

University of Minnesota, MN Nano Center

Standard Operating Procedure

Equipment Name: P1 CVD 2D Film Dep Planartech

Revision Number: 3

Badger Name: TMD 2D film Planartech

Revisionist: Tony Whipple

Model: Planar GROW- 5S

Date: 3/27/2020

Location: PAN, Bay 1

1 Description

The TMD 2D film Planartech is a chemical vapor deposition system (CVD) for growing transition metal dichalcogenides (TMDs) monolayer thin films and single crystals. This CVD system consists of tube 1 and tube 2. At this writing, they are used to grow WSe₂ (Tube1) and MoS₂ (Tube 2) materials, respectively. This first section will refer to processing in Tube 2 for MoS₂ films, for Tube 1 process see the last section.

2. Safety

- a. Be careful when opening the hot furnace for rapid cooling. Don't touch the tube. It can burn your fingers/hand.
- b. It is possible that the reactions used to deposit these materials could produce H₂S or H₂Se. The latter is toxic at sufficiently high concentrations. Be aware of the toxic gas detection system and remain outside the growth area with the doors closed during the deposition and during cool down until the tube has been well purged.

3. Required Facilities

- a. Electrical: 208 VAC (120 VAC for Hydrogen Generator)
- b. Compressed dry air
- c. Process Gas Cylinders: Ar
- d. Process Gas Generator: H₂
- f. Toxic/Combustible Gas Monitoring
- g. Fume hood for removing S/Se based compounds.
- h. House Process Chilled Water (for chamber o-ring)

4. Sample Preparation

- a. Take a 4×4 cm² SiO₂/Si substrate. Clean the substrate using Piranha solution for 1 hour. Piranha solution is prepared by mixing H₂SO₄: H₂O₂ in ratio of 10:1. It is recommended to put H₂SO₄ in a dish, then pour H₂O₂ in the dish with (H₂SO₄) slowly. See details at below link:

http://www.lamp.umd.edu/Sop/Piranha_SOP.htm

- b. After cleaning in piranha solution, place the substrate in HMDS vapor for 5 min.

- c. Decorate small droplets of Perylene-3,4,9,10-tetracarboxylic acid tetra-potassium salt (PTAS) by pouring at different location on SiO₂/Si (see image a) is an option that some do.
- d. After decorating PTAS, heat the substrate at 110 °C to evaporate PTAS (see image b).

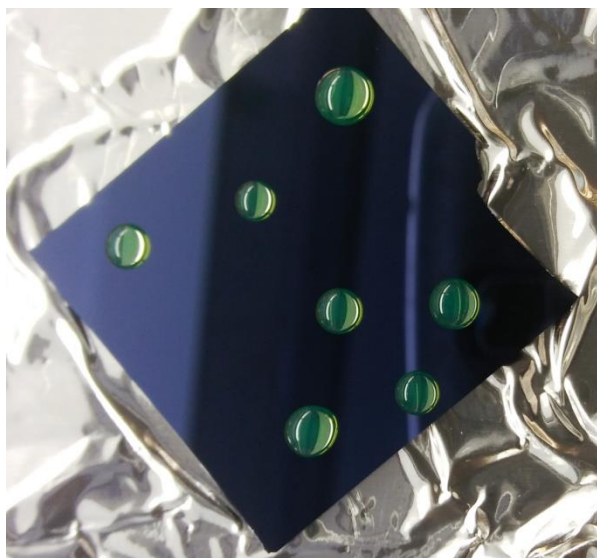


Image 1

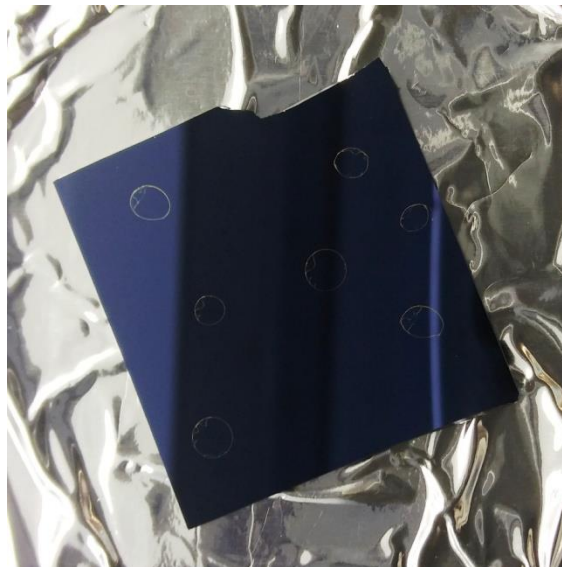


Image 2

5. Operating Instructions

- a. Enable the ‘TMD 2D film Planartech’ in Badger.
- c. First check that the control software is working correctly and is not locked up
- d. Click on the top tab on the far right called ‘MFC (Alicat) READ ‘ image3 then look at the measured data values. If they are all zero then you must stop the control software and start it up again. Image4a A correctly working system will have some values that are not zero, which are good see image4b.

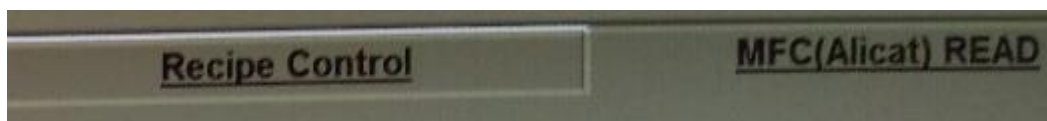


Image3

Measurement Data(Ar)		Measurement Data(H2S)	
0	Absolute Pressure	0	Absolute Pressure
0	Gauge/Diff. Pressure	0	Gauge/Diff. Pressure
0	Barometric Pressure	0	Barometric Pressure
0	Temperature	0	Temperature
		0	Volumetric Flow

Image 4a

Measurement Data (Mo-1)		Measurement Data(Mo-3)	
14.26	Absolute Pressure	14.02	Absolute Pressure
0	Gauge/Diff. Pressure	0	Gauge/Diff. Pressure
0	Barometric Pressure	0	Barometric Pressure
24.07	Temperature	24.18	Temperature

Image 4b

- c. A
- d. On
- e. Click on “VENT.V” icon of system 2 to vent the tube of system 2.

f. In one ceramic boat, take 0.5 mg MoO_3 precursor (see red circle in image 5). In another boat take 200 mg Sulfur precursor (see image 6).



