Purpose of this Instrument: The Oxford Plasmalab 80+ PECVD is used for the deposition of silicon oxide and silicon nitride films.

Location: ESB G75 Cleanroom

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The Shared Research Facilities are operated for the benefit of all researchers. If you encounter any problems with this piece of equipment, please contact the staff member listed above immediately. There is never a penalty for asking questions. If the equipment is not behaving exactly the way it should, contact a staff member.

START-UP

1. Sign into the FOM software.
2. Before entering the cleanroom, check to ensure that the roughing pump is operating in the chase. If the pump is not operating, please notify a Shared Facilities staff member.
3. While in the chase area, turn ON the process gas bottles for your process. The next section discusses how to properly open the gas bottles.
4. Inside the cleanroom, sign in on the logbook and record the chamber base pressure.
5. If the operating software is closed, open the PC PLUS software and select “Installation Engineer” to log in.
6. Press the PROCEED icon to be directed to the system Status Display screen.
**OPENING AND CLOSING PROCESS GAS BOTTLES**

**WARNING:** Do not attempt to change gas bottles. If a gas bottle is empty and needs to be changed contact a Shared Facilities staff member. Some of the gasses used in the PECVD processes are toxic or volatile.

**WARNING:** Failure to follow the proper sequence in the opening and closing of the process gas bottles could lead to exposure to toxic or flammable gases.

To initiate Silane flow, (See Figures 1 and 2 below):

**WARNING:** The 5% Silane used in the PECVD system will ignite in air and produce flames. Follow the procedure written, and do not open any additional valves. The additional valves are required to flush the gas lines when the gas cylinder is changed. Opening additional valves may create Silane pockets that may cause injury to persons servicing the equipment.

1. In the Silane gas cabinet, open the stem valve on the Silane gas cylinder.
2. Turn the valve labeled 2B in the gas cabinet so that it shows open.
3. Turn the valve labeled 2C in the gas cabinet so that it shows open.

*Figure 1: The gas manifold for the Silane gas.*
4. Press the black OPEN valve button on the gas safety monitor controller above the gas cabinet and verify an increase in output pressure on the regulator valve as show in Figure 2.

![Step #4]

Figure 2: Gas safety monitor controller located above the PECVD gas cabinet

5. When shutting off the Silane gas, press the RED button on the gas safety monitor controller to stop the flow of Silane gas and close the valves in the reverse sequence.

To initiate Ammonia flow, (See Figure 3 below):

**WARNING:** The Ammonia used in the PECVD system is toxic. Follow the procedure written, and do not open any additional valves. The additional valves are required to purge the gas lines when the gas cylinder is changed. Opening additional valves may create ammonia pockets that may cause injury to persons servicing the equipment.

1. In the Ammonia gas cabinet open the stem valve on the ammonia gas cylinder.
2. Check that the purge valve is open. It should be fully turned counterclockwise.
3. Turn the silver valve in the center of the gas panel to OPEN.
4. Open the regulator outlet valve and verify an increase in output pressure on the regulator valve.
5. To shut off Ammonia gas, close the valves in the reverse sequence.
Figure 3: The gas manifold for the Ammonia gas.

To initiate Nitrous Oxide flow, (See Figure 4 below):

1. Plug-in the heater

   **WARNING:** To avoid burns, do not touch the heater as it will become very hot during processing.

2. Open the stem valve on the gas cylinder.

3. Open the regulator output valve and verify an increase in output pressure on the regulator valve.

4. To shut off Nitrous Oxide gas, close the valves in reverse sequence and unplug the heater.

Figure 4: NOS heater and regulator valve control locations
SAMPLE LOADING

NOTE: The PECVD operates at a temperature of 300°C. This temperature is above most resists’ glass transition temperature. Running a PECVD process on samples coated with photoresist or other polymers may cause irreversible damage to the sample.

NOTE: The cathode and chamber walls build up residual silicon oxide and silicon nitride films with each run. It is recommended that the system chamber be cleaned after 10 microns of material deposition. Depositions occurring with a dirty chamber can adversely affect film quality and thickness. The process for chamber cleaning is defined in the CHAMBER CLEAN section.

NOTE: It is recommended that the chamber be conditioned with the process material if depositing a material that is different than the previous run or after the chamber has been cleaned. To condition the chamber run a short process for the material being deposited without a sample in the chamber to coat the cathode and chamber walls.

