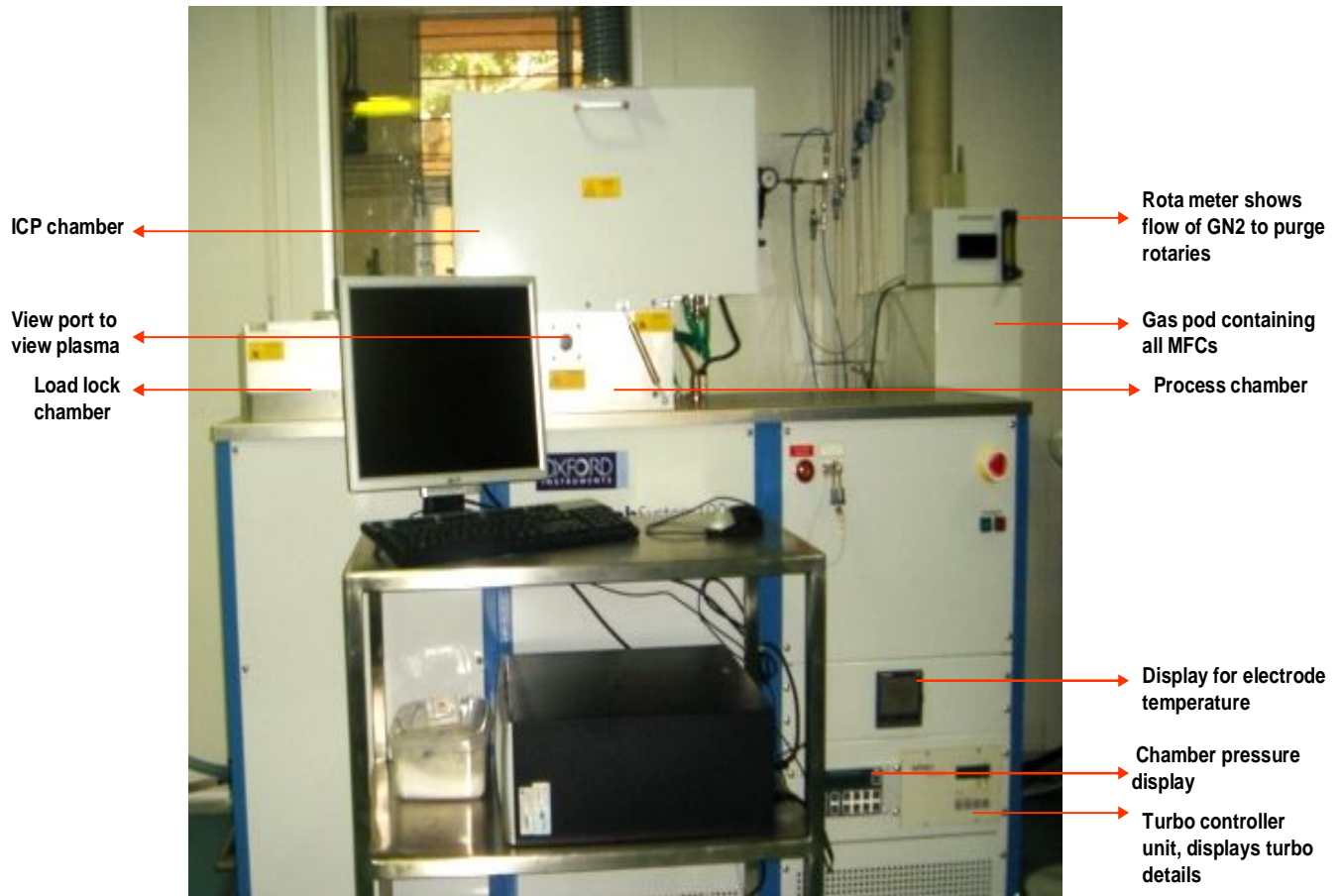


# ICP- CVD

## Inductively Coupled Plasma - Chemical Vapor Deposition

Tool Identifier	Plasma lab System 100 ICP180
Document version:7.0	Date:11 <sup>th</sup> July 2019



