Usage Policies Notebook for NanoFurnace Furnace (EasyTube 3000 System)

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INRF UCIrvine

Emergency Plan for Nano Furnace

Standard Operating Procedures for Emergencies

Contact information

Person	Phone number
Lab Manager	Jake Hes, 949-824-8239 (day), 562-522-8328 (alternate)
Director	G.P. Li: 949-824-4194 (day), 949-824-2047 (alternate)
Staff	Mo Kebaili: 949-824-8239 (day), 949-494-5892 (alternate)
Super User	Phi Pham (720) 939-4500

Hazardous chemicals, gases, and conditions

Hazard name	Description of hazard
High voltage	Electrical shock, ignition source
CH_4 (Methane)	Flammable, Non-corrosive
H_2 (Hydrogen)	Flammable, Non-corrosive
N_2 (nitrogen) gas	Asphyxiant Non-flammable,
Ar (Argon)	Non-flammable, non-toxic

Alarms or indications of danger

Alarm type	Condition and response
Gas Detector (Enmet) H_2 and CH_4	Problem with process state. Halt process and correct problem or notify the staff or the lab manager before continuing.
O_2 flow Alarm	Problem with process state. Halt process and correct problem or notify the staff or the lab manager before continuing.
Pungent or foul smell	Gas leak. Shutdown the tool at once and evacuate the area. Contact the staff and the lab manager.

Emergency shutdown plan #1

In the event of an emergency, when there is very little time, press the large red emergency shut-off button at the entrance of the room I, this will shutdown the gas system, and will stop the gas flow. Leave the facility at once, and then contact the lab manager or the staff.

Emergency shutdown plan #2

In the event of an emergency, when there are a few minutes available, turn off the gas switches on the gas panel, the control power and the main power. This will stop gas flow into the system. If there is no fire, and no smell of gases, enter the room and close off all gas cylinders by turning them fully clockwise. Check the oxygen tank in room w2349, feel the door for possible fire, and if safe, close the oxygen tank by turning fully clockwise. Leave the facility at once, then, contact the staff and the lab manager.

IMPORTANT INFORMATION

Hydrogen and methane combustible gases are used at atmospheric pressure and low pressure in the 5" EasyTube3000 Furnace to grow carbon nanotubes, graphene, and annealing. To avoid any potential fire or explosion, it is important to understand the lower and upper explosive limits of these combustible gases.

A combustible gas will ignite only if the three following conditions are met:

- There is enough combustible gas to start a fire
- There is enough oxygen to oxidize the combustible gas
- There is a source of energy to ignite the combustible gas

If all of the above three conditions are not present at the same time, then the combustible gas will not ignite and burn. To have enough combustible gas to start a fire means that the concentration of the combustible gas present has to be between its lower explosive limit (LEL), or lower flammable limit (LFL) and upper explosive limit (UEL), or upper flammable limit (UFL).

For hydrogen:

- 100% LEL is equal to 4% hydrogen by volume in air
- The UEL is equal to 75% hydrogen by volume in air

For methane:

- 100% LEL is equal to 5% hydrogen by volume in air
- The UEL is equal to 16% hydrogen by volume in air

Hydrogen and methane will auto ignite in the presence of oxygen at a temperature around 500°C When the 5" EasyTube3000 is used to grow carbon nanotubes, and graphene, we already meet two of the three conditions to have a fire or an explosion: Inside the quartz tube:

- We have enough combustible gas (hydrogen and methane)
- We have the energy source (900°C furnace temperature)
- The missing condition is the presence of oxygen

Outside the quartz tube:

- We have enough oxygen
- We have the energy source (900°C furnace temperature)
- The missing condition is the presence of combustible gas

To prevent a fire or an explosion, we need to keep the oxygen from getting inside the quartz tube, and we need to keep the combustible gases from getting outside the quart tube.

Usage Policies for Nano Furnace

Standard policies for usage

The 5” nano furnace can accept up to 5” substrate to grow carbon nanotubes. Mass flow controllers digitally control H2, CH4, N2, and Ar gas flows. The programmable temperature range is up to 1000°C.

Contact information

The INRF staff or the lab manager can be reached at (949) 824-8239 or (949) 824-9831.

Authorized users

Only INRF registered users who have completed the training and passed the certification can use this equipment. Users can only use the portion of the system for which they have been trained.

Training

Users must have received direct training from the staff in order to use this equipment. Users are expected to understand the nature of the system, as well as the proper control and use of the gases. Training varies slightly, depending on the process to be performed. Contact the staff for details and to arrange for a training session.

Usage log sheets

Users are required to log all activities in the log sheets provided. All users must log in when they used the furnace (date and time), which gases they used, and when they completed their process in the user log sheets. If users notice anything unusual, they should record it in the user log sheets, and add details in the main comments area on the log sheet. Any maintenance to the tool will be logged in the maintenance log sheet (maintenance staff only).

Safety equipment

As safety equipment for use on this equipment, cleanroom gloves and tweezers should be used when handling pieces in the tube. Care should be taken to avoid burns when working near the furnace tube. The users need to use the gas leak detector to make sure that no gas is leaking from the furnace tube end cap.

Standard equipment and materials

The laboratory provides the following gases: N_2, H_2, CH_4, and Ar. Other gases must be cleared with the lab manager.

User maintenance *

To keep the furnace contaminant free, keep the end cap on the tube when it is not in use to keep a positive pressure within the furnace tube under N_2 flow.

