PROTEMP DIFFUSION FURNACE

STANDARD OPERATING PROCEDURES (SOPs) 2020 (v.1)

NCPRE FABRICATION LAB

SO - Sandeep Kumbhar Co SO-Sreejith K. P.

CONTACT: sandeeo.kumbhar@gmail.com, sreejithanni@ee.iitb.ac.in

9969347860 9930837262

TABLE OF CONTENTS

INTRODUCTION

The PROTEMP DIFFUSION FURNACE is an Atmospheric furnace, which can accept four-inch, five inch and six-inch large substrates. Each of the three furnaces tube is setup for a specific process, Phosphorous Diffusion and Thermal Oxidation. The furnace tubes are horizontally placed, and the gas flows are controlled by mass flow controllers (MFCs). The maximum temperature range is up to 1200°C

Equipment Configuration

1. Capability to process 4'', 5" and 6" sized wafers.
2. Process tubes available:
   TOP Tube: N Type Deposition ($POCl_3$)
   MIDDLE Tube: Dry Oxidation
   BOTTOM Tube: P Type Deposition (BBr_3) (currently unavailable)
3. **Source Type :** Liquid source
4. **System Temperature Capability:** 1200°C (for all three tubes)
5. **Gas Inlets available**: O_2, N_2 with inline MFCs for each tube.
6. **Temperature controllers**: EUROTHERM.
7. **Software interface**: Timcom Tymplex, Tymedit for programming temperature controllers & recording process recipes:
8. **Bubbler Temperature controller:**
   Fujifilm Temp. Controller (TC2000)
   Mains Voltage supply: 230VAC, 50 or 60 Hz
   Power consumption: 200 Watt Maximum
   Ambient temperature: 40°C
   Temperature accuracy: 0.2°C
9. **Local Supporters**: OM Scientific Pvt. Ltd.
10. **Original Manufacturer**: ProTemp Product, Inc.
    3511 Thomas Rd., Unit 4
    Santa Clara, CA 95054
    Tel: 408-855-8222 x102
    Fax: 408-855-8224
    www.protempproducts.com

Photoresist, kapton tape, metals, glass, or other foreign material are not allowed in any of these furnaces.

SAFETY PRECAUTIONS

- Safety Symbols

			
Ground Terminal	Electric shock hazard	Hot surface	Pinch point caution

- During loading and unloading the wafers, wear goggles and face shield. The loading and unloading temperatures are quite higher of the order of 700- 800 °C.
- Never touch the hot furnace components and the wafers with gloves when they are at high temperatures.
- The system has software safety interlocks, to prevent hazards.
- Avoid touching hot exhaust scavenger.
- Cross check that the exhaust is ON before starting the process.
- Check with the facility team for cylinder and gas line pressures for N_2 and O_2 before starting the process.
- Provide enough time (approx. 20 min) for cooling to room temperature before transferring the samples after the process.

CLEANING METHODS:

- Cleaning of the outer walls of the furnace, computer and table can be done using IPA and lint-free cloth.
- Quartz boats can be cleaned (if required) by keeping it in 2% HF solution for 24 hours followed by rinsing with DI water.

OPERATION THEORY

Basics of Diffusion

The diffusion of dopants into silicon via high-temperature thermal processes is one method in which silicon wafers are doped with extrinsic elements such as boron or phosphorous. During a diffusion process, extrinsic elements are introduced, commonly in a gaseous or liquid phase, at high temperature and come into contact with the silicon wafer. A commercial diffusion process may consist of one or two steps including, a deposition step in which the dopant source is supplied into the furnace and a drive-in step, in which the source is cut-off and no further dopants are introduced into the furnace.

The deposition process, if performed in a two-step process, is typically at a lower temperature than the drive-in process. During this step, the target material is coated in dopant source to supply the surface with a uniform diffusion source. The point of the drive-in step is to provide the thermal energy for the dopants to diffuse into the material. A two-step process is potentially more time and resource consuming than a single step process. However, the separation of the drive-in and deposition processes allows for greater control of the resulting diffused atom profile and hence the overall performance of the device.