1. On the top toolbar above the System Display screen, choose the MANUAL ➔ STATUS screen to enter manual mode. The manual mode status screen is shown in Figure 5. This mode is used to vent the chamber and to return the tool to the idle state. Press the ABORT icon to stop any current processes.

Figure 5: System Control screen showing placement of icon to enter manual mode.
2. At the CURRENT ACTION section of the System Control screen, use the drop down menu to select VENT, as shown in Figure 6. Then press the green arrow icon next to START to begin the venting sequence. The chamber gate valve will close and the system chamber will be purged with nitrogen to bring the chamber up to atmosphere. The status bar will read VENTING CHAMBER.

![Figure 6: Status display of the system control screen](image)

3. The user should notice a rise in the CM GAUGE PRESSURE. Once the chamber reaches atmosphere the VAC/GAS POD INTERLOCK set-point will read FAULT and a hissing will sound can be heard from chamber around the lid.

4. Select the red square icon next to STOP to halt the flow of the vent nitrogen and the hissing sound from the nitrogen gas will cease.

5. Turn the chamber switch on the front panel of the tool to OPEN as shown in Figure 7.
Figure 7: Chamber switch for opening and closing of the chamber

6. Open the chamber by pressing both HOIST buttons simultaneously. The chamber lid will lift and swing backwards. Once the lid stops moving, release the HOIST buttons as shown in Figure 8.

Figure 8: Hoist button locations

7. Check that the chamber wall and cathode are clean as shown in Figure 9.
**NOTE:** If the chamber wall and cathode are not clean, it is recommended that the chamber be cleaned and conditioned. Instructions for cleaning the chamber are found in the CHAMBER CLEAN section of this SOP.

8. Place the samples on the cathode. Verify that the samples are not floating on a pocket of air by adjusting sample position with tweezers. Air pockets will cause the samples to move during processing, as shown in Figure 10. Users may add multiple samples to cover the cathode, but sample process time will vary from individual samples because the user is increasing the surface area to be coated.

**NOTE:** If the cathode is hot, then the samples will always float and will move during the pump down cycle.

*Figure 9: Image of cathode after cleaning.*

*Figure 10: Adjusting of sample position on the cathode.*
9. Turn the chamber switch to the CLOSE position.

10. Close the lid by pressing both HOIST buttons simultaneously. The chamber lid will swing forward and close. Once the lid is closed, release both HOIST buttons.

**RECIPE SET-UP**

**NOTE:** The cleanroom is a multiuser facility. It is recommended that users always verify process parameters and time settings prior to deposition. The Shared Facilities staff cannot guarantee that the recipe parameters have not been changed.

1. Choose EDIT→PROCESS.
2. Choose FILE→OPEN and choose a recipe to be edited in the File Selection window. Highlight the recipe and select OK.
3. Highlight and select each step to verify parameter setting(s) in pop-up window (Figure 11).

![Figure 11: Screenshot showing selection of Step #3 of siox600_SRF and process parameter screen.](image)

4. Once the process parameters and steps have been verified, choose FILE→SAVE. It is recommended that users name recipes using the following naming convention (Deposition type/Deposition time in seconds). For example:
   a. Silicon oxide deposition for 10 minutes → siox600
   b. Silicon nitride deposition for 5 minutes and 25 seconds → sinx325
5. Charts 1 and 2 display recommended process parameters for Silicon Oxide and Silicon Nitride. These parameters will generate a plasma and deposit a film. These may be adjusted as desired.

**Silicon Oxide**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Base Pressure</td>
<td>Pressure: 0.010 Torr</td>
</tr>
<tr>
<td>Process – Chamber IDLE Pump</td>
<td>Time: 00:15:00, Pressure: 0.010 Torr, Substrate Temp: 300°C</td>
</tr>
<tr>
<td>Process</td>
<td>Pressure: 1.00 Torr, Substrate Temp: 300°C, Rf: 20W, Gas Flow: (N2O=710 sccm), (Silane=170 sccm)</td>
</tr>
<tr>
<td>Chamber Vent</td>
<td>Substrate Temp: 300°C</td>
</tr>
</tbody>
</table>