## Inductively Coupled Plasma Enhanced Chemical Vapor Deposition Tool

<b>Name of the Equipment</b>	Inductively coupled plasma CVD (ICPCVD)
<b>Category</b>	semi clean b
<b>Operator</b>	Vivek Surana, Pijush Chakraborty Navneet Bhardwaj
<b>System Owner</b>	Hitesh Kamble hit.kamble@gmail.com
<b>Short Name</b>	ICPCVD
<b>Make</b>	Oxford Instruments
<b>Model</b>	Plasma lab system100
<b>Serial Number</b>	94-814852
<b>FootPrint</b>	Length 1.93 mtr , Breadth 2.28 mtr, Height1.57
<b>InstallationDate</b>	08/01/2008
<b>Equipment Type</b>	Deposition, Growth and Annealing systems
<b>Location</b>	Nano Lab
<b>Local Dealer</b>	Aimil Ltd  Amol Gawade Sr. Service Engineer Aimil Ltd., Mumbai Naimex House 11th Floor 'B' Wing, Bsel Tech Park Sector 30A, Opp. Vashi Rl. St. Navi Mumbai - 400 705 India Tel. : +91 22 39182698 +91 22 39183554/55 Board line Mobile : +919920271186 Fax : +91 22 39183562
<b>Actual Dealer</b>	Oxford instruments  thomas.miller@oxinst.com
<b>Tool Facilities Requirements</b>	Nitrogen, Argon, Silane, CF4, N2O, Ammonia, Hydrogen, Water connection
<b>Lab Phone No</b>	4411

<b>Substrate allowed</b>	Si/Ge/GaN/Quartz/GaAs
<b>Substrate Dimension</b>	Pieces to 4 inch wafer
<b>Material allowed</b>	Si,TiN,ZnO, GaN,GaAs,Al
<b>Chemical allowed</b>	IPA for cleaning
<b>Gases allowed</b>	GN <sub>2</sub> ,PN <sub>2</sub> ,Ar,N <sub>2</sub> O,SiH <sub>4</sub> ,NH <sub>3</sub> ,H <sub>2</sub> ,He,CF <sub>4</sub> /O <sub>2</sub> for cleaning
<b>Contamination remarks</b>	Gold contaminated wafers not allowed.

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## **Warning**

- **After checking if the following conditions are satisfied only, one should enter the lab to start the system:**
  - The 3 water lines – Turbo: 1lpm, RF Generator: 8lpm, ICP/ AMU: 2lpm are fine
  - The 2 exhausts are switched ON- Leak exhaust and Turbo exhaust
  - High purity Nitrogen (PN2) cylinder is at 4bar
  - GN2, Commercial Nitrogen Plant is working and giving a minimum pressure of 6bar
  - The AHU in the lab is working
  - There is no condensation of water along water lines going into the system.
- Please don't open the process chamber under any circumstances without consulting the system owner.
- **The authorized users are allowed to carry out only the 'Automatic Process Run' on their own, processes at >200C needs to be done in the presence of SO's or by SO's. Tool must be operated manually beyond 200C by experienced user only**
- Whenever the transmission disc or wafer clamp needs to be changed, please consult the system owner.
- Make sure that the processes you want to run are contamination protocol compliant.  
person(s).
- Refer to the **people list** in case of any doubt or emergency and consult the relevant

## People List

System Owner:Hitesh Kmable	hit.kamble@gmail.com	9096066365
Authorized User:Vivek Surana	viveksurana89@gmail.com	
Authorized User:Navneet Bhardwaj	nav.bhardwaj07@gmail.com	
Authorized User:Pijush Chakraborty	pijushmatsc@gmail.com	
Authorized User:Hemant Kshirsagar	hemantvkshirsagar@gmail.com	
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Lab Manager: SatyaValli Palluri	valli@ee.iitb.ac.in	
Faculty In-Charge: Prof. Dipankar Saha	dsahaiitb@gmail.com	

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Plasma Technology  
(OIPT): OIPT Engineer

## **Introduction**

**Plasma Enhanced Chemical Vapor Deposition (PECVD)** is a process to deposit thin films from a vapor phase to a solid state on a substrate. There are chemical reactions involved in the process which occur after creation of plasma of the reacting gases or vapors.

### **Plasma**

- Ionized gas
- High free electron content
- Unique state of matter

### **Inductively coupled plasma (ICP)**

Plasma can be generated by two ways: DC discharge and RF discharge.