Image 5

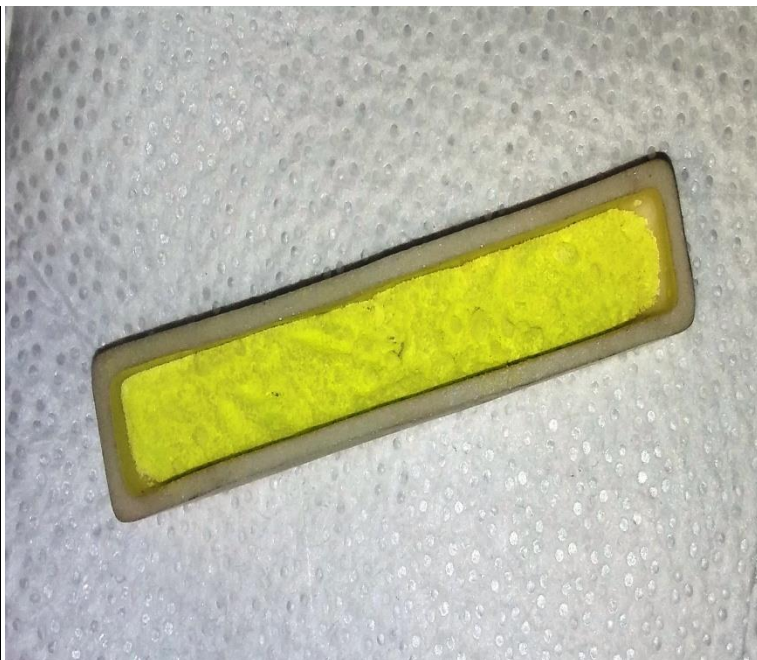


Image 6

g. Mount the substrate (facing down) on the ceramic boat having MoO_3 precursor (see image 7).



Image 7

h. First load MoO_3 boat (having substrate on it) in tube of system 2 with help of a metal rod (the rod is used to push the boat). Push the boat until it reaches a point near to middle of furnace. To be ready for deposition, the MoO_3 boat should be to the right edge of center line of the furnace towards sulfur boat. (see image 8).

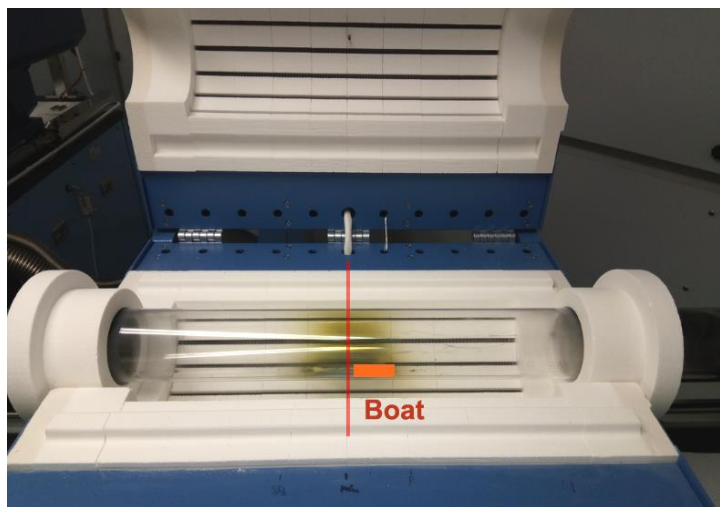


Image 8

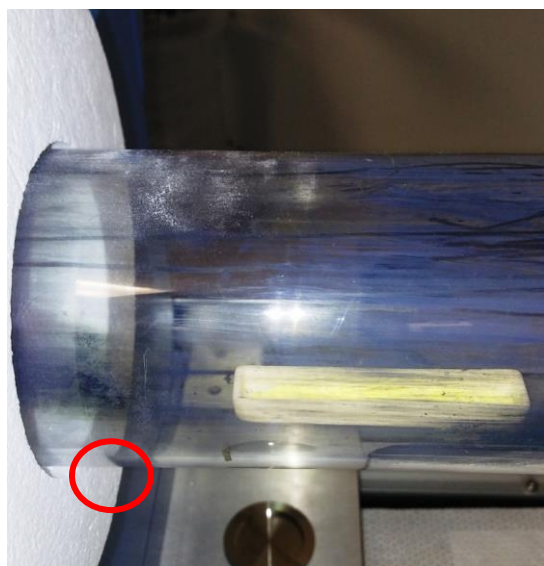


Image 9

- i. Load Sulfur boat in tube 2. Push the boat until it reaches the edge of the white collar, image 9. Remember that it is best to not to have extra powder fall anywhere in the chamber
- j. Close the door of the furnace tube of system 2. Make sure the O-Ring and the flange edge is clean before closing. On “System 2 APC Control” section of software window, select position mode (see arrow in image 12). Then write 25% in “position set” and make sure the right box also have the word POSITION displayed.