The depth and the profile of the dopants can be estimated using the Fick's law. Solid solubility determines the dopant concentration at the surface by pre-deposition process.

Atomic diffusion in solid state materials such as silicon is driven by Fick's law. Atoms diffuse from the high concentration regions to low concentration regions. The concentration of the dopants, *C* depends on the depth *x,* and the time, *t* i.e., *C*(*x*,*t*). The number of atoms that diffuse as a unit area in unit time is determined using Fick's 1st law,

$$F = -D\frac{\partial C}{\partial x}$$

where *F* is the atomic flux, *D* is the diffusivity and is the concentration gradient. The diffusivity is a function of temperature which can be expressed in Arrhenius form,

$$D = D_0 exp^{-\frac{E_a}{kT}}$$

where D_0 is the pre-exponential constant, E_a is the activation energy, *k* is the Boltzmann constant and *T* is the temperature. The diffusion length, λ is the distance at which the concentration of the diffusing atoms drops to a lower value. it can be calculated based on diffusivity and the time:

$$\lambda = \sqrt{4Dt}$$

Fick's 2nd law is used to determine the dopant concentration in a cross-sectional area as a function of time. The change in concentration with time is proportional to the diffusion flux,

$$\frac{\partial C(x,t)}{\partial t} = -\frac{\partial F}{\partial x}$$

Which can be expressed as,

$$\frac{\partial C(t)}{\partial t} = -\frac{\partial}{\partial x}(D\frac{\partial C}{\partial x})$$

If the diffusivity (*D*) is constant, it can also be re-written as:

$$\frac{\partial C(x,t)}{\partial t} = -D\frac{\partial^2 C}{\partial x^2}$$

At steady stated, therefore and $C(x,t) = a + bx$, which is a linear concentration profile over distance that is independent of time. This would be an example of thermal oxidation of silicon.

STANDARD OPERATING PROCEDURE

The system is operated in manual mode. Three Eurotherm temperature controllers of each tube provides a flat temperature profile in the furnace. They are arranged in a master/slave configuration and the temperature profile is set up in manual mode. The gases are introduced through a quartz injector, located at the Source end of the tube. The Gas Control Module allows to set up and monitor the Mass Flow Controller flow for each gas, and also manages the safety hardware interlocks.

Following fig shows Closed tube phosphorus diffusion system diagram.