DC discharge is not useful in depositing insulating materials because plasma created gets extinguished as soon as a layer of insulator is deposited. Plasma driven from AC signal can solve this problem.

When a time varying electric current is passed through a coil, it creates time varying magnetic field around it, which in turn induces azimuthal currents in the rarified gas leading to break down and formation of plasma. Thus plasma is generated by electromagnetic induction. The RF coil around the ICP chamber is operated at 13.56MHz and with a maximum power of 1KW.

### **Advantages of ICP-CVD over conventional PECVD**

- High density plasma (higher than capacitively coupled systems)
- Low deposition pressures
- Low operation temperatures
- Lower chances of cracking deposited layers
- Good dielectric properties of deposited layers
- Good step coverage (by deposition + etch process)

### **Disadvantages of ICP-CVD**

- Toxic byproducts
- High cost of equipment

### **Applications of thin films deposited by ICPCVD**

- Passivation layer for packing of devices
- Solar cells, optical fibres – Amorphous silicon and anti reflective coating
- Antireflection and antiscratch layers in optics
- Interlayer dielectric

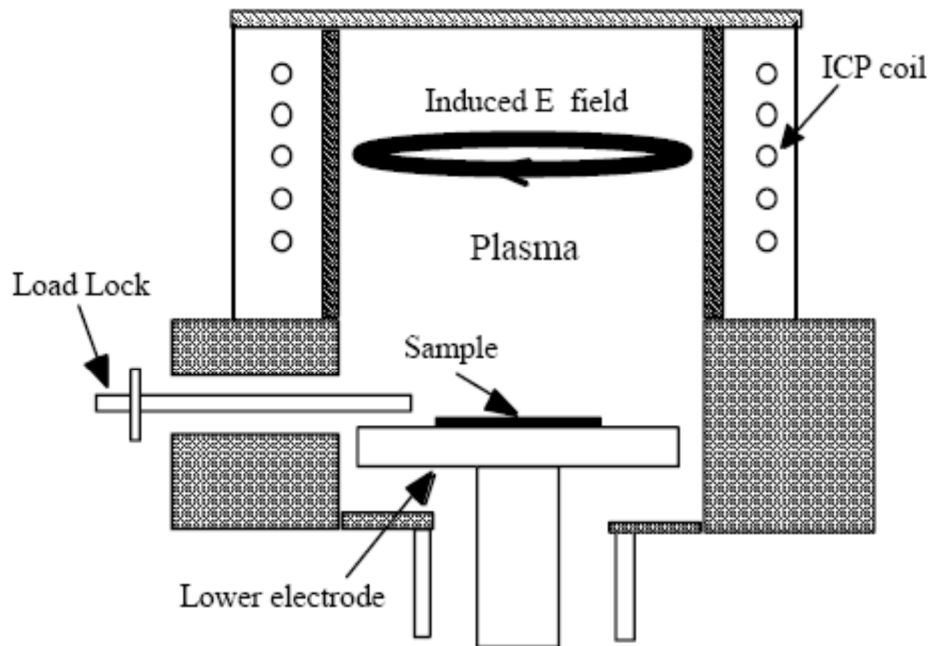
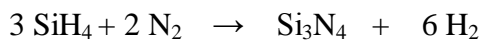
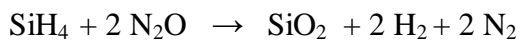


Fig. 1. Schematic diagram of the ICP CVD system.

Helium gas –Some pressure is applied to the back of wafers to provide good thermal contact between chuck and wafer. All process gases pass through a transmission disc and Silane is introduced through a gas distribution ring before entering the Process Chamber. [1]