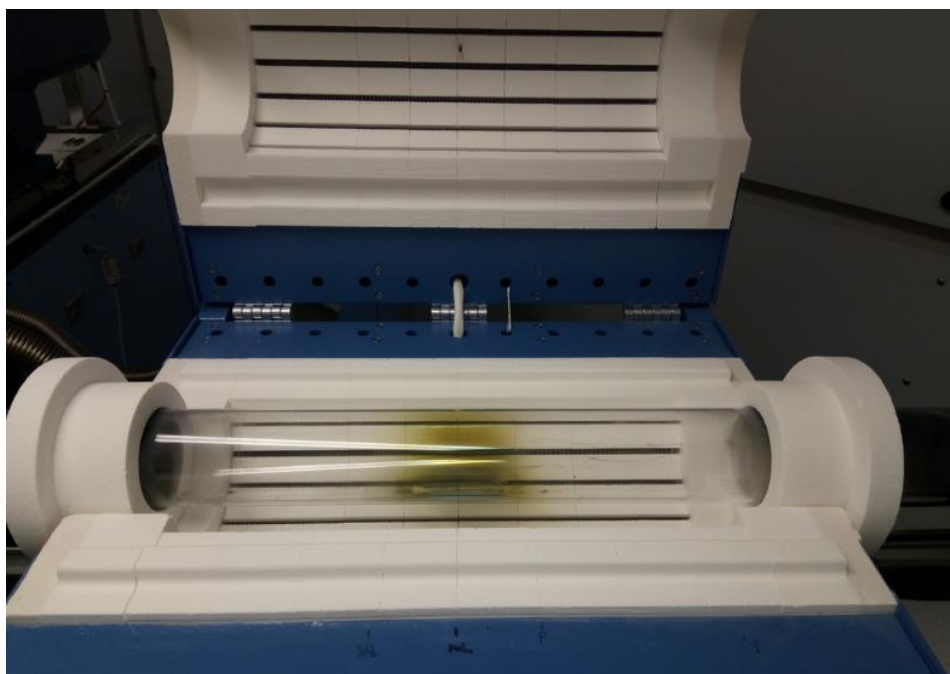


Image 10

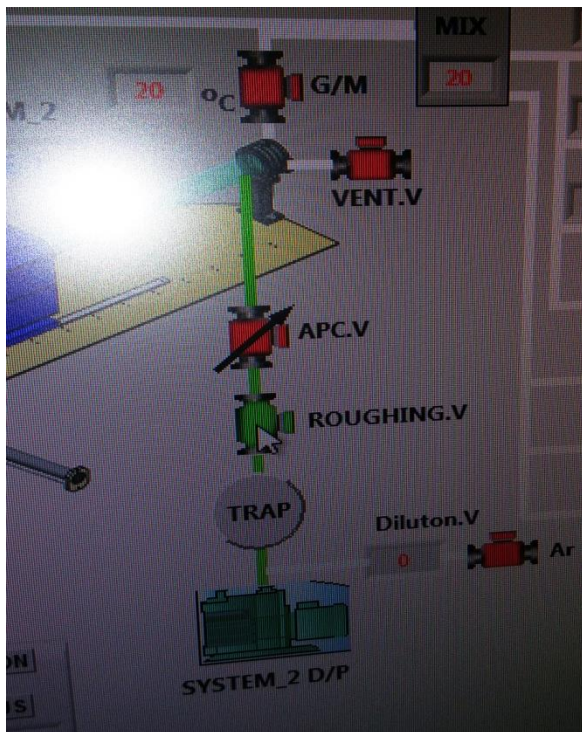


Image 11



Image 12

k. Turn on vacuum pump by clicking on “SYSTEM_2 D/P”. Then Click on roughing valve. You will see a green color in the software (see image 10). Then quickly click “Run” (see arrow in image 14).

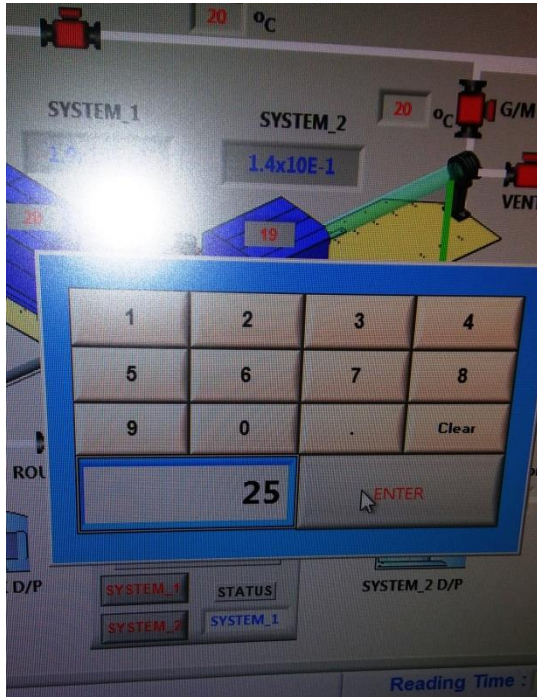


Image 13



Image 14

l. After the pressure reaches 1 Torr, click “OPEN” (see arrow in image 15).

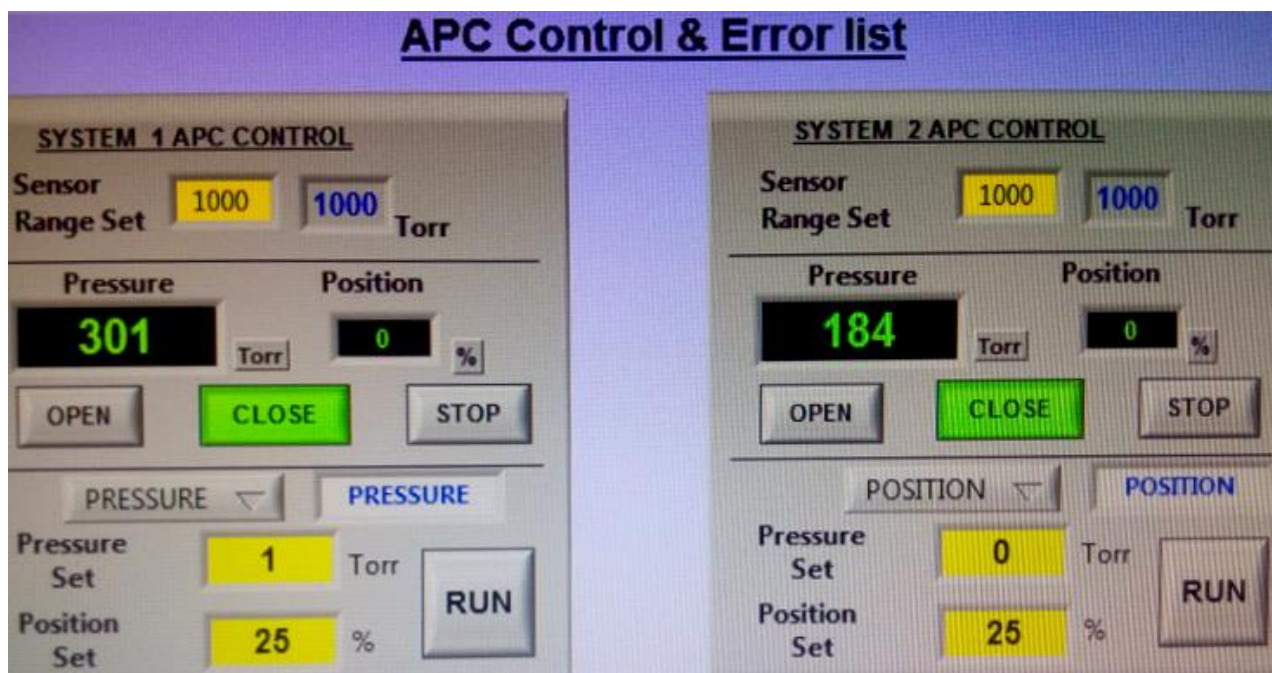


Image15

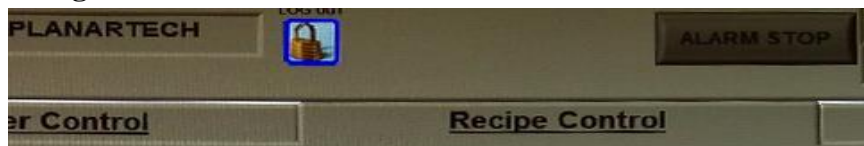


Image 16

m. Click on “Recipe Control” tab (see image 16). A recipe writing section will be opened. Select “System_2 Recipe” (see arrow in image 16). On the “System_2 Recipe” section, click on “Open File” (see arrow in image 17).

