August 2008

Parylene CVD Operating Instructions

Purpose

The Parylene deposition system model 2010, by Specialty Coating Systems, is a vacuum system used for the vapor deposition of a Parylene polymer, onto a variety of substrates. Table 1 shows a few basic properties of the commonly used polymers. The coating is truly conformal and pinhole free. The clear polymer coating formed provides an extremely effective chemical and moisture barrier with high dielectric and mechanical strength. The system consists of a Vaporizer furnace, Paralysis furnace, Deposition chamber, Cold trap, and Vacuum pump. The Parylene CVD, shown in Figure 1, is located in the D-bay of the Scifres Nanofabrication Laboratory.

<table>
<thead>
<tr>
<th>Polymer Properties</th>
<th>Parylene N</th>
<th>Parylene C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dielectric Strength (V/mil)</td>
<td>7,000</td>
<td>5,600</td>
</tr>
<tr>
<td>Volume Resistivity (ohm-cm @ 23 C)</td>
<td>$1.4 \times 10^{17}$</td>
<td>$8.8 \times 10^{16}$</td>
</tr>
<tr>
<td>Melting Point (deg. C)</td>
<td>420</td>
<td>290</td>
</tr>
<tr>
<td>Deposition Rate (um/hour)</td>
<td>~0.75</td>
<td>~5.0</td>
</tr>
</tbody>
</table>

Table 1. Common Parylene Polymers and a few of their properties, for more details refer to the website.

Figure 1. The Parylene CVD furnace as seen in bay D of the BNC cleanroom. The deposition chamber is seen on top of the case which contains the furnaces as well as related power and control hardware.
Process Description

There are three main steps in the Parylene deposition process. Figure 2 shows Parylene N at each of the differing stages.

1. Vaporization:
   Parylene is vaporized from its solid dimer phase inside the vaporizer furnace starting at approximately 120 °C.

2. Pyrolysis:
   Pyrolysis occurs in a high temperature furnace (> 600°C) which converts the dimer to monomer phase by breaking the methylene-methylene bond. The temperature will depend on the type of Parylene being deposited.

3. Polymerization:
   Polymerization happens at room temperature on the substrate surface inside the deposition chamber. The pressure is ~0.1 torr yielding a mean free path of ~0.1 cm. The short free mean path helps achieve conformal application along with the polymerization process.

In summary, the system converts Parylene dimer to a gaseous monomer which propagates to the deposition chamber where the material polymerizes at room temperature onto the substrate. There is no intermediate liquid phase or separate cure cycle. The substrate temperature never rises more than a few degrees above ambient.

![Figure 2](image.png)

Figure 2. The three main steps in the polymerization process for Parylene N. The only difference for other Parylene types is the temperature of vaporization and pyrolysis.

- **LEAVE THE MACHINE Powered ON**
- If you have any questions, please contact Hasan Sharifi (hsharifi@purdue.edu) or Geoff Gardner (geoff@purdue.edu).
Preparing to Coat:

The instrument will typically be unloaded, cleaned and at room temperature.

1. Power on the PDS2010 by enabling the instrument in CORAL. Verify power by observing the ON indicator and that each of the temperature controllers is illumined.

2. Clean the chamber. If there is not already a layer of Parylene, wipe the chamber with a chem-wipe that has been sprayed with the provided soap to apply a very thin film to all interior surfaces of the process chamber and the cold trap, careful not to apply too much particularly on the top cover. CAUTION: Too thick a layer of soap may be a source of out-gassing and process contamination.

3. Prepare the source material boat by forming aluminum foil around the provided tube. Set the boat on the scale and zero it. Add material until you reach the desired amount. The Parylene C will deposit approximately 2 µm for each gram of material while Parylene N will deposit approximately 1 µm for each gram of material.

