

SOP

Standard Operating Procedure: 2D materials CVD

Instruction for operators

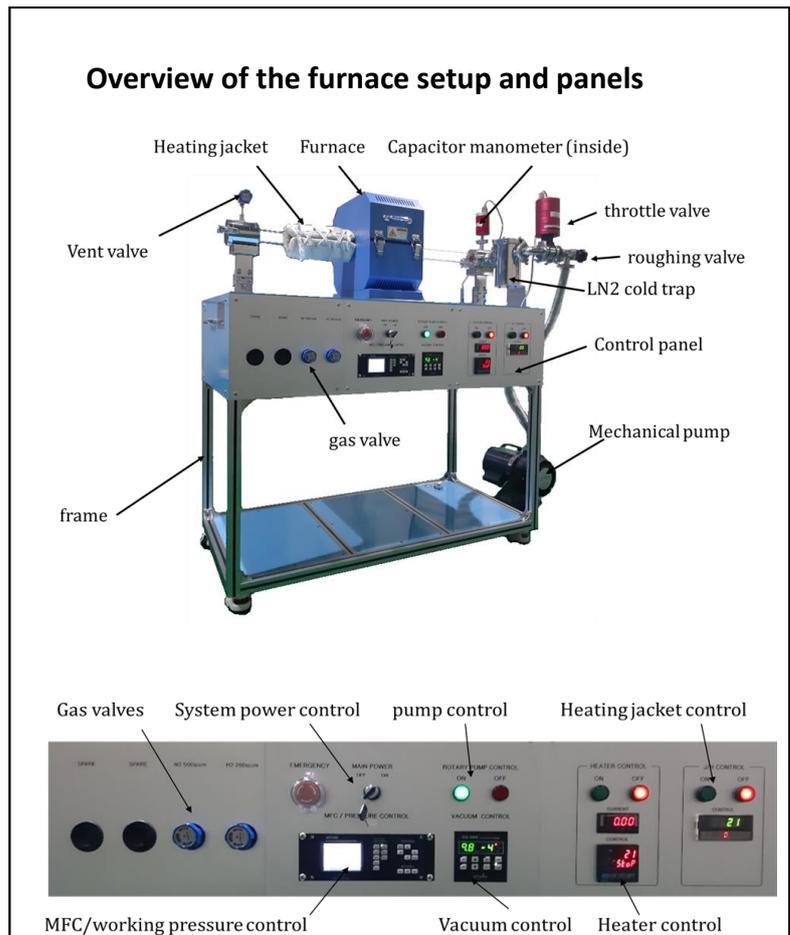
- Before handling anything: put on gloves
- If any of the tanks are empty, replace them before starting.
- Turn on main power and switch the system on and check if all controllers are working fine.

Note-1: The Atovac ACM200-13TBA is a capacitance manometer rated for 1000 Torr. Needed for reading pressure between 1 Torr – 1000 Torr and this gauge is connected to throttle valve and used for displaying pressure on APC200. Another small convection gauge is used to read low pressures and displayed on CG2000 display. To turn on this gauge, press sensor on/off.

Note-2: Vacuum control gauge is less accurate at atmospheric pressure compared to MFC control (at low pressures, it is vice versa)

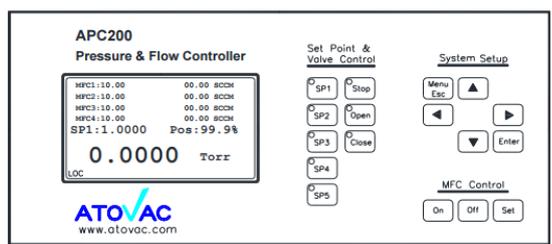
Step-I: Prepare the system and samples

1. Clean the substrates and alumina boats.
2. Weigh the precursors and transfer them to alumina boats.
3. Set the furnace program before starting.
4. Place substrates up-side down on the alumina boats.
5. Place the substrates + boat assembly at the loading end and push using a metal rod carefully to the desired location inside the quartz tube (furnace center). Avoid scratching the quartz tube with the metal rod.
6. Push the sulphur boat to the center of the heating jacket.



Step-II: Vent the system and load the substrates and precursors into the system

1. Ensure the throttle and pump valve are closed. Vent the chamber to atmospheric pressure by flowing 500 sccm Ar or N2 gas using MFC-2 as shown in Figure-3. Press Set-> change flow to desired value-> Press "on"-> Press "set".