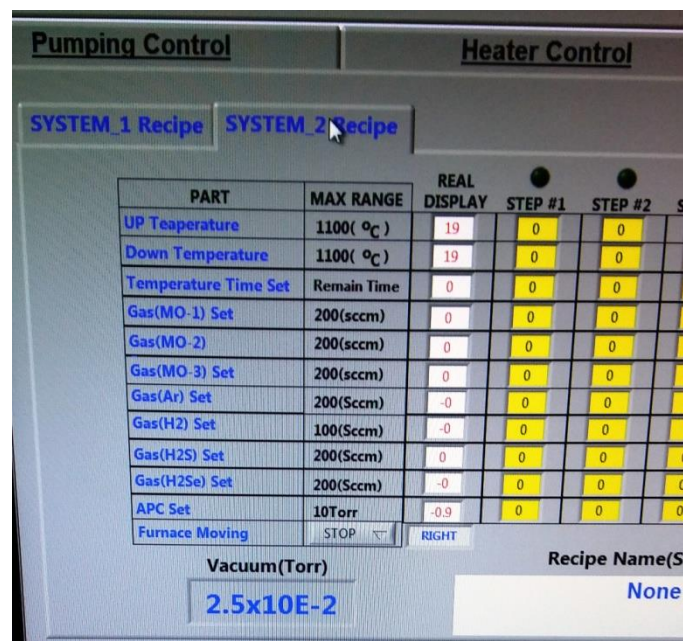


Image 17

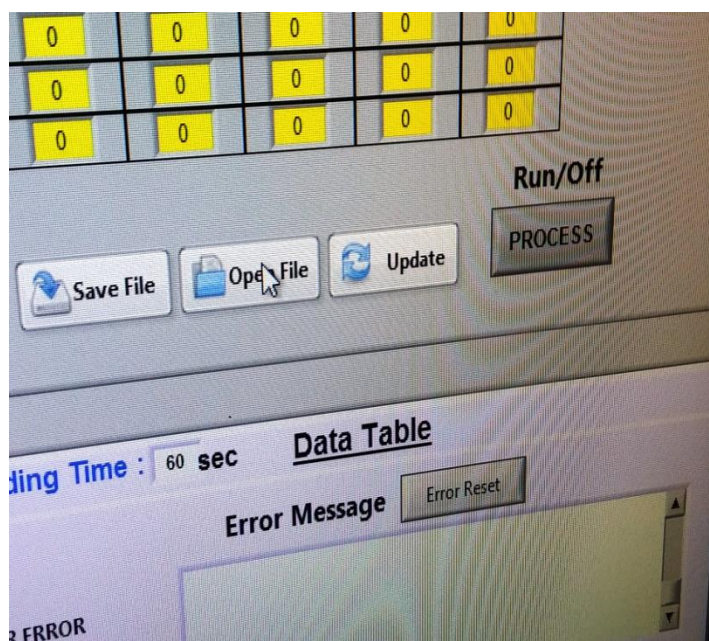


Image 18

n. After clicking on “Open File”, a new window will pop up. Select “Recipe MoS₂ 1” on pop up window. After that click “OK” (see arrow in image 18).

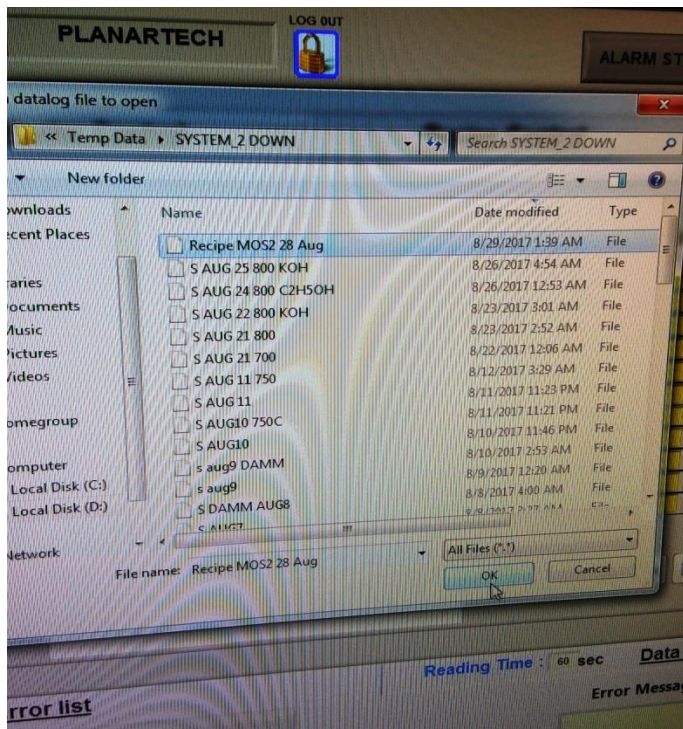


Image 19

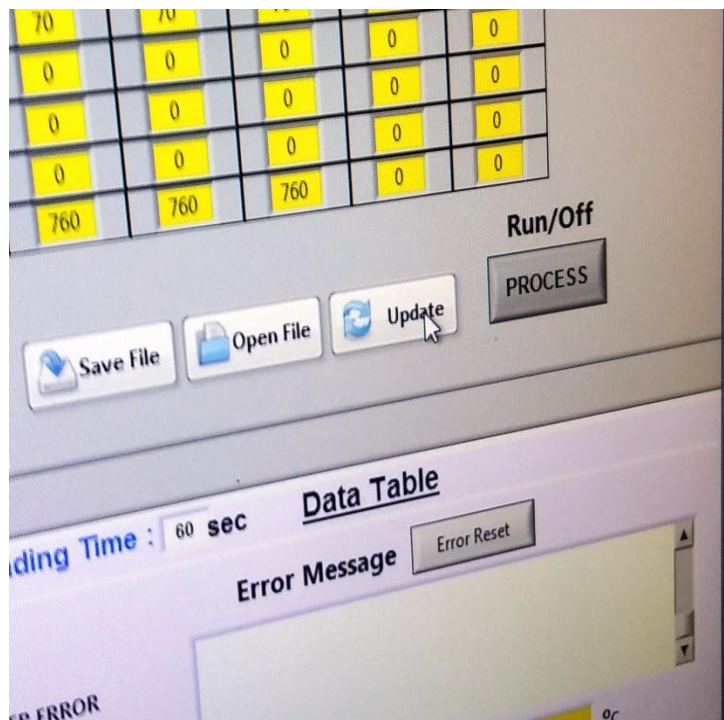


Image 20

o. In the “Recipe name” section, you will see the name of your selected recipe. Then Click on “Update” (see arrow in image 19).

p. Click on “Heater Control”. In “SYSTEM_2 HEATER DOWN STREAM” section (see image 20), you will see an updated value of temperature and time for your selected recipe.