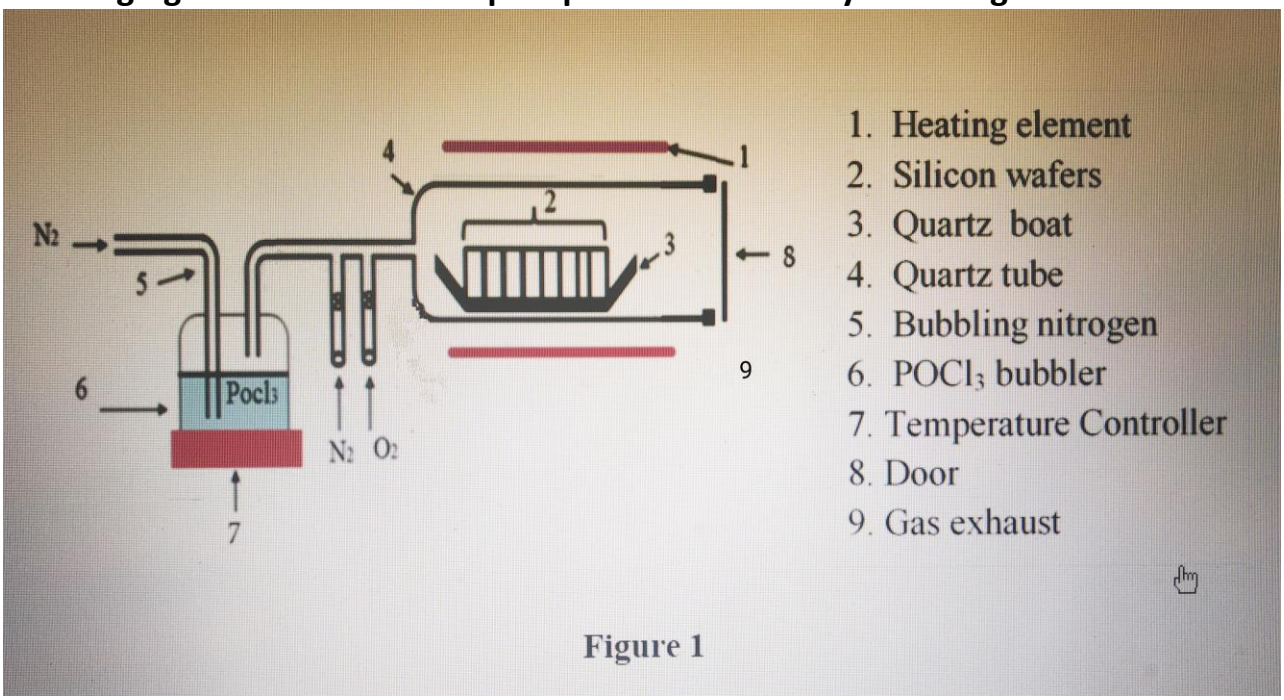


Figure 1

1. Ensure the following before and after entering the clean room (Pre Checks):

a. All system components should be ON and running 24 hours, 7 days a week. (**Note:** If the system is not ON or is not operating in a normal condition, contact the SO and do not attempt to start the system without assistance.)

b. Verify that the diffusion tube is at idle temperature of 450°C (400°C in case of Oxidation) and nitrogen is flowing at 1 slpm to make positive atmosphere in the tube. Clear any fault condition before process operation, if any.

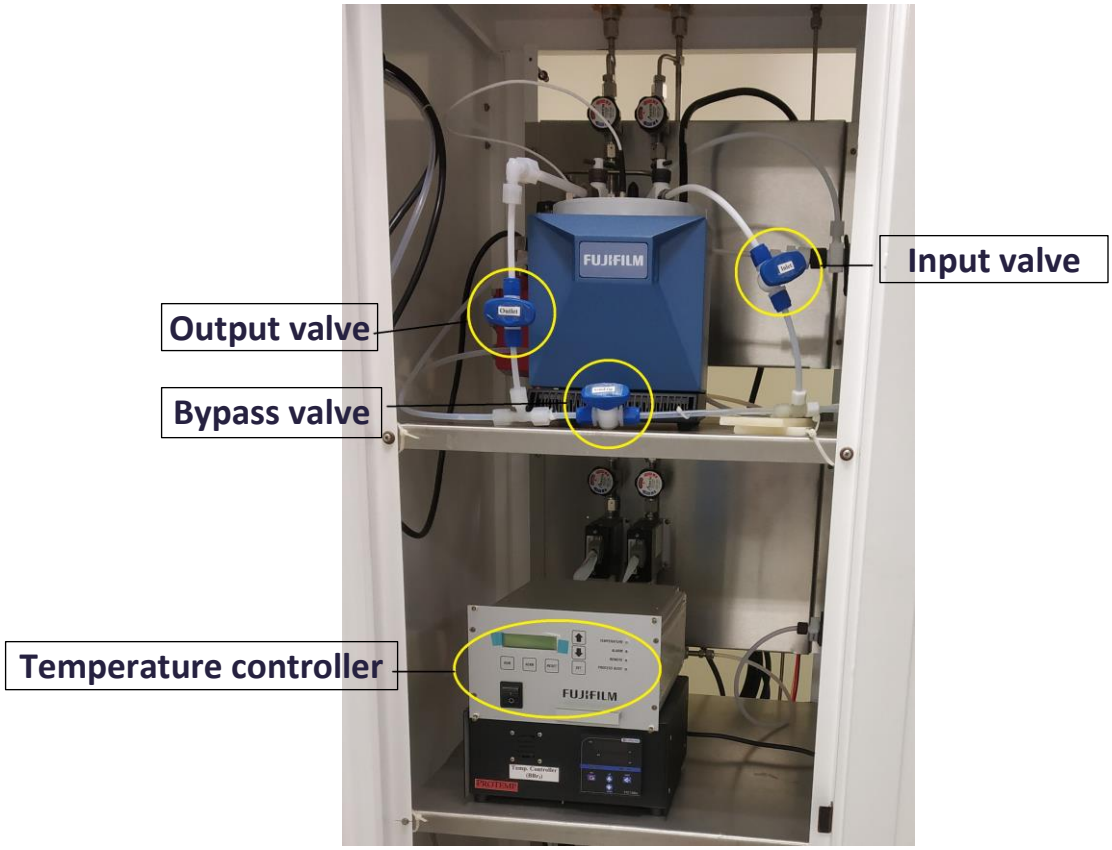
c. Ask the facility member to switch ON the Oxygen (O_2), at least 1 hour, prior to the process.