*Chart 1: Silicon Oxide Parameters*

**Silicon Nitride**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Base Pressure</td>
<td>Pressure: 0.010 Torr</td>
</tr>
<tr>
<td>Process – Filling Process Lines w/ Gas Mixture</td>
<td>Time: 00:15:00, Pressure: 0.010 Torr, Substrate Temp: 300°C</td>
</tr>
<tr>
<td>Process</td>
<td>Pressure: 1.00 Torr, Substrate Temp: 300°C, Rf: 20W, Gas Flow: (NH3=35 sccm), (Silane=400 sccm)</td>
</tr>
<tr>
<td>Chamber Vent</td>
<td>Substrate Temp: 300°C</td>
</tr>
</tbody>
</table>

*Chart 2: Silicon Nitride Parameters*

6. Return to System Display by selecting MANUAL → STATUS.

**DEPOSITION**

**NOTE:** The following procedures are for operating the tool in Automatic Mode. Please contact a Shared Facilities staff member for assistance in operating the tool in Manual Mode.

1. Press the SELECT icon on the toolbar and choose the desired process recipe from the FILE SELECTION window. Then select OK to exit, as shown in Figure 12.
2. The SELECT icon will change to START and the Status Display will disappear. Press the START icon to begin the process.

NOTE: The icon on the toolbar can be pressed at any time to halt the process.

3. Select DISPLAY → STATUS on the top toolbar to open the Status Display (Figure 13) to monitor the current process step parameters.

The current step and actual measured values will be displayed inside the white boxes. Next to the actual parameters are process indicator bars, which may show grey, green, yellow or red.

- Grey indicates that the process parameter is not active.
- Green indicates that the process is running inside the set point tolerance
- Yellow indicates that the value is changing
- Red indicates that the process parameter is no longer within the set-point tolerances.

The system will halt the process and flash an alarm message if any gas parameter is out of tolerance. If this occurs, please refer to the PECVD Troubleshooting Guide. The recipe set points are shown next to the process indicator bars.
4. Once the deposition starts, monitor the gas flow set-points and verify that a plasma is present by checking through viewport on the front of the chamber lid (Figure 14). Record the following parameters in the log book:

- Recipe
- Temperature (°C)
- Gas flow rates (sccm)
- Forward and Reflected RF Power (W).
- Total Time (minutes)

**NOTE:** A high reflected power indicates that the auto matching network is not tuned properly. A poorly tuned network may generate a plasma, but the thickness of the deposition material may not be properly controlled. Halt the process by pressing the ABORT icon the process and contact a Shared Facilities staff member so that the matching network can be re-tuned.
5. The chamber will automatically vent to atmosphere, once the deposition is completed. Monitor the rise in pressure at the CM GAUGE PRESSURE. When the chamber pressure reaches atmosphere the VAC/GAS POD INTERLOCK set-point will read FAULT and a hissing sound from the nitrogen gas exiting the chamber around the lid can be heard.

**WARNING:** Do not abort or bypass the vent procedure after a process run. This system uses ammonia which can be toxic and Silane which is flammable. The system will properly purge the chamber during the vent process. For your safety, an interlock will prohibit the system from being open if the vent process is bypassed.

6. Choose the red square icon next to STOP to halt the flow of nitrogen. The hissing sound from the nitrogen gas will cease.

7. Turn the chamber switch on the tool to the OPEN position.

8. Open the chamber by pressing both HOIST buttons simultaneously. The chamber lid will lift and swing backwards. Once the lid stops moving, release the HOIST buttons. Using tweezers, remove sample(s) as seen in Figure 15.

**WARNING:** The cathode is HOT and will quickly heat metal tweezers.

**NOTE:** Sample(s) will be HOT. Let samples properly cool before placing in storage.
9. Once samples have been removed, turn the chamber switch to the CLOSE position.

10. Close the lid by pressing both HOIST buttons simultaneously. The chamber lid will swing forward and close. Once the lid is closed, release both HOIST buttons.

11. Select MANUAL → STATUS to go to the manual processing window.

12. At the CURRENT ACTION section of the System Display screen, use the drop down menu to select PROCESS, as shown in Figure 16. Click the green arrow icon next to the START to begin the pumping sequence.