**Chemical reactions for the Silicon dioxide and Silicon nitride depositions:**





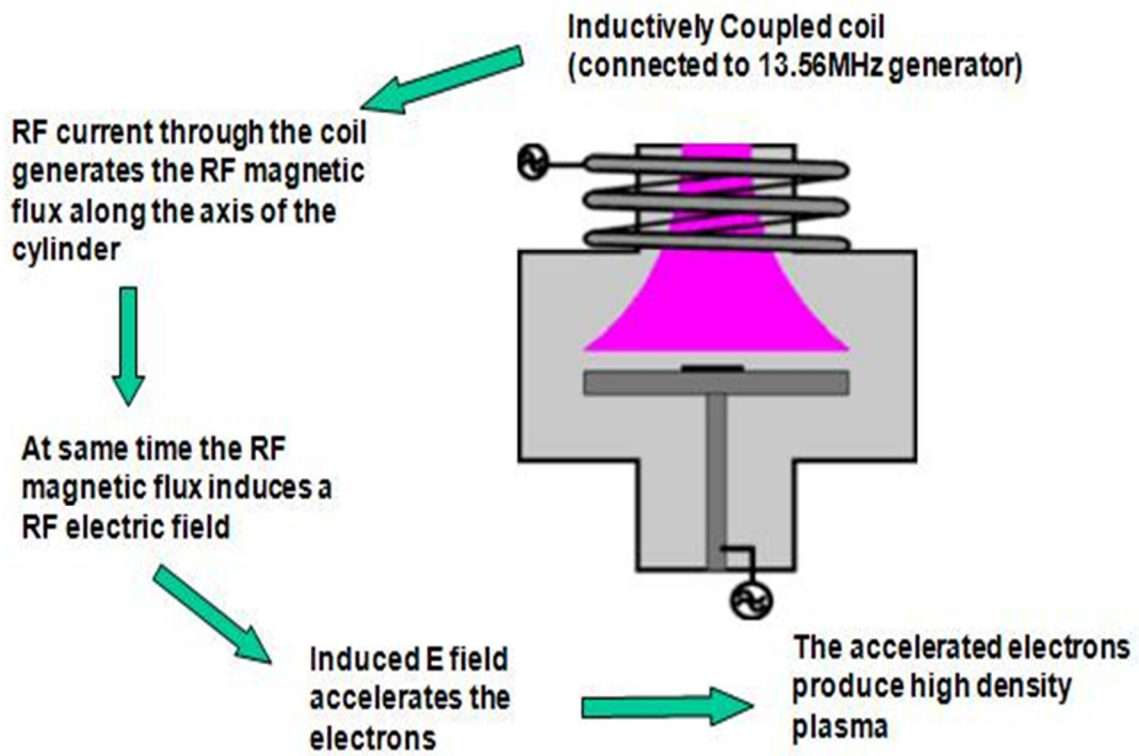


Fig.2. Plasma Strike [2]

## Specifications



- **Substrate:** Si, GaN, GaAs, quartz, glass, other wafers (subject to contamination clearance, presently given by Prof. Anil Kottantharayil).
- **Substrate size:** 2", 4", 8" wafers and small pieces.
- **Types of depositions:** Silicon nitride, Silicon dioxide, Silicon oxynitride, Amorphous Silicon.
- **Vacuum attained :** Load lock chamber ~  $10^{-3}$  Torr ;  
Process chamber ~  $10^{-7}$  to  $10^{-9}$  Torr.
- **Temperature of substrate:** room temperature to 350degC.
- **ICP power :** 3KW (maximum)
- **Substrate bias power :** 300W (maximum)
- **Gases presently connected to system:** Silane, Nitrogen, Ammonia, Nitrous oxide, Argon, Helium, CF<sub>4</sub>/O<sub>2</sub> (clean gas).
- **Mass Flow Controller (MFC) Limit/Range :**  
Ar: 200sccm, SiH<sub>4</sub>:50sccm, N<sub>2</sub>O:50sccm, NH<sub>3</sub>:50sccm.

## Operating Procedure

### Precautions

- The system can be operated by authorized users only. The names of the authorized users can be found in the *people list*.
- All the gas cylinders must be open to 20~30psi, Helium gas to be open to 15psi, SiH<sub>4</sub> to be open to 12psi. **Gas cylinders to be operated only by lab staff trained in handling specialty gases.** Please check the *people list* for the names.
- Rota meter reading should read between 4.5 and 7slpm: adjustment can be done only after the System starts some procedure or the other. It displays nitrogen flow for purging rotary pumps.
- Silane gas is connected to Furnaces and ICP-CVD System with a T-connection in the gas cabinet located in the service corridor. Care must be taken while opening the cylinder, i.e. **the other line to Furnaces should be closed.**
- **Coordination with RTP, ICPRIE users** is a must as all 3 systems share the same gas lines of Ar, N<sub>2</sub>. Also, RTP uses high power, hence deposition must not be made when RTP is ON, i.e., **system can be ON but the RF generators must not be ON.**