d. Pressure meter showing the tool exhaust suction (The reading should be between 50 and 25). This ensures there is a sufficient CFM suction provided by the tool exhaust.

e.

2. Bubbler operation:

a. **$POCl_3$** (Phosphorus Oxychloride)
   $POCl_3$ is a toxic liquid that reacts vigorously with water or atmospheric moisture to form corrosive hydrogen gas, phosphoric acid and heat.
b. When the furnace is not in use the bubbler lines are purge (24X 7) with N_2 continuously to avoid clogging, through bypass. If line is clogged, process will not start due to interlock alarm. (Keeping i/p and o/p closed.)
c. Bubbler is kept in the FUJIFILM TC2000 Source temperature controller. It is designed to keep the liquid source chemicals at a constant temperature throughout the process ($POCl_3$: 23.5°C). Turn ON the temperature controller before process to cool the bubbler.



d. Above figure showing the $POCl_3$ bubbler in off condition (with bypass on). Turning the respective "T's" as shown in figure makes it in "on" as required before the process of diffusion.
e. If any gas detector alarm is triggered, it automatically shuts off the Lo-N_2 flow through the bubbler (all detectors are integrated in a single GLD panel).

3. Contamination Control Procedures:

a. Substrates should be RCA clean
b. Do not handle wafer boats with hands. Use pick up tools all the time.
c. New boats should be cleaned before entering to lab.

4. Wafer loading/ unloading:

a. Press 'unload' mechanical switch to UNLOAD the wafer Jig. The cantilever and hence wafer boat will slowly come out, and will stop after it reaches the outer limit switch. DO NOT REMOVE WAFERS WHEN THE BOAT LOADER IS MOVING!!!!