Waste disposal

Dispose of alcohol soaked wipes in a waste container marked for flammable solid waste.

Scheduling

Reservation can be done online, and the system can be used on a first come, first served usage if no reservation was made.

Other issues

Users should remain physically present in the cleanroom facility during the entire use of the nano furnace.

At no time should a user adjust a pressure regulator on a gas line. Gas control should be “on” or “off” only, using only the appropriate valves. For most gases, this is usually the valve at the cylinder head.

Non-standard use

Users may not modify any hardware on this equipment. For use of non-standard processes, gases or materials, contact the staff or the lab manager.

Usage Notes for EasyTube3000

Guide for using the EasyTube3000

All gas cylinders should be turned on or off at the cylinder valves. At no time should a user adjust a pressure regulator. Clockwise for all valves, means CLOSED. The standard off configuration for the system is to close the cylinders valves, but leave all other valves alone.

System Setup

The 6” nano furnace can accept up to 4” substrate to grow carbon nanotubes, graphene, and perform vacuum anneals. Mass flow controllers digitally control H2, CH4, N2, and Ar gas flows. The programmable temperature range is up to 1000°C.

When the 6” nano furnace is in the idle mode, 1 L/min of nitrogen gas flows in the quartz tube. This nitrogen flow maintains a positive pressure in the quartz tube to keep it clean from particulates contamination.

There are six mass flow controllers dedicated for H2, CH4, N2, and Ar, C2H2 used by the 6” nano furnace. Each mass flow controller can be digitally set to maintain a preset gas flow.

Safety Check

The 6” nano furnace is monitored by a set of combustible gas leak detectors. There is a combustible detector installed in the back of the furnace where the mass flow controllers are located. There is a second combustible gas detector installed above the wafer loading area of the furnace front.

The users should verify that the exhaust scavenger has a negative flow.

The users should check for any potential leak of combustible gases with the handheld leak detector, at the front-loading area of the 6” nano furnace.

In case of a potential combustible gas leak, the users should abort their process, they should stop flowing combustible gases inside the 6” nano furnace, by setting the hydrogen and methane mass flow controllers to zero. The users should immediately contact the INRF staff or the lab manager.

Usage Notes for Nano Furnace

Guide for using the Nano Furnace

Gas Cylinders

All gas cylinders should be turned on or off at the cylinder valves. At no time should a user adjust a pressure regulator. Clockwise for all valves, means CLOSED. The standard off configuration for the system is to close the cylinders valves, but leave all other valves alone.

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The Easy Tube 3000 Nano Furnace can be used to do carbon nanotube growth, graphene growth, and annealing at both ambient and low pressures. It is important to know the dangers of each recipe you are running including gas flow and pressure gauges.

Generally, when you walk in, the furnace will be in standby mode which means the loading chamber and the growth chamber are at ambient pressure, and under N2 gas purge. Verify that the sample loader is in the “unloaded” position, and the pressure inside both chambers are ~760 Torr.

1. Turn on the computer monitor, and open the WinCVD program and select your recipe. You will need to turn on the burn box to begin (recipes will not run unless burn box is over 700 C). Set “Burn Box Temp” to 800, and “Burn Box Power” to 90%. Let this warm up before you run any recipe.

2. Open the door to the loading chamber to load your samples. Gently remove the quartz boat, and load your samples on the boat. Be careful to gently place the quartz boat back onto the loading arm. Lock the loading chamber door.
3. Using the key, open the bottom panel (under the loading chamber), and make sure to pull out the white alarm knob. Turn on the vacuum and verify that the pressure gauge reads ~ 23.

4. When your sample is ready to be loaded, press the “load sample” button on the CVD panel. At this point, make sure to hold the chamber door closed, and verify that the system is pumping down on the loading chamber. After the chamber reaches 5 x 10-2 Torr, the system will purge back to atmosphere with N2. Then, the sample should automatically be loaded into the growth chamber.

5. After the sample is fully loaded, turn off the vacuum pump, and close and lock the panel door.

6. Go into service chase and verify that all necessary gas cylinders are open, and that the chiller is running. (Call Phi before you run the furnace so he can turn on the gases and adjust any of the pressure regulators. If any of the gases still need to be turned on, contact Phi).

7. After the burn box reaches a temperature above 700 degrees, you can begin the recipe. To start the recipe, in the CVD program click “Reset!”. This should start the recipe and an alarm will sound. “Silence” the alarm, and “reset” the alarm.

8. The system will begin to pump down the growth chamber in order to check for leaks. Verify that the system pumps down to 500 mTorr after it cycles through the pump-down leak-back sequence. If the system passes this criterion, the recipe will start.

9. While the recipe is running monitor and verify pressures, flow rates, and temperature.

10. After the recipe is fully complete (furnace cooled down to 200 C, and pressure back to ambient), unload sample by pressing “Unload Sample” button.

11. When sample holder is fully moved back to the “loading chamber”, open chamber door and remove samples (again remember to use care when moving quartz boat).

12. Close chamber door.

13. Go into manual mode and set “Burn Box Temp” and “Burn Box Power” back to “0”. After burn box drops below 700 C, silence alarm.

14. Contact Phi to close all gas valves.

15. Verify that both chamber pressures are around ambient pressures, flow rates for reaction gases are 0, and temperatures are near room temperature.

16. Close program and shut off monitor.