## SWITCH ON Procedure


- Start the three water lines
  - Turbo: 1lpm RF Generator: 8lpm ICP/ AMU: 2lpm
- PN2: 4 bar, High purity nitrogen used as a precursor gas, and to purge turbo molecular pump, process chamber, load lock chamber. We have 2 MFCs for PN2. MFC rating for purging is 100sccm; MFC rating for process gas is 50sccm.
- GN2: 6bar, Commercial grade nitrogen used for operating solenoid valves and purging rotary pumps.
- Check if Process Chamber leak Exhaust and Turbo Exhaust are ON.
- Switch ON the mains of the system. Turn the green button of the System ON.
- Open valves for PN2, GN2, and Helium inside the lab.
- Turn ON the PC.
- Choose the software **PC2000**.
- We get a display as
  - Name **OPT** Password **\*\*\*\*\***
- Click **OK** ..... Is load lock empty **OK**.....Sure **OK**
- Go to **System**.....**Service**.....
- To activate both the Rotary pumps, click on the schematic for pumps. When a yellow lining appears around the pumps, we have control over them. The pumps will start working.
- Go to **System**.....**Service**.....**Exit Service**.....
- We get a display: **Danger, Machine is about to reset, Stand clear....This means the machine will reset to home position** .....click on **OK**.
- We are in the **Pump Control** page now.
- Wait for about 20min., for warming up of the pumps.
- Click on  for both chambers.
- We get a display...**Enter Wafer ID**...**Cancel** if there is no wafer/give ID otherwise, and then click on **OK**.
- Wait for a few minutes, Turbo pump starts, initially... **accelerates**, after ~6minutes it shows... **At speed**. This means the Turbo is at its maximum speed of 34000rpm. And we need to wait for the Process Chamber to reach the required vacuum  $\sim 10^{-7}$  Torr, Load lock chamber at a vacuum  $10^{-3}$ Torr.
- When both the chambers are ready, green arrows  appear on both the chambers.
- Now the system is ready for all processes.

## To increase substrate temperature

- For depositions beyond 300degC, wafer clamp must be removed. At 300degC, wafer clamp need not be removed.
- For depositions higher temperatures above 100degC, please use heat resistant gloves.
- Go to...**Process**...**Chamber1**.
- Give a relevant time to **STEP TIME**. This time does not signify ramping time for increase in temperature. This time is the process time that starts countdown after the electrode reaches the desired temperature.
- Feed the temperature parameter in **CRYO**.
- Please increase the temperature in steady steps and also take care of the overshooting of temperature. To avoid overshooting, increase the temperature in smaller steps as you get closer to the required temperature.
- For example, if the required temperature is 250degC. When we start at room temperature, say, 25degC, we increase temperature in steps of 50 degrees initially. When we reach 225degC, we increase temperature in steps of 5 degrees.
- All other parameters in this page must be zero.

## Procedure for Deposition – Automatic Process Run

- Check the log book, **if the thickness deposited since last cleaning exceeds 2um** or if you are **depositing a different film run the cleaning recipe.**
- Go  to....**System**....**Pumping**....
- for Load lock Chamber.
- After Load lock Chamber reaches atmospheric pressure, the Load lock Chamber lid can be opened.
- Load the appropriate carrier inside the chamber and place the sample on it. *A list of carriers used for different kinds of substrates is given in the table below.* **Utmost care must be taken to not drop the sample inside the Load Lock Chamber.**
- Close the lid      the Load lock Chamber.
- A Dialogue Box appears for **Wafer ID**. Enter your wafer ID.
- After the Load lock Chamber attains  $10^{-3}$ Torr, green arrows   appear on the Load lock Chamber.
- Select **Process**, choose **Recipe** option.
- Open the valves for all the required process gases inside the lab.
- Drag the relevant recipe using the 'mouse'.

- **Ask the facility people to switch ON the scrubber just before introducing toxic gases. After process is finished inform facilities for switching OFF scrubber.**
- **Run the recipe.** It can be **Edited** according to the requirements. **Helium pressure is always set at 10Torr.** The Helium flow is taken by the system according to the requirement. **We do not set the Helium flow rate.**
- After deposition, a yellow alert saying **Process Complete** is displayed....**Accept.**
- Automatically the wafer moves from Process Chamber to Load lock Chamber.
-  For Load lock Chamber.
- Open Load lock Chamber and remove the sample.

