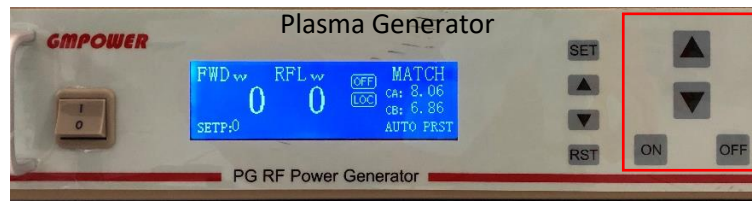


Standard Operating Procedures for 2D-CVD System

updated on August 19, 2020 by Chun-Li Lo



Status before operating:

1. Quartz tube is at vacuum (maybe not base vacuum, but certainly not at atmospheric pressure)
2. Furnace is at room temperature (or < 40 °C)
3. All Solenoid Valves (valves 1 through 7) are closed
4. Vacuum pump (in the chase) is turned off

Before running process:

1. Enable the system on iLab ("2D-CVD" under furnace core)
2. Turn on vacuum pump with the switch (in the chase)
3. Wear solvent gloves. Loosen the ultratorr fitting clockwise about 1-2 turns (no need to fully loosen it all the way)
4. Turn on Switch 3 (N2 Purge) to backfill the tube.

5. Have your hands on the ultratorr fitting to “feel” it push off the end of the tube. Then, set it aside.
6. Turn off Switch 3 (N2 Purge)
7. Wear acid gloves. Manipulate your sample – pull out boat / put on sample / push in boat
8. Take off acid gloves. Reinstall the ultratorr fitting to the quartz tube, and snug down the fitting. Finger tight (counterclockwise) is fine.
9. Turn on Switch 1 (Slow Pump)
10. Wait for tube to pump down to below 1000 mT with the Pirani gauge.
11. Turn off Switch 1 (Slow Pump)
12. Turn on Switch 2 (Fast Pump)
13. Watch to make sure the pressure continues to go down
14. Turn on Switch 3 (N2 Purge) for 5 or 10 seconds, then turn it off
 - a. This purges the tube with N2: Pressure will rise to about 35 Torr with the N2 on, and then return to base pressure once N2 is off. Wait for the pressure to return to 100 mT or less before you continue.
 - b. Note: For the reading of the “35 Torr,” use the display on Throttle Valve Controller to read if Pirani Gauge is not working.
15. Repeat this purge sequence for 10 times.
 - a. At the end of the purge sequence, Switch 3 (N2 Purge) should be off, and Switch 2 (Fast Pump) should be ON.
16. Record the base pressure. Should be around 5-20 mTorr.

Running process:

1. Ramp up temperatures on the furnace controller. Set temperatures on each of the 3 temp controllers and wait for the temperature to reach the set value.
2. Turn on Switch 5 (Ar process); turn on Ar on MFC Controller. Adjust flow rate of Ar and record the pressure.
3. Turn on Switch 7 (H₂S process); turn on H₂S on MFC Controller. Adjust flow rate of H₂S and record the pressure.
4. Wait for 5 minutes to stabilize flow rates.
5. Turn on plasma.
6. Increase plasma power slowly (~ 10 W /10 sec) until reaching the targeted power.
7. **Main process:** wait for required process time. Check the stability of plasma often.
8. Decrease plasma power to 0 W.
9. Turn off plasma
10. Turn off H₂S on MFC Controller. Then, turn off Switch 7 (H₂S process).
11. Turn off Ar on MFC Controller. Then, turn off Switch 5 (Ar process).
12. Turn down temperature of the furnace to 0 °C.
13. Record the base pressure. Should be around 5-20 mTorr.
14. Disable the system on iLab.
15. Wait until the furnace reach room temperature before unloading the sample (takes ~8-10 hours). Fast pump can be either on or off during furnace cooling.

After running process (sample unloading & tube cleaning):

1. Make sure the temperature is below 40 °C before unloading.

2. Enable the system on iLab.
3. If pump is off, do the following steps (a-e). Otherwise, do step 4.
 - a. Turn on vacuum pump with the switch (in the chase).
 - b. This Turn on Switch 1 (Slow Pump).
 - c. Wait for tube to pump down to below 1000 mT with the Pirani gauge.
 - d. Turn off Switch 1 (Slow Pump).
 - e. Turn on Switch 2 (Fast Pump).
4. Turn on Switch 3 (N2 Purge) for 5 or 10 seconds, then turn it off
 - a. This purges the tube with N2: Pressure will rise to about 35 Torr with the N2 on, and then return to base pressure once N2 is off. Wait for the pressure to return to 100 mT or less before you continue.
 - b. Note: For the reading of the “35 Torr,” use the display on Throttle Valve Controller to read if Pirani Gauge is not working.
5. Repeat this purge sequence for 10 times.
 - a. By doing this, you ensure the residual gas left in the tube is safely diluted.
 - b. At the end of the purge sequence, Switch 3 (N2 Purge) should be off, and Switch 2 (Fast Pump) should be ON.
6. Prepare to backfill the tube
 - a. Turn off Switch 2 (Fast Pump)
 - b. Wear solvent gloves. Loosen the ultratorr fitting clockwise about 1-2 turns (no need to fully loosen it all the way)
 - c. Turn on Switch 3 (N2 Purge) to backfill the tube.
 - d. Have your hands on the ultratorr fitting to “feel” it push off the end of the tube. Then, set it aside.
 - e. Turn off Switch 3 (N2 Purge)
 - f. Wear acid gloves. Manipulate your sample – pull out boat / put on sample / push in boat
7. Take off acid gloves. Reinstall the ultratorr fitting to the quartz tube, and snug down the fitting. Finger tight (counterclockwise) is fine.
8. Turn on Switch 1 (Slow Pump)
9. Wait for tube to pump down to below 1000 mT with the Pirani gauge
10. Turn off Switch 1 (Slow Pump)
11. Turn on Switch 2 (Fast Pump)
12. **Run clean process:** Clean Recipe must be run after each sulfurization process.
13. After completing clean process, decrease plasma power to 0 W, turn off plasma, turn off Ar, and set temperature to 0 °C.
14. Keep Switch 2 (Fast Pump) on for 10 minutes, and then turn it off. (Note: Fast pump can be on for more than one day to help reach lower base pressure.)
15. Record the base pressure. Should be around 5-20 mTorr (ideally, < 10 mTorr).
16. Turn off the vacuum pump with the switch (in the chase).
17. Disable the system on iLab.

Tube must be at a low vacuum, with temperature controllers set at 0 °C, and with plasma off before leaving.

After cleaning for 1 hour and overnight pumping, base pressure should be < 10 mTorr. Otherwise, contact staffs.

Recipes:

Clean recipe (**must be run after each process**)

Temperature	Time	Ar (sccm)	Power
800 °C	1 hour	90	70 W

TaS₂ growth (8 nm)

Temperature	Time	Ar/H ₂ S (sccm)	Power	Metal
400 or 800 °C	20 mins	10/10	70 W	~2-nm Ta

MoS₂ growth (~6 nm)

Temperature	Time	Ar/H ₂ S (sccm)	Power	Metal
400 or 800 °C	20 mins	20/10	70 W	~2-nm Mo