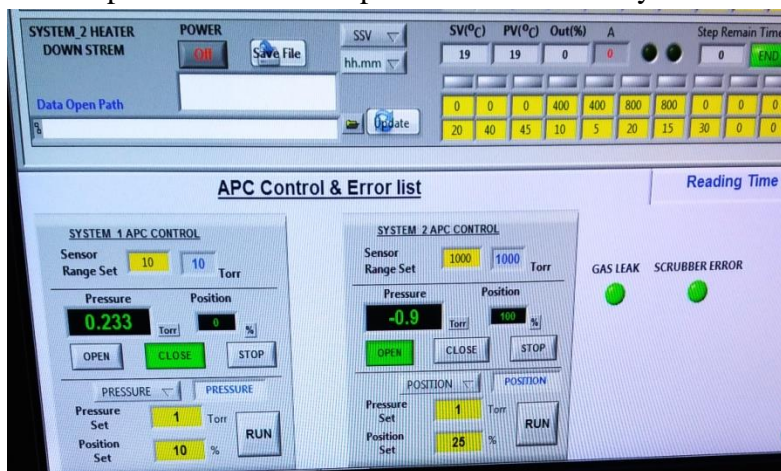


Image 21

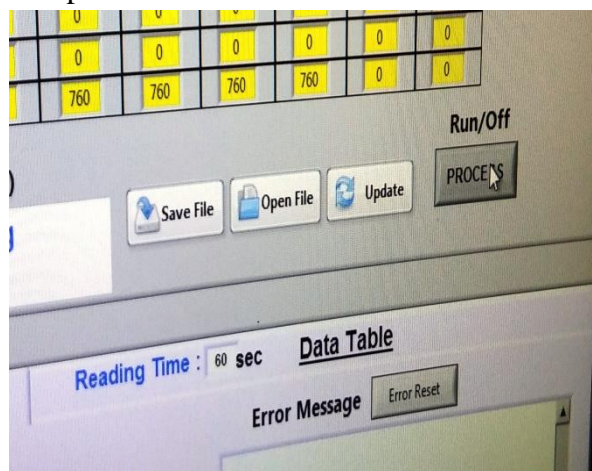


Image 22

q. Go again to “Recipe Control”. Click on “PROCESS” (see arrow in image 21). A window will pop up asking “Do you want to start the auto process with recipe” (see image 22). Click “OK”. Your growth will start automatically (see green signal on process icon in image 23).

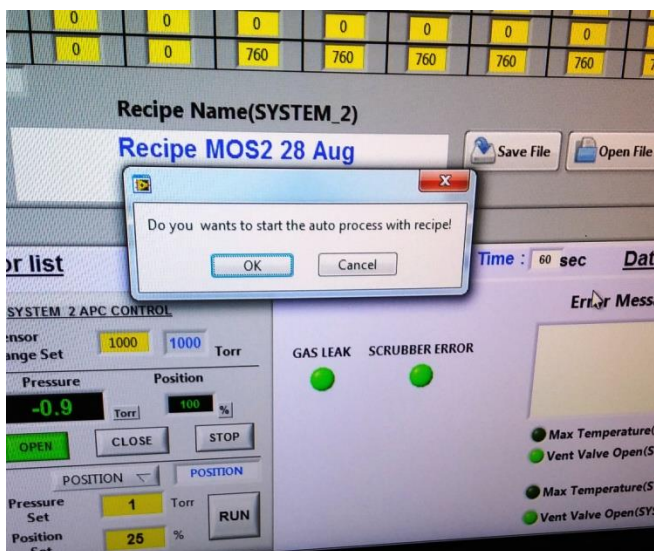


Image 23



Image 24

r. Observe the temperature profile and timing of each step on “SYSTEM_2 HEATER DOWN STREAM” section of “Heater Control” (see image 24).

s. When running recipe will reach to 7th step (see profile on “SYSTEM_2 HEATER DOWN STREAM”) e. g. 800 C, wait to complete this step (see red circle to know 7th step in image 25). After completion of this step, the temperature will start going down (observe the reading at red circle position in image 25). Keep observing continuously. The exact step might not be 7 – but will be the last before the temperature goes lower.