Description of the substrate	Description of the carrier to be used	Treatment of carrier before processing batch/sample	Treatment of carrier after processing the batch/sample
Silicon wafers (2", 4" or 8" diameter) subjected to CMOS processing and not contaminated with metals and photo resist.	8" Pocketed Silicon Carrier1.	Etching in 2% HF.	Etching in 2% HF.
Silicon wafers contaminated with Na, K or any other metallic contaminants. E. g.: Solar cells wafers processed in a typical cell manufacturing facility	Al carrier1 designated for the purpose	Carrier is plasma cleaned in the chamber using 'chamber cleaning' procedure.	Carrier is plasma cleaned in the chamber using 'chamber cleaning' procedure.
GaAs or other III-V semiconductor samples	Al carrier2 designated for the purpose	Carrier is plasma cleaned in the chamber using 'chamber cleaning' procedure	Carrier is plasma cleaned in the chamber using 'chamber cleaning' procedure
Other samples	8" Pocketed Silicon Carrier2	Etching in 2% HF	Etching in 2% HF

## **Evacuation of gas lines**

**Evacuation to be done up to the valves inside the lab at the end of the day.**

**Evacuation to be done up to the cylinder at the end of the week, i.e., Friday.**

- Close the gas cylinders and keep the regulators open or only the valves of the gases inside the lab as the case may be.
- At a time, one gas line is to be evacuated.
- Go to **Process.....Chamber1**.
- All parameters on this page must be zero. **PUMP TO PRESSURE...must not be ticked.**
- **CRYO**.....feed the temperature of the electrode at that point of time.
- Feed 15 min **Step Time** value for non toxic gas and 30 min for toxic.
- Give maximum possible suitable value for the MFC which is to be evacuated.....**Start**.
- Watch the flow rate, it must show 0sccm.
- If you do not get this flow rate, start the evacuation step again.
- This will evacuate the gas lines.
- MFC Limit/Range :  
N2: 50sccm, Ar: 200sccm, SiH4: 50sccm, N2O: 50sccm, NH3: 50sccm, H2: 200sccm.

## **Procedure for SHUTting DOWN the system in normal course**

- Shutting down of the System must be done only after all the gas lines have been evacuated according to **Evacuation of gas lines** procedure.
- Go to **Pumping page**.....click on '**STOP** for all automatic processes' which is on right hand side top corner of the page.
- The Turbo molecular pump will start 'breaking'. A window opens asking '**Do you want to stop all processes?**'... Click on '**Yes**'.
- Turbo starts decelerating, and finally comes to a halt showing 0rpm. **Till the turbo reaches 0rpm, please do not switch OFF the mains of the system.**
- Go to **System .....Exit .....Are You Sure.....Yes**.
- **Switch OFF** using red button on System. The rotary pumps stop.
- **Switch OFF** the mains for **the PC and the system**.

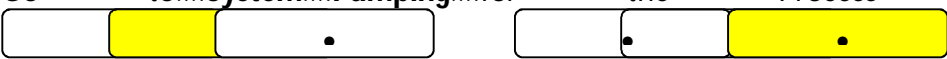
## Procedure for SHUTting DOWN the system in case of power failure

- While the process is on, if there is a power failure, we have about 20min. time to Shut down the System.
- If you are in the middle of a process, you can click on **STOP** on the recipe page.
- Immediately, a display will be seen asking if the process is to be **Aborted, Accept** and the sample will come to the Process Chamber automatically.
- **SHUT DOWN** the system according to the given procedure.
- After power returns, start the system according to normal **SWITCH ON** procedure.

## Procedure for SHUTting DOWN the system in case of an emergency

- If one needs to **SHUT DOWN** the system on an emergency, say, while a process is going on, it can be done by pressing on the **RED EMERGENCY BUTTON** located on the right side of the tool. When this is done all automatic processes will stop immediately and all solenoid valves will close so that no gases will enter the Process Chamber.
- Switch off the mains.
- After the situation is under control, **SWITCH ON** the system according to the normal procedure. If the 'arm' is in the Process Chamber, **wait**, it will come back automatically into the Load lock Chamber.
- After that, one can proceed with depositions.