![Figure 15: Sample removal from cathode.](image)

![Figure 16: Status display of the system control screen](image)
13. Manually enter the following idle parameters in the Chamber Parameters section of the System Display screen:
   - Chamber Pressure: 0.000 Torr
   - Substrate Temperature: 0°C

14. Exit the cleanroom and enter the chase area. Turn OFF the process gas bottles being used by following the process defined in the OPENING AND CLOSING GAS BOTTLES section at the beginning of this document.

15. Reenter the cleanroom and evacuate the process gas lines. Change the gas flow rate set-point(s) to 500 sccm in the Process Gases section of the System Display screen for each of the process gases which were used. The actual flow rate will change to 500 sccm and then decrease to a value between 0-40 sccm as the mass flow controller value opens and the excess gas in the line back is pumped out. Once the gas flow rate returns to 0-40 sccm, change the gas flow set-point(s) to 0 sccm.

16. The tool is now in a proper IDLE state.

### CHAMBER CLEAN

**NOTE:** The chamber should be cleaned for after 10 microns of material has been deposited.

1. Go into the chase area and open CLEAN gas bottle.
   
   To initiate CLEAN Gas (80% Tetrafluoromethane / 20% oxygen) flow: (See Figure 1 below.)
   
   1. Open the stem valve on the CLEAN gas cylinder.
   2. Open the regulator outlet valve and verify an increase in output pressure on the regulator valve.
   3. To shut off the CLEAN gas, close the valves in the reverse sequence.
2. Choose EDIT → PROCESS on the system control screen.

3. Choose FILE → OPEN and verify the CLEAN1 and CLEAN2 recipes are set to the following – see Charts 1 and 2 below.

### CLEAN 1 RECIPE

<table>
<thead>
<tr>
<th>Component</th>
<th>Parameter</th>
</tr>
</thead>
<tbody>
<tr>
<td>Base Pressure</td>
<td><strong>Pressure</strong>: 0.010 Torr</td>
</tr>
<tr>
<td>Process (80% CF4 / 20% O2)</td>
<td><strong>Time</strong>: 01:00:00, <strong>Pressure</strong>: 0.700 Torr, <strong>Substrate Temp</strong>: 300°C, <strong>Rf</strong>: 200W, <strong>Gas Flow</strong>:</td>
</tr>
<tr>
<td>Base Pressure</td>
<td><strong>Pressure</strong>: 0.010 Torr</td>
</tr>
</tbody>
</table>

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**Chart 1: Clean1 Recipe Parameters**

### CLEAN 2 RECIPE

<table>
<thead>
<tr>
<th>Component</th>
<th>Parameter</th>
</tr>
</thead>
<tbody>
<tr>
<td>Base Pressure</td>
<td>0.010 Torr</td>
</tr>
<tr>
<td>Process (80% CF4 / 20% O2)</td>
<td><strong>Time</strong>: 01:00:00, <strong>Pressure</strong>: 0.350 Torr, <strong>Substrate Temp</strong>: 300°C, <strong>Rf</strong>: 200W, <strong>Gas Flow</strong>:</td>
</tr>
<tr>
<td>Chamber Vent</td>
<td><strong>Substrate Temp</strong>: 300°C</td>
</tr>
</tbody>
</table>

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**Chart 2: Clean 2 Recipe Parameters**

**NOTE:** Both Clean 1 and 2 recipes should be used. If the chamber is still not clean, alternate cleaning procedures until it is clean.

4. The Clean recipes must start from a vented state. To vent the system, select MANUAL → STATUS to go to the manual processing window.

5. At the CURRENT ACTION section of the System Display screen, use the drop down menu to select VENT. Press the green arrow icon next to the START icon to begin the vent up sequence.

6. Monitor the rise in pressure at the CM GAUGE PRESSURE. When the chamber reaches atmosphere the VAC/GAS POD INTERLOCK set-point will read FAULT, and nitrogen gas can be heard exiting at the chamber lid.

7. Once the vent process is complete, press the SELECT icon on the toolbar and choose the CLEAN 1 recipe from the FILE SELECTION window as shown in Figure 2. Then select OK to exit.
8. The SELECT icon will change to START and the Status Display will disappear. Press the START icon to begin the process.