To set MFC flow rate

Select MFC1 using

Set the flow rate using button and then

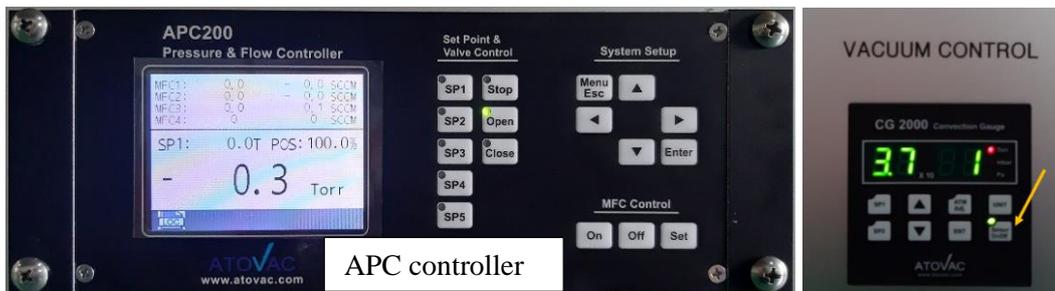
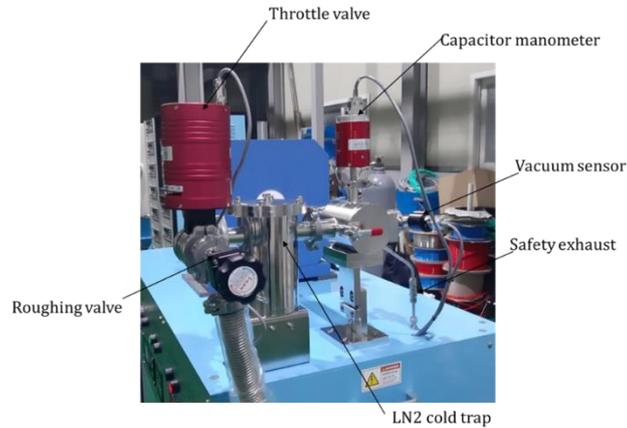
To turn on/off MFC

Select MFC1 using and then and then <= If gas valve is opened, the gas will be flown

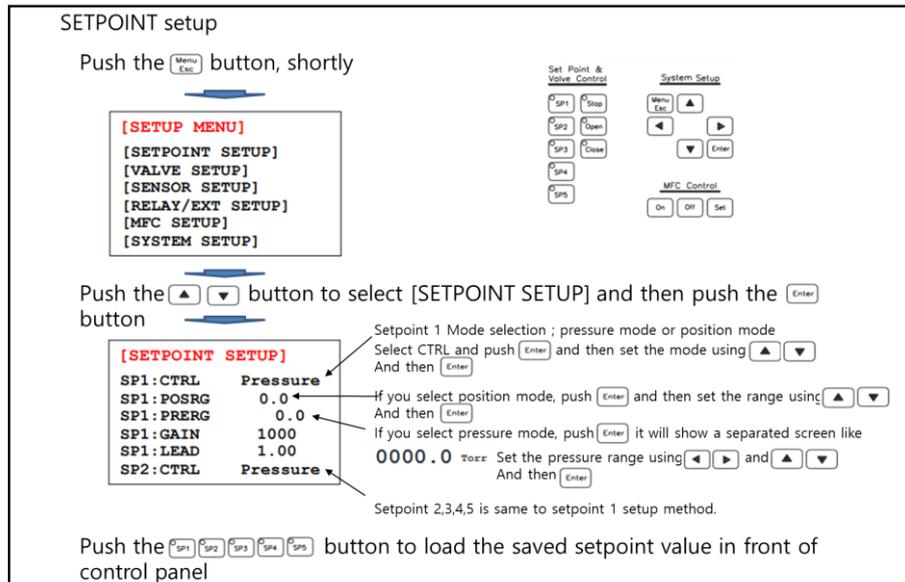
Select MFC1 using and then and then

Step-III: Pump-purge cycles; in order to achieve a good base pressure and remove oxygen

2. For pumping down, ensure the throttle valve and pump/manual valve are closed. Start the rotary pump and warm up for 1-2 minutes.
3. Open the throttle valve fully using the APC controller followed by slowly opening the manual valve. Watch the pressure decrease.



4. Switch on sensor (convection gauge) for monitoring pressure below 10 Torr.
5. Note down the base pressure.
6. Bring the system to desired process pressure.
 - a. Close the throttle valve
 - b. Set the carrier gas flow rate (N_2 500 sccm, MFC-2) to 500 sccm. Press Set-> change flow to desired value-> Press "on"-> Press "set"
 - c. Switch off sensor (convection gauge) during high pressure conditions.
7. Use SP1 set point of throttle valve to set to maintain desired process pressure



Step-IV: Setting up the furnace

1. Long press on heater program-2 for heating the furnace and starting the program set earlier.
2. Note: To change the flow rate during the run, increase or decrease in small steps to avoid pressure fluctuations.
3. Set the J/H heater to desired temperature in two subsequent steps for vaporizing sulfur to avoid overshoot.
4. After the deposition time, heater control shows **Pend** (process end) when the process is over. Switch off heater control and J/H control.
5. Let N₂ flow be low (10 – 30 sccm) until the temperature drops below 500°C.
6. Once it reaches 500°C, increase the flow to 300 sccm.
7. Wait for the temperature to decrease <180°C open the furnace very carefully for faster cooling.
8. After the furnace reaches room temperature, switch off N₂ flow. Press set-> off -> set
9. Close throttle valve and manual valve and switch off rotary pump.
10. Unload the samples
11. Keep the system pumped down to base pressure. [If forming or hydrogen gas line is used during the growth, open line pressure valve of forming/hydrogen gas, open manual MFC-1 which is dedicated for forming/hydrogen and then pump down to ensure no forming/hydrogen gas is left in the line.]
 - a. For pumping down, ensure the throttle valve and pump/manual valve are closed. Start the rotary pump.
 - b. Open the throttle valve fully using the APC controller followed by slowly opening the manual valve. Watch the pressure decrease to base pressure.
 - c. Close the throttle valve and manual valve and turn off pump.
12. Turn off main power and switch the system off.