4. Load the sample by placing it on chamber carousel base plate. If needed install the turn table and distribution post. (the deposition rate will be slower due to the increased surface area)

Figure 3. On the left the control panel of the Parylene CVD system is shown. Left to Right, the controllers are, the vertical or pyrolysis furnace, chamber gauge, vaporization furnace, and pressure readout for the deposition chamber. The door to the vaporization chamber, where the Parylene filled boat is loaded, is shown on the right.

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Coating in Auto-Mode Operation:

1. Complete steps 1-4 in the Preparing to Coat section.

2. Verify that the cold trap is plugged in, and that there is no Parylene blocking the chamber gauge port. Figure 4 shows both of these.

![Figure 4. The cold trap is shown on the left. Circled in red is the plug to enable the device. This must be plugged in. During operation the cold trap will make an audible noise when filling. The interior of the deposition chamber is shown on the right. The Parylene monomer inters though the opening circled in green, while the chamber is exhausted through the opening circled in yellow. The vacuum gauge port shown in red must be clear.](image)

3. Set the temperature controllers at the desired settings as shown in the Typical Process Settings section.

4. Press the PROCESS START/STOP button, it will turn green. This enables the heaters.

5. Switch the FURNACE and CHAMBER GAUGE selector to enable. The heaters will begin to heat.
   a. The Chamber Gauge refers to the vacuum gauge for the process chamber. It is important to heat the gauge to at least 135, to prevent the Parylene from depositing on the gauge.

6. Switch the VAPORIZER selector to enable. The turn table will begin to rotate, this ensures even coating. The Vaporizer heater will not come on until the process all conditions (pressure and furnace temps) have been met.

7. Switch the Vacuum selector to vacuum to begin pumping of the system. This will enable the LN2 trap as well. It is necessary to hold the trap in position until the vacuum seals and the pressure begins to drop.

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a. The chamber should pump to the base pressure. It will take some time for the pyrolysis furnace to reach temperature. Once the chamber gauge, and the furnace temperatures are at temperature, the Vaporizer heater is enabled.

b. The Vaporizer will heat to the lower set-point temperature, vaporizing the dimer (which raises the pressure), as long as the chamber pressure remains below the Vacuum set point. The vaporizer will try to heat to its standard set point, being regulated by the vacuum controller.

c. Once the max set point is reached, and the pressure reduces back to the base pressure (below 5 mT), the vaporizer furnace will shut off after a 5 min wait period. The minimum process time is 30 minutes.

d. If the base pressure does not drop below 5mT or you need to stop the process,

8. Wait until the vaporizer pressure is less than 100°C before venting the chamber. Vent the chamber by turning the vacuum selector to vent.

9. Remove the cold trap from the exhaust port and place it in the holder.

10. After waiting ~10 min clean the cold trap by pealing the condensed Parylene spot from the trap. Be sure to coat it with the microsoap when finished.

11. After you remove your sample please clean the O-ring with chem-wipes, finishing with a fine coat of microsoap.

12. If you have removed any Parylene from the lid or any other surface you must coat the exposed surfaces with microsoap.

13. Finally, pick up after yourself and disable the device in coral.

Final notes on next page . . . . . .
NOTICE:
Make sure the Liquid Nitrogen (Cold Trap) is running during the process!!! This prevents contamination of the vacuum pump by Parylene.

You must apply Microsoap solution to all surfaces that will be coated with Parylene such as main chamber and cold trap.

After deposition, you MUST clean the vaporizer tube, the chamber and the cold trap thimble.

Adhesion Promoter:
Silane A174 is used as adhesion promoter for Parylene polymer.

Typical Process Settings:

Type C:
Vapor Heater Temp.: 175C
Pyrolysis Heater Temp.: 690C
Chamber Gauge Temp.: 135C
Pressure: Base pressure + 15 Vacuum Units (~35 mTorr)
Deposition Rate: 5um/hour

Type N:
Vapor Heater Temp.: 160C
Pyrolysis Heater Temp.: 650C
Chamber Gauge Temp.: 135C
Pressure: Base pressure + 55 Vacuum Units (~60-75 mTorr)
Deposition Rate: 0.75um/hour

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