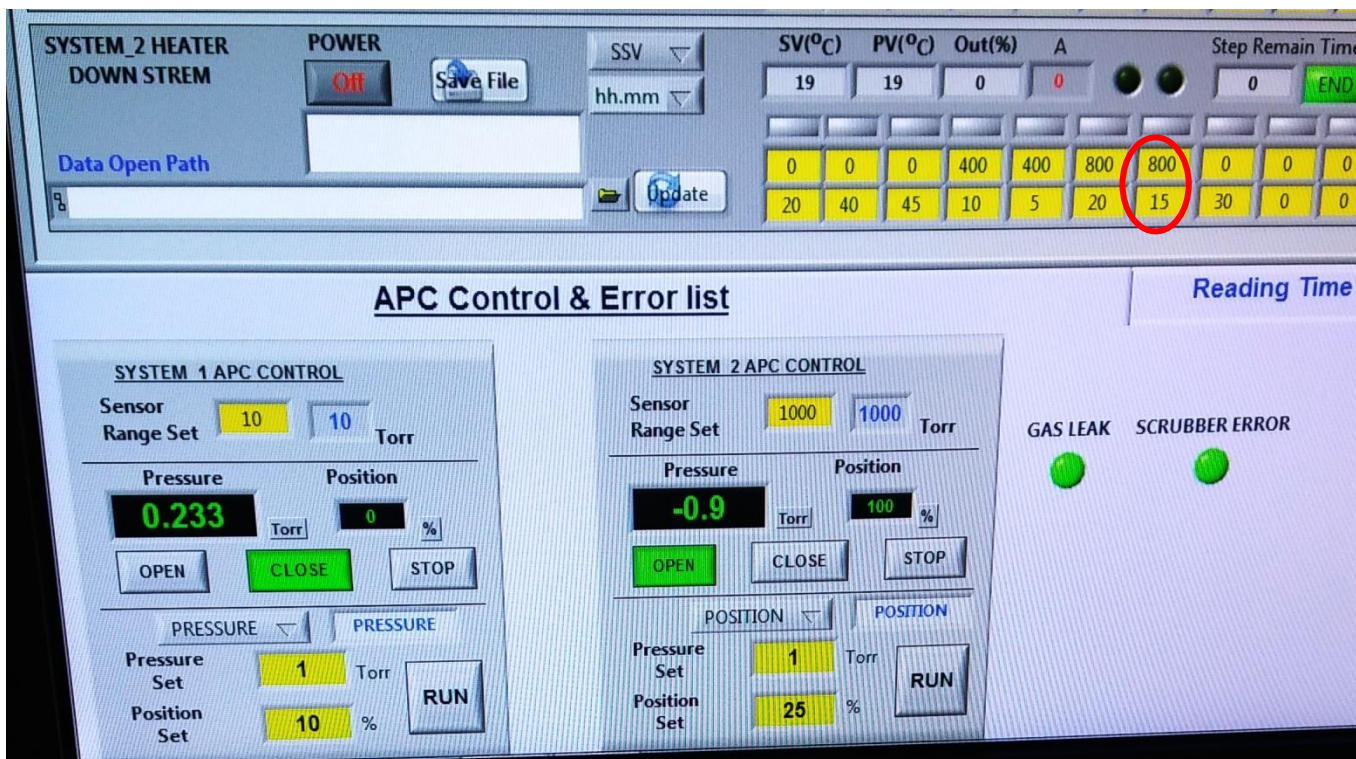


Image 25

t. When temperature reached at 650 °C, **move the furnace** by clicking on “LEFT” in “SYSTEM_2 CONTROL” section (see arrow in image 27).

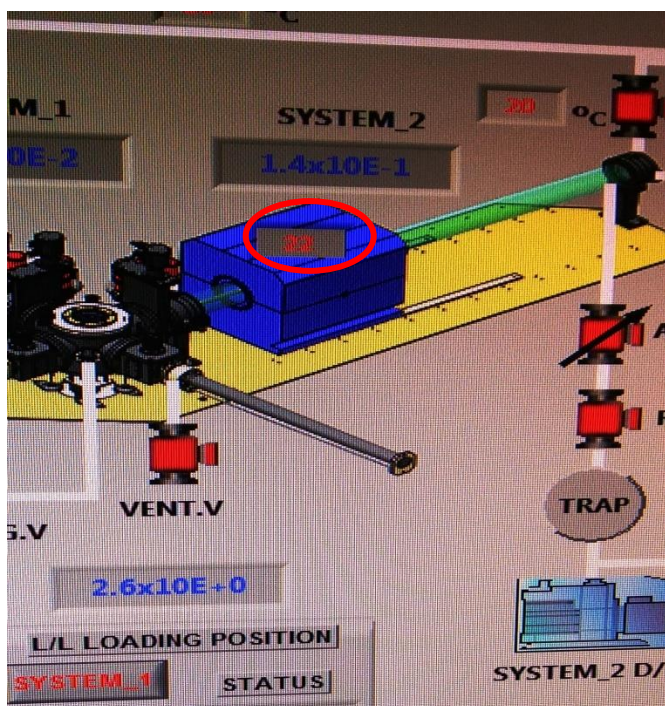


Image 26

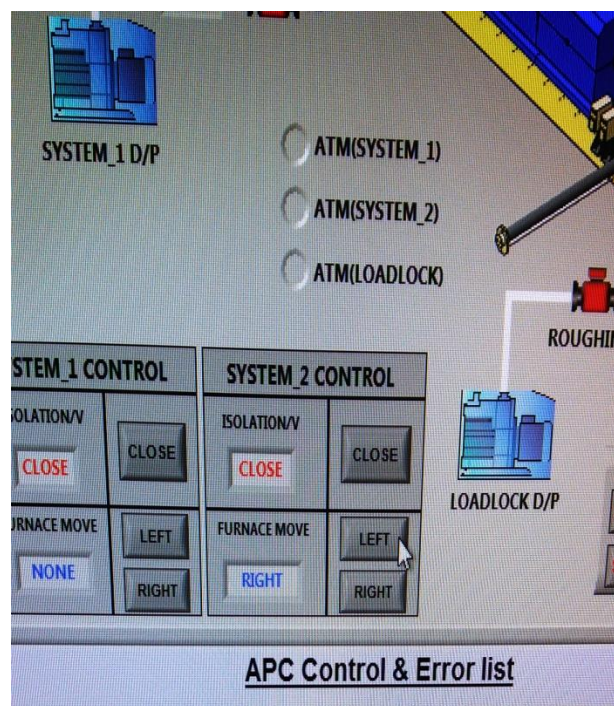


Image 27

u. When recipe will complete. A window will pop up saying “Manually open APC” (see image 28). Click “OK”. Then open throttle valve by clicking on open (see arrow in image 14).