   **NOTE:** The icon on the toolbar can be pressed at any time to halt the process.

9. Select DISPLAY→STATUS on the top toolbar to open the Status Display to monitor the clean process step parameters.

10. Record the following parameters in the log book:

    - Recipe
    - Temperature (°C)
    - Gas flow rates (sccm)
    - Forward and Reflected RF Power (W)
    - Total Time (minutes)

    If the reflected power is high, notify a Shared Research Facility staff member to re-tune the auto-matching network.

11. Once the process is complete, the chamber will VENT to atmosphere. Monitor the rise in pressure at the CM GAUGE PRESSURE. When the chamber reaches atmosphere the VAC/GAS POD INTERLOCK set-point will read FAULT, and the nitrogen gas can be heard exiting the chamber lid.

12. Next, select the red square icon next to STOP to halt the flow of nitrogen.
13. Enter the MANUAL → STATUS to go to the manual processing window.

14. Choose the SELECT icon on the toolbar and select the CLEAN 2 recipe from the FILE SELECTION window. Then press OK to exit.

15. The SELECT icon will change to START and the Status Display will disappear. Press the START icon to begin the process.

16. Select DISPLAY → STATUS on the top toolbar to open the Status Display to monitor the clean process step parameters.

17. Record the following parameters in the log book:
   - Recipe
   - Temperature (°C)
   - Gas flow rates (sccm)
   - Forward and Reflected RF Power (W)
   - Total Time (minutes)

   If the reflected power is high, notify a Shared Research Facility staff member to re-tune the auto-matching network.

18. Once the process is complete, the chamber will VENT to atmosphere. Monitor the rise in pressure at the CM GAUGE PRESSURE. When the chamber reaches atmosphere the VAC/GAS POD INTERLOCK set-point will read FAULT, and the nitrogen gas can be heard exiting the chamber lid.

19. Next, select the red square icon next to STOP to halt the flow of nitrogen.

20. Turn the chamber switch on the tool to the OPEN position.

21. Open the chamber by pressing both HOIST buttons simultaneously. The chamber lid will lift and swing backwards. Once the lid stops moving, release the HOIST buttons.

22. Inspect the chamber and cathode for any residual film. If necessary, go back to step 2 and repeat.

23. Close the lid by turning the chamber switch on the tool to the CLOSE position, by pressing both HOIST buttons simultaneously. The chamber lid will swing forward and close. Once the lid is closed, release both HOIST buttons.

24. Once the chamber is clean, select MANUAL → STATUS to go to the manual processing window.

25. At the CURRENT ACTION section of the System Display screen, use the drop down menu to select PROCESS. Press the green arrow icon next to the START to begin the pumping sequence.

26. Manually enter the following idle parameters in the Chamber Parameters section of the System Display screen:
   - Chamber Pressure: 0.000 Torr
- Substrate Temperature: 0°C

27. Exit the cleanroom and enter the chase area. Turn OFF the CLEAN gas bottle according to the SOP at the bottle.

28. The tool is now in an IDLE state.

**FINISHING UP**

1. Clean up working area.

2. Verify that the required parameters and information are properly recorded in the log book.

3. Sign out on the FOM.

**EMERGENCY PROCEDURES**

If no one is available and the machine is not acting as expected, the user should do the following:

- Abort the process by pressing `<STOP>` on the software window.
- Send an email through the FOM by selecting **PROBLEM REPORT** and entering details of the issue in the **COMMENTS** section

Do not leave the machine running in an abnormal state. If the machine cannot be placed in **STANDBY MODE**, immediately contact:

**Primary Staff Contact:** Dr. Kolin Brown  
(304) 366-6551  
Office: ESB G75D  
kolin.brown@mail.wvu.edu

**Secondary Staff Contact:** Harley Hart  
(412) 443-1514  
Office: WH 409  
harley.hart@mail.wvu.edu

If it becomes necessary to leave the instrument then the user should leave a large, legible note on the **PECVD** stating the tool is **DOWN**. The user should also add a comment using the FOM software reporting the tool status.

If a dangerous situation is evident (smoke, fire, sparks, etc.), the user should press the **EMO** (EMERGENCY OFF) button on the top of the machine, ONLY if it is safe to do so. The user should notify all other cleanroom persons within the cleanroom to evacuate and leave the clean room immediately. The user should then contact proper emergency personnel from a safe place. The contact numbers below can also be found posted outside the clean room.