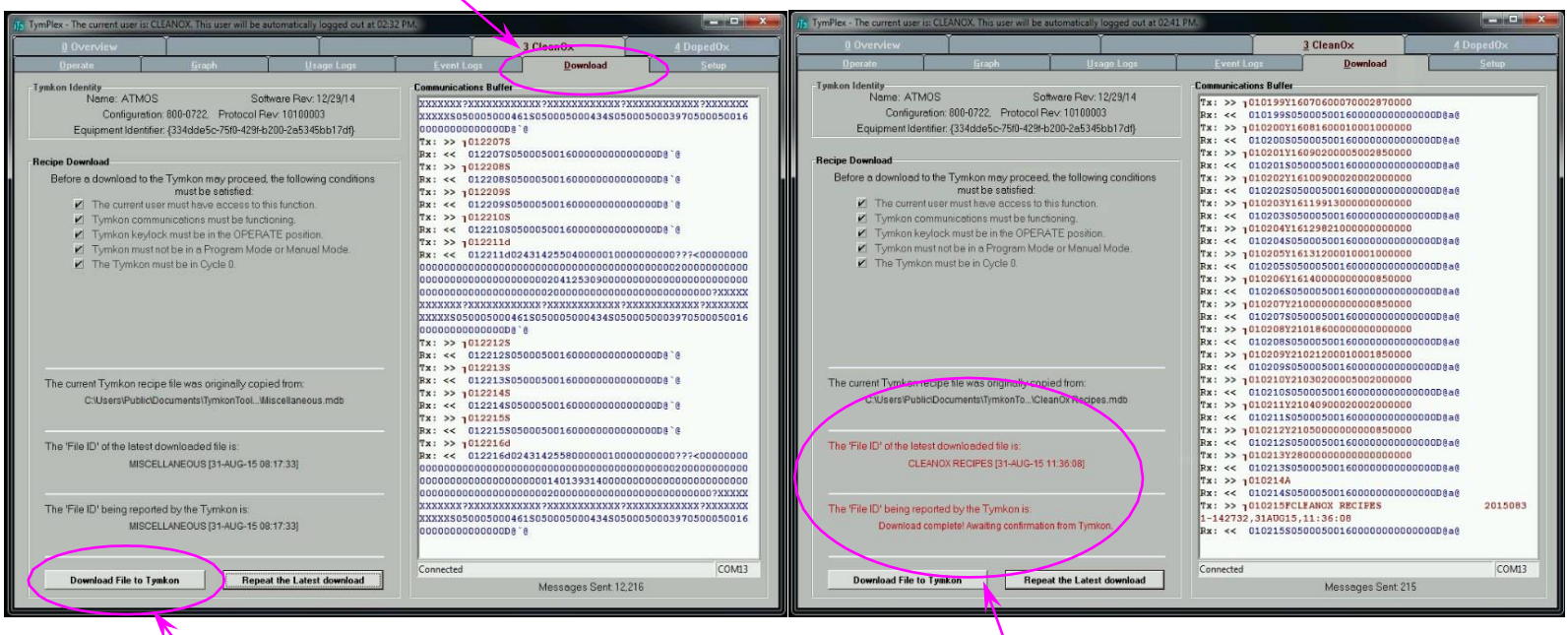
b. Remove the quartz cassette from the cantilever using the pickup tool. (USE CAUTION-WEAR HEAT PROTECTIVE GLOVES and other required PPE!!!!)
c. Carefully lower the cassette to the cleaned table at the load station and let it cool down to Room Temperature.
d. Remove/ make a space in between dummy wafers and place your device wafer(s) in the empty slot. Make sure the wafers are properly aligned.
e. Put the boat back on the cantilever. Make sure the boat is seated at level and centered on the quartz rails.
f. Press the mechanical switch to LOAD to load the boat. The wafer boat will slowly go in, and will stop after it reaches the inner limit switch.

5. Make the recipe:

a. On the computer monitor, click on icon “timedit”.
b. Select “$POCl_3$ folder” (each tube has separate folder for operation)
c. Go to the “Files” and select any saved recipe. It will direct you to “edit process” mode.
d. Selected recipe can be edited now with segments (process steps), % gas flows, time etc.
e. In “Edit Temperature” temperature can be edited.
f. Click on “Apply” to save the changes.
g. Clicking on “Validate” will validate the recipe.
h. Go to “Files” and “Save” the recipe.

6. Download the Recipe :

a. Click on “tymplex”
b. Go to “$POCl_3$” tab and open by clicking on it.
c. Make sure the system should be in “Reset” mode.
d. select the **Download** subtab
e. Click the **Download File to** tymcom button.
f. From the dialog box, locate and select the correct recipe file.
g. From the **Ready to Download** dialog box, click the **Begin Download** button
h. Wait for the recipe to complete downloading. (Sections will change from red to black text.



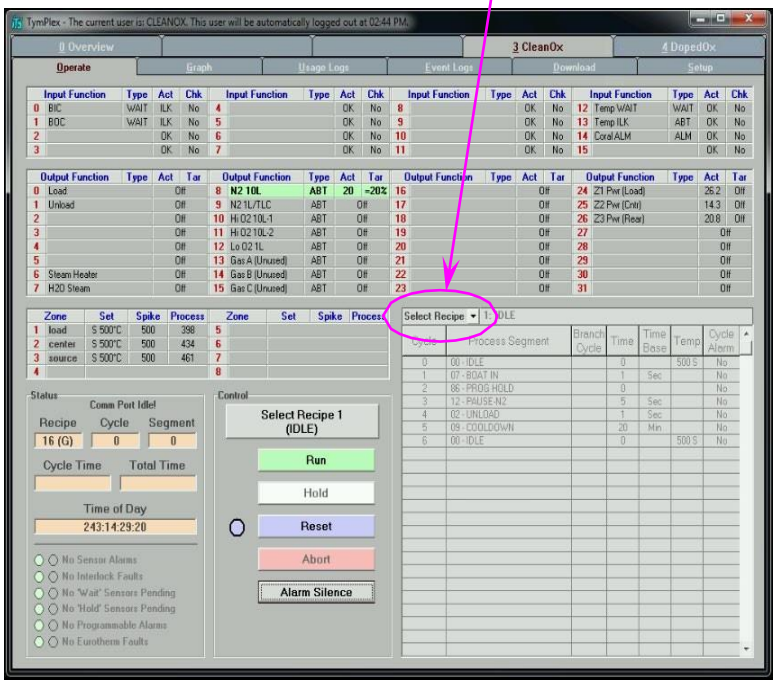
7. Select the Recipe:

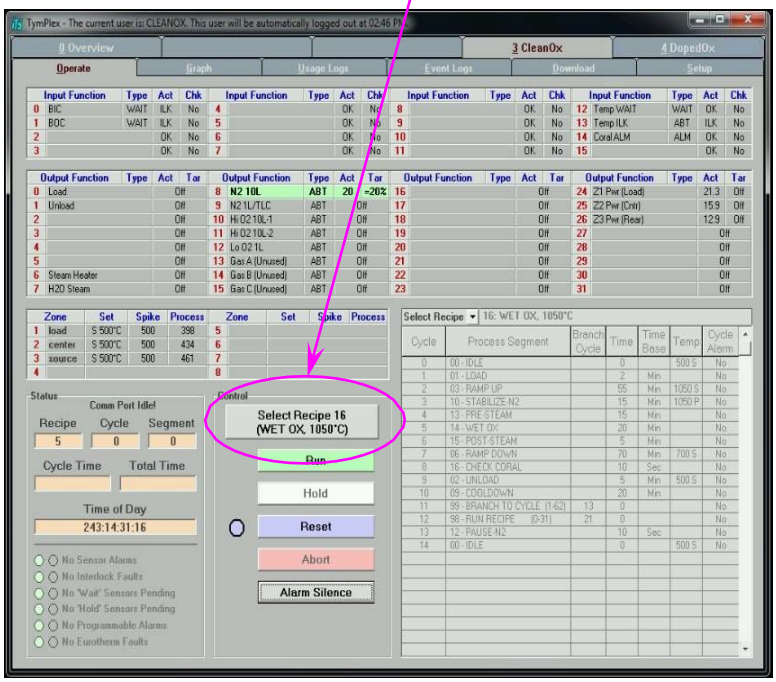
a. Select the operate subtap
b. Click select Dropdown box. 

c. Select the desired recipe.
d. Activate the recipe by clicking select Recipe # Button

Select Recipe pull down box

Select Recipe button

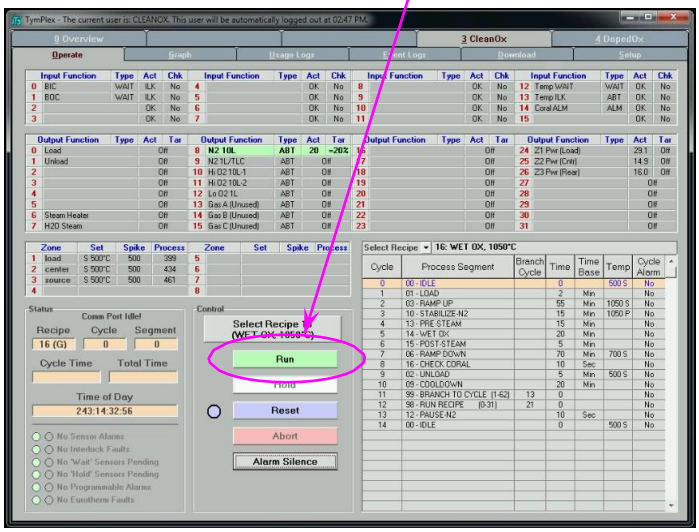




8. Run The recipe:

In the **Operate** subtab, click RUN button.

Run



9. After completing the process turn Off the Bubbler valves and keep the bypass open. Inform the facility person to turn off the gases (keep the N2 On for purging.). Load and run standard Purging recipe.