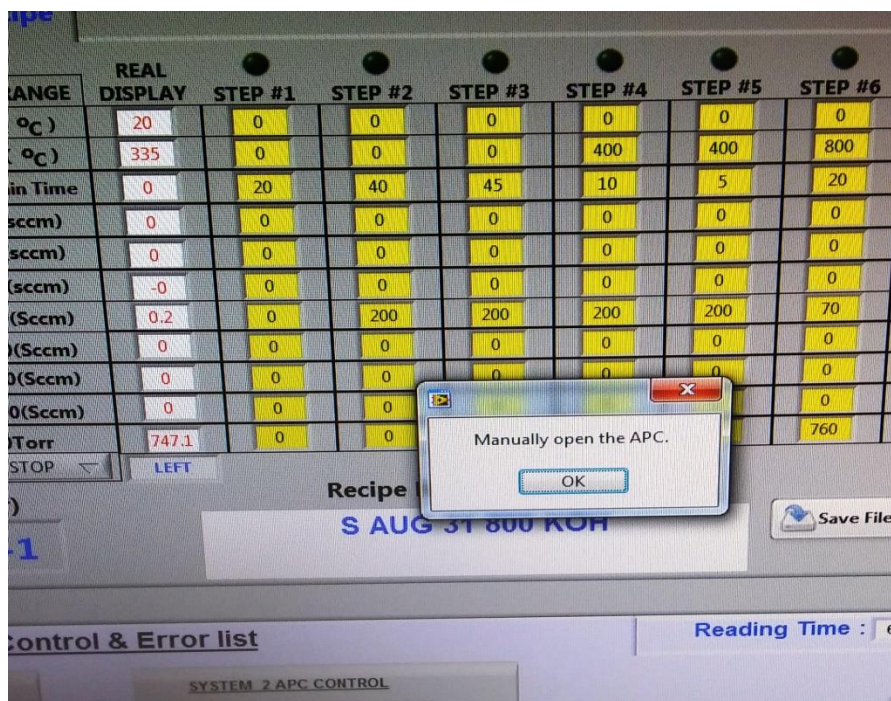


Image 28

v. When the temperature reached at 200 °C, close the throttle valve on the system 2 APC control window (see image 14 for help). Then close the roughing valve (see image 11 for help). After closing the roughing valve, turn off the pump. The best is to turn on Ar gas line until the tube pressure is at 500 torr, then turn it off. Then click on “VENT.V” to vent the tube. After venting, close the vent valve by clicking on “VENT.V” again. Open the tube door to collect your sample.

x. Keep the ceramic boat in the tube, they can be kept close to the door edge. Close the lid of tube. Repeat the steps J to L.

y. When system reaches a vacuum of $\sim 2 \times 10^{-2}$ torr, click on “CLOSE” on the system 2 APC control window (see image 14 for help).

z 1. Close the roughing valve by clicking on “Roughing.V”. Then close the vacuum pump by clicking on “SYSTEM_2 D/P”. After this close the software.

z 2. Log out of Badger.

If the System is not running the control software :

b. On computer desktop there is an icon called ‘T-CVD (UMN)’ (see image 1). Double click on it.



Image 1



Image 2



Image 3

c. A window will pop up prompting you to enter the appropriate user name and password. The user name is “PLANARTECH” and the password is “0000”. Press the **Login** key to finish logging in to the system. A window will pop up highlighting “**You can use control screen**” (see image 3). Press OK.

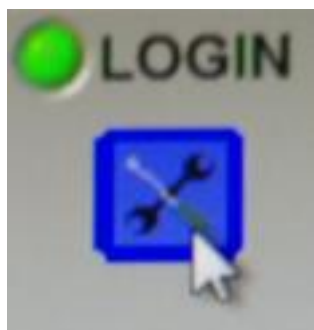


Image 4

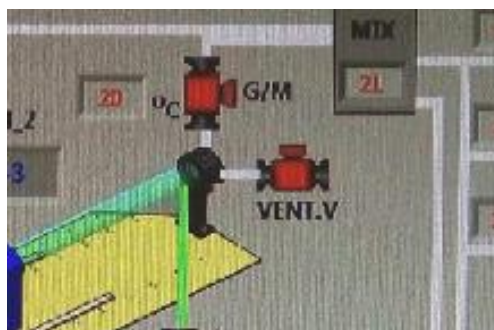
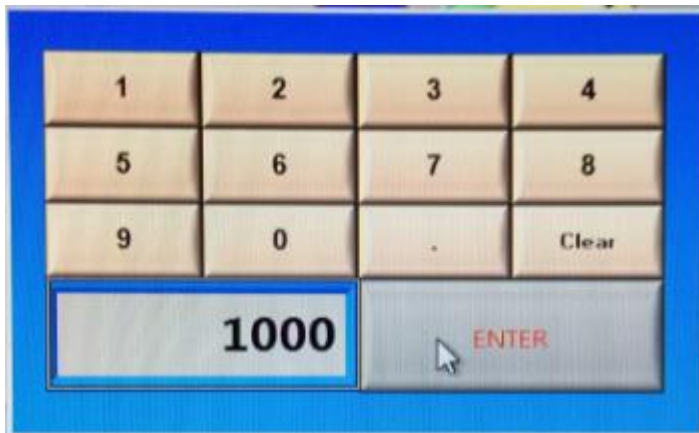


Image 5

d. On login screen, Click on blue icon as shown in image 4. Now you see a screen having the software control details of the system.



see the Sensor range is at 10 - they need to be set to 1000



Enter the correct value for each – then press enter.

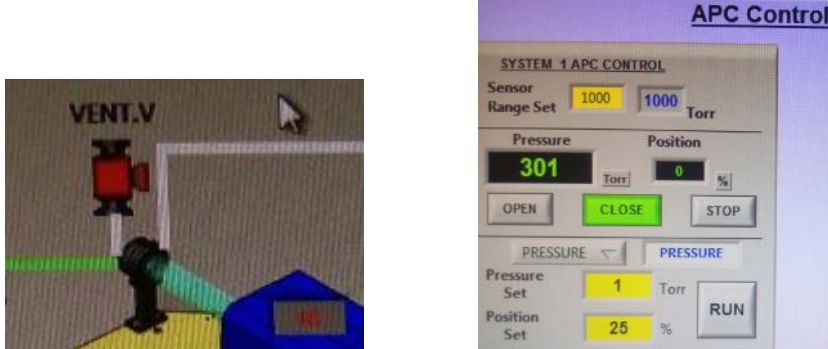


Corrected values for Sensor range

TMD Tube 1 (LEFT Side) Process info

Tony Whipple 26 June 2018

Vent the left furnace Vent.V Using the left side image.



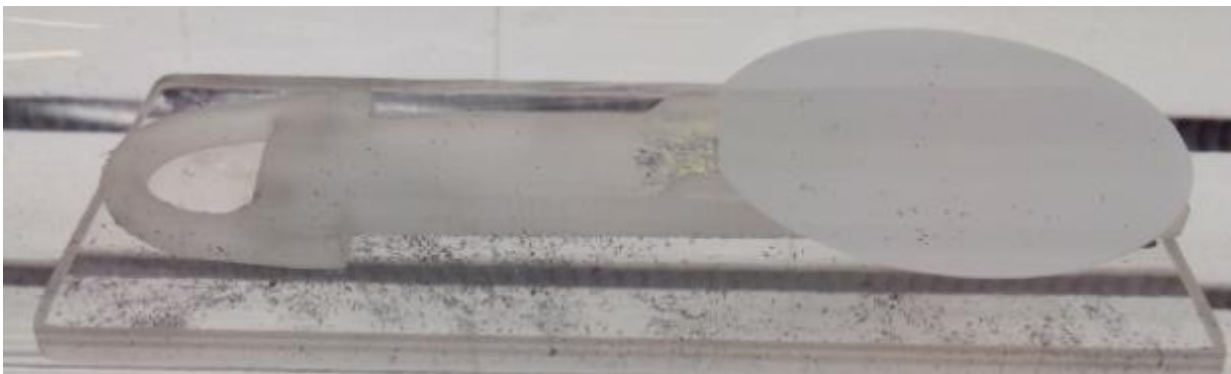
Select the VENT.V for the left of furnace_1 follow the normal steps in SOP but for the left (furnace_1)
For pumping down make sure to select the SYSTEM_1 APC



Placement source material and the wafer surface. Notice this has two furnaces – they DO NOT move.