## Changing Transmission Disc and Wafer Clamp

- Go to....**System....Pumping....**for the Process Chamber  
....it takes ~20min.
- **Venting Time Left** is displayed on the screen. When it shows zero seconds, the Chamber can be opened.
- To change the disc and wafer clamp proper care must be taken. At least 2 people are needed to perform this operation.
- **Do not drop screw/screw driver in the chamber. This will damage the wafer clamp. Locating the screw will also be a problem.**
- After this, close the Process Chamber,



## Chamber Cleaning Procedure – Semi Automatic Process- CF4/O2 (80:20)

### Plasma clean

- Preferably done at room temperature, as during cleaning procedure the temperature keeps increasing.
- **A DUMMY WAFER MUST BE USED.** This protects the lower electrode. Otherwise there could be sputtering of the electrode.
- After deposition of every 2 microns, **extensive cleaning has to be done.**
- When different kinds of depositions are done, **cleaning is essential.**
- **Cleaning should be done till the color of plasma changes to white.** If the color doesn't change in given time run the recipe again.
- Whenever the Process Chamber is opened, **cleaning is to be done.**
- Go to .....**System**.....**Pumping**..... for Load lock chamber  
 place dummy wafer  
 (Wait till the required vacuum  $10^{-3}$  Torr is attained).
- **Click on wafer**, we move to the **Robot Control** page now automatically. **Click on destination, i.e., Process Chamber.** Wafer gets transferred to **Process Chamber**.....Go to **Process**.....**Recipe**.....**Load CLEAN CHAMBER**.....**Edit** if needed....**Run**.
- A dialogue box, **WARNING**.....**There is a wafer in the process chamber, do you wish to continue**....**appears, click on YES.**
- After plasma cleaning is done, **click on wafer** which is in **Process Chamber**, **click on destination, i.e., Load lock chamber**...wafer gets transferred to **the Load lock Chamber**.
- Go to.....**System**.....**Pumping**.....for Load lock Chamber  

- Open the Load lock Chamber after it reaches atmospheric pressure. Remove the dummy wafer.

### Recipe for Cleaning Chamber

#### **STEP1**

- Set Pressure.....25mTorr MFC for CF4/O2.....150sccm
- CRYO.....Temp of electrode at that point of time. Time.....25min.
- RF .....100W ICPRF.....1500W




## STEP2

- Set Pressure.....20mTorr                      MFC for CF4/O2.....100sccm
- CRYO.....Temp of electrode at that point of time.                      Time.....20min.
- RF.....100W                      ICPRF.....1500W

## Process Gas Lines Leak Testing

*This must be carried out if there is a long gap of not using the system or if there is a doubt of any leakage of the gas lines.*

- Switch **ON** the System according to the **SWITCH ON** procedure.
- After the green arrows  appear on both the chambers, go to ....
- **Process.....Chamber1.....Leak Detection.**
- On this page, suitable parameters for **Step Time**, Log Interval, have to be fed. All other parameters must be zero. Pump to Time is to be **ticked**.
- Click on **Mass Flow Calibration**, all MFCs will be displayed.
- Choose one gas line at a time. Close the respective valve for the gas line. Feed the value for the MFC.....**START**.
- Depending on the time we have set for **Pump to Time**, the line will be evacuated. After that we get a graphical display of the leak rate.
- The leak testing will go on till the given **Step Time** is completed.
- Leak rate must not exceed **1mTorr/min**. If it does, **please contact the system owner immediately.**