EMERGENCY RESPONSE

Power shut down:

1. If you have some time remaining before the power shut down follow the following steps
   1a) Abort the process running in the TYMPLEX.
   1b) Switch off the furnace power (switch marked as top corresponding to the phosphorous diffusion tube).
   1c) If the temperature of the tube is high > 800^{o}C and your samples are inside. Since we cannot wait for the temperature to reach the unloading temperature leave the sample inside itself. Else if the temperature of the tube is the unloading temperature and power is available for the time to unload the samples, unload and close the loading station.
   1d) shut down the PC.
   1e) Switch off the furnace control unit power (labelled as 120 V service)
   2. If the power to the furnace is suddenly not available.
   2a) switch off the furnace power and the control unit power wait for the power to come to unload the wafer or plan a new process

Gas detector Alarm triggered:

1. It automatically shuts off the lo-nitrogen flow through the bubbler once the alarm is triggered.
2. Immediately get out of the clean room and inform the facility staff.

If alerted to go out of the lab immediately:

1. Abort the process running in the PC.
2. Switch off the furnace power (switch marked as top corresponding to the phosphorous diffusion tube).
3. Run the recipe named as emergency recipe (on desktop) which will facilitate the flow of high flow N_2 during the ramping down.
4. Leave the lab (all the above processes can be done within a minute. If it's taking a long time please mind to leave the lab without taking care of all this).

System malfunction:

Contact the system owner, other authorized users for help.

STANDARD PROCESS RECIPE:

To understand the overall process flow a common recipes has been given below.

$POCl_3$ Process

Baseline $POCl_3$ Recipe for 100 ohms/cm^2

Step #	Name	Time	Temp	Gas Flow in SLPM
0	Idle	----	750C	N_2@8.15L
1	Boat In	10'	835C	N_2@8.15L, O_2@0.45L
2	Oxide	5'	835C	N_2@8.15L, O_2@0.45L
3	Stabilize	15'	835C	N_2@8.15L
4	$POCl_3$ Deposition	10'	835C	N_2@8.15L, O_2@0.45L, $POCL_3$@0.45L
5	Purge	5'	835C	N_2@8.15L, O_2@0.45L
6	Drive in	Note 2	Note 2	N_2@8.15L, O_2@0.45L
7	Boat Out	----	750C	N_2@8.15L
8	Idle		750C	N_2@8.15L

Baseline $POCl_3$ Recipe for 50 ohms/ cm^2

Step #	Name	Time	Temp	Gas Flow in SLPM
0	Idle	----	750C	N_2@8.15L
1	Boat In	10'	860C	N_2@8.15L, O_2@0.45L
2	Oxide	5'	860C	N_2@8.15L, O_2@0.45L
3	Stabilize	25'	860C	N_2@8.15L
4	$POCl_3$ Deposition	18'	860C	N_2@8.15L, O_2@0.45L, $POCL_3$@0.45L
5	Purge	5'	860C	N_2@8.15L, O_2@0.45L
6	Drive In	Note 2	Note 2	N_2@8.15L, O_2@0.45L
7	Boat Out	----	750C	N_2@8.15L
8	Idle		750C	N_2@8.15L

Baseline $POCl_3$ Recipe for 10 ohms/ cm^2

Step #	Name	Time	Temp	Gas Flow in SLPM
0	Idle	----	750C	N_2@8.15L
1	Boat In	10'	960C	N_2@8.15L, O_2@0.45L
2	Oxide	5'	960C	N_2@8.15L, O_2@0.45L
3	Stabilize	25'	960C	N_2@8.15L
4	$POCl_3$ Deposition	18'	960C	N_2@8.15L, O_2@0.45L, $POCL_3$@0.45L
5	Purge	5'	960C	N_2@8.15L, O_2@0.45L
6	Drive In	Note 2	Note 2	N_2@8.15L, O_2@0.45L
7	Boat Out	----	750C	N_2@8.15L
8	Idle		750C	N_2@8.15L

NOTES: 1) Use these recipes as baselines for sheet resistances of 10, 50 & 100 ohm/cm^2. Fine tuning is always required for process optimization
2) If Drive-In is required typical process is 36 minutes at 860C