As with the other side notice this also has 2 chemicals and the wafer is placed above one of them.

The source material holding / boats are stored in the plastic box labeled TUBE 1 So keep them away from the Tube 2 supplies. (I will be ordering more boats to have spares soon.)



The right furnace side has the wafer above the source material of Tungsten Oxide -> 5mg.



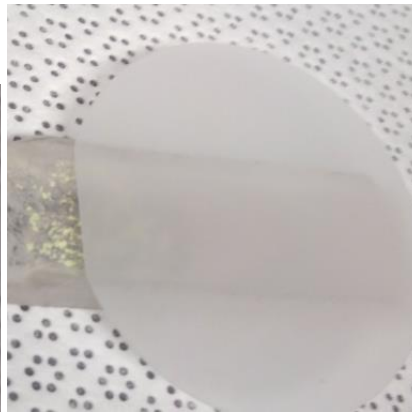
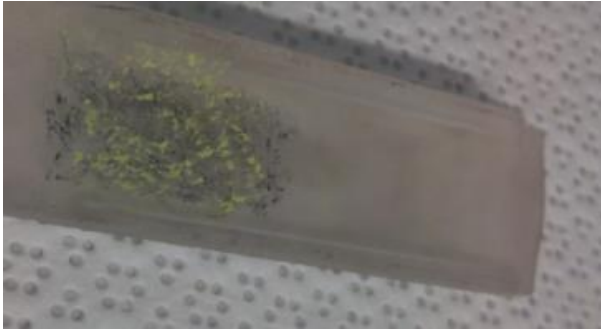
Left furnace side has the normal looking boat – only use the one from the Tube 1 box only!



Correct holder for right side source and wafer holder to be used.



Notice this one is wider – DO NOT use this one for now.



Right furnace uses WO_2 with a total amount of 5 mg Spread it around the flat area some then place your wafer shiny side face down.



The left furnace uses a boat of 1gr of Se and spread it uniformity across bottom.



Put the sample and thin boat on to a small flat part of glass that should be in the box for Tube 1.



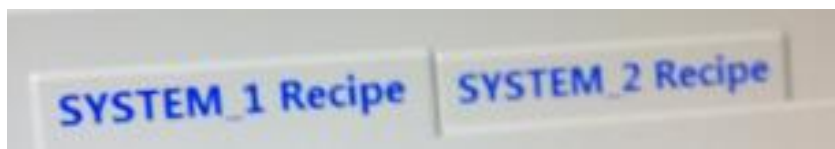
Put the sample and glass and then place this into the tube to the center of the tube right furnace.



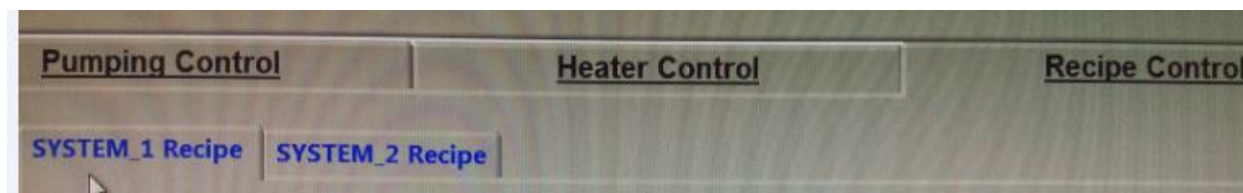
Left furnace end place the source of Se in to the center of the Left furnace.

Close the tube as normal. Pump down the tube 1 using the left side controls. System_1_APC

Turn off Vent.V and then turn on System_1 D/P and then Roughing.V Then open as normal with things at 25%. The slower the better as you do not want any powder to blow around inside the tube. Then open once pumped down.



In the recipe control tab select the System_1_Recipe box.

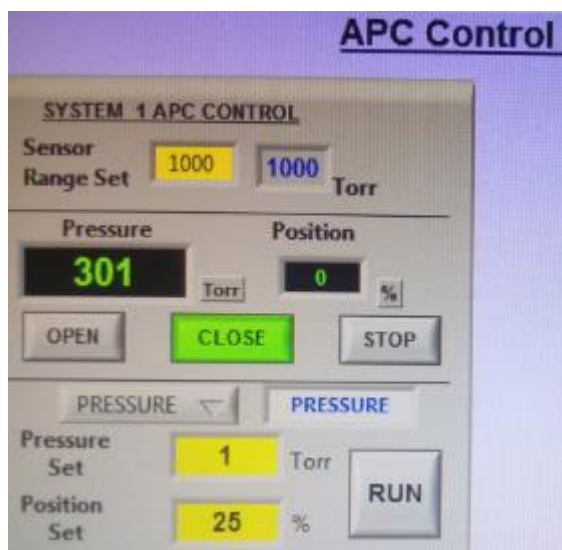


In the recipe control tab select the System_1_Recipe box, then select the recipe from there.

Go to the directory of UMN / Temp Data/ System_1_UP and select the recipe

Recipe to start with is: **WSe2 850C SE 500** Other recipes with other parameters can be selected.

Update and start the recipe as normal – at step 3 start adding LN2 – this is now for the furnace 1 cooling trap. Watch each step during the run.



When the screen notice appears " PROCESS COMPLETE " follow the normal steps – but DO NOT open the tube until the temperature is below 100C. Vent the tube by shutting off the pumps and allow Ar into the tube up to 500 torr. Put the boats back in tube and pump as normal.

Remember that this tube has Selenium power in it. Any waste from that tube should go into the special yellow bag for that type of waste. If the bag is full open another bag and close the full one and enter the date on label. And enter data for the new bag.