## Clean Gas Line Leak Testing...CF4/O2

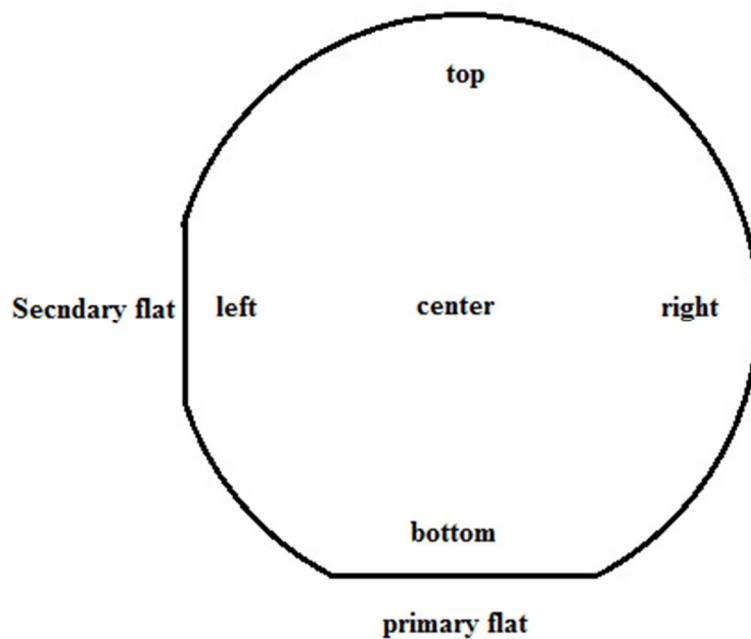
- Go to ....**System.....Process.....Recipe.....Clean.....Load.....Clean MFC Test.**
- MFC value for CF4/O2 must be given as 300sccm.....**Run**.
- Leak rate must not exceed **1mTorr/min**. If it does, **please contact system owner immediately.**
- Watch the flow rate, it must show 0sccm.
- If you do not get this flow rate, start the evacuation step again.
- This will evacuate the gas lines.

## Check List before Leaving the System

- All gas lines evacuated up to the valves inside the lab everyday and up to the cylinder at the end of the week.
- PN2, GN2 and all process gas cylinders are closed. **Check if someone else in the lab is using the common cylinders for their systems before closing.**
- Turn OFF the water lines.
- Switch OFF the mains for the System and PC.
- Check if the log book has been updated. Check if the gas cabinet sheet is updated.
- Keep the surroundings clean.

### **Characterization**

- *It is mandatory for all users to do Ellipsometry immediately after deposition and enter the actual thickness in the ICPCVD log book along with the relevant process parameters.*



## Trouble shooting

- Whenever plasma doesn't strike, change the chamber pressure parameter and try.
- Plasma may not strike if the chamber pressure is less than 20mTorr sometimes. Use the **Low Pressure Strike** option. Typical values are **25,20,8**, corresponding to **Pressure, DC bias, ramp down time (in seconds) of pressure** respectively. This means that to start with the chamber pressure will be **25mTorr** to help in striking the plasma, there will be a bias of **20volts** given to the lower electrode, and in **8seconds** the pressure will ramp down to the desired chamber pressure according to the recipe.
- Whenever reflected power is high, try again changing chamber pressure OR the recipe can also be run by clicking on **HOLD** on the **Recipe** page. This means the automatic matching unit (AMU) chooses the last best position for plasma strike.
- Whenever plasma is flickering, click on 'manual', give appropriate values for capacitor positions and click on '**START**' in the recipe page during deposition only.
- If still there is flickering, give required value to APC valve position in degrees, click on '**Position**', and click on '**START**' on the recipe page.
- If points 4 and 5 do not work within a few seconds, recipe gets 'Aborted' and wafer is out of the chamber.
- If there is a wafer inside the system, but for some reason the wafer is not being recognized by the software or the vice-versa, there is a way to include/exclude the wafer. Go to.... **System ...Service**....Click on the location (Load lock or Process Chamber) where the wafer is supposed to be,.....we get on to the **ROBOT CONTROL** page which shows a dialogue box.....**Add/Kill wafer**. The appropriate option can be chosen.....**Exit Service**.

## Maintenance

- Load lock chamber to be wiped with IPA
- Process Chamber to be **only wiped dry with lint free cloth.**

## Contact Details

- Local Engineer; A.M.Bapat, "**A M Bapat**" <[ambapat@aimil.com](mailto:ambapat@aimil.com)>  
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- Plasma Technology, North End, Yatton, Bristol BS49 4AP UK  
[www.oxford-instruments.com](http://www.oxford-instruments.com)
- Process Engineer, Mr. Dave Exon: **HOTLINE Tel.no. ; 00441934837070**  
Engineers available 8am to 8pm (UK Time)

## REFERENCES

[1] Deposition of ammonia free silicon nitride using inductively coupled plasma at low temperatures – H.Zhou, C.Sim, C.Hodson, R.Kinsey, C.D.W.Wilkinson.  
Nanoelectronics centre, Department of electronics and electrical engineering,  
Glasgow Univ., UK OIPT, Bristol, UK - ISPC 16

[2] Customer Presentation – OIPT