PDS 2010 LABCOTER 2 PARYLENE DEPOSITION SYSTEM SOP

November 2013

Purpose:
This system is designed to deposit a thin film of Parylene, a polymer that, depending on the type of Parylene used, provides thermal, moisture, and dielectric barriers to properly prepared substrates.

Preparation and Precautions:
This process uses a vacuum system to move the Parylene vapor from its initial entry point, through the vacuum chamber and onto the sample(s), and finally, residual Parylene is drawn onto the refrigerated probe to which the unspent monomer is attracted, sparing the vacuum pump from being coated internally with Parylene.

Parylene is deposited at all surfaces along the path; therefore, care must be taken in order to clean the entire pathway. Some parts of this path are difficult to access and are thus periodically replaced. The main method of discouraging adhesion is to use Microsoap on parts of the machine to which Parylene should not make an intimal bond. Another method of adhesion protection is to mask areas not to be coated with Mylar tape that is green in color, comes on rolls resembling masking tape, and is supplied in varying widths.

NOTE: The refrigeration system must be powered on 45 minutes prior to any coating process to allow the cold probe to reach minimum temperature. Failure to do this may result in damage to the vacuum pump if vaporized dimer is allowed to deposit in the pump.

Just after the end of the process cycle and after the refrigeration until has been switched off (approximately 5 minutes), the cold probe must be removed from the cold tarp so that the ice forming on the probe does not have an opportunity to melt and allow liquid to enter the vacuum system. However, the cold probe and the hose to which it is attached must be handled with EXTREME caution so as not to damage the hose to which is still very cold and brittle. About 5 minutes of rest will allow the hose to have begun to warm up enough so that it can be better handled, but not enough time for the ice on the probe to begin melting. It must be moved VERY slowly maintaining as large a bend radius as possible and placed into the clamping probe holder next to the cold trap. There it may rest until the refrigeration unit has completely warmed to room temperature, where upon it may be cleaned.
Before beginning a process cycle, always make sure the chamber and cold probe are clean and free from earlier coatings of Parylene. Then make sure these cleaned parts are completely coated with Microsoap and allowed to air dry before continuing. The cold-probe chamber walls should also be sprayed although these walls are not easily cleaned of earlier deposits due to limited access.

Adhesion promotion is achieved by using another product, ethacryloxypropyltrimethoxysilane, or also called A-174 Silane. This product can be directly applied to selected areas prior to a process cycle, then air-dried, or can be vapor deposited on all samples by placing a small amount into some small receptacle inside the chamber until evaporated completely before the prepared deposition cycle is started.

Samples must be as dust and moisture-free as possible before placing them in the vacuum chamber. Any object that remains on the surface of the substrate, such as dust and fingerprints will be coated and remain on the sample beneath the conformal Parylene coating. Any moisture in the chamber at the time of deposition may result in the coating appearing to be cloudy or opaque. This is why all cleaned parts should be allowed to air dry before continuing with a deposition.

Parylene is resistant to most chemicals and solvents. Two solvents, chloronaphthelene and benzyl benzoate at temperatures above 150°C will solvate Parylene C putting the polymer into solution. Swelling is noted with organic solvents such as acetone (ketone) or alcohol (isopropyl) however, after the solvent was removed and dried the Parylene film returned to its original state and size. Inorganic reagents such as nitric and chromic acids (both oxidizing acids), hydrochloric, and sulphuric (both non-oxidizing acids) acids, and sodium and ammonium hydroxides caused some small percentages of swelling in the deposited Parylene. In these cases, no attempt was made to check the reversibility in removing and drying the samples although it was noted that for short exposure times of less than 60 minutes to any of these acids, bases, or solvents (with the note4 exception of chloro and di-chlorobenzenes), the adhesion of the coating was not compromised.

The thickness of deposition is a function of exposed surface area and initial mass of Parylene dimer. The rate of deposition for Parylene C is about **0.0002 in/hr or 5.0 microns/hr**. Parylene N requires twice the chamber pressure of Parylene C and deposits at a lower rate of **0.00003 in/hr or 0.75 microns/hr**. These Parylene types are biocompatible and are at present the only types of Parylene in use at U of L, however other types of Parylene are available, each with unique mechanical, chemical, electrical and thermal properties.

**Steps for Operation**

1. Release the EMO button by turning while pulling outward.

2. Make sure that the vaporizer and furnace switches are turned to DISABLE. Press and release the white POWER button on the front panel.

3. Choose the appropriate vacuum chamber for the size of substrate or deposition scheme. If the tumbling option is desired, it is necessary to install the tumbling apparatus in the large chamber by removing the port window.
4. Thoroughly clean the inside of the chosen vacuum chamber. This includes all internal chamber parts, i.e., the inside chamber wall, the inside of the port window, the rotating aluminum horizontal disk, the stainless steel bottom of the chamber, the incoming vapor port (as much as possible), chamber gage port, and the outgoing vapor port (as much as possible). Loose pieces of previously deposited film may dislodge and reduce the effectiveness of the deposition and/or the vacuum pump. Therefore removing, as much as possible, any previous deposition reduces potential problems. Use a Scotch-Brite type abrasive pad to facilitate the removal of deposits. If a very thin previous deposition exists, less than 1-2 microns, it is suggested to allow a forthcoming deposit to adhere to it before removal since very thin deposits tear easily and are more difficult to remove. It is still necessary to remove a very thin deposit from the cold trap probe because any deposition on the probe will cause a decrease in refrigeration efficiency.

5. Use a Scotch-Brite-type abrasive pad to completely remove Parylene deposits on the metal cold-trap probe, spray with a 2% solution of MicroSoap Micro 90 and DI water, and wipe or allow to air dry.

6. Place the cold trap probe into the cold trap well at the top of the right rear of the machine and TURN ON the refrigeration unit by flipping the green power switch to ON position located on the front panel of the refrigeration unit. This refrigeration unit should be turned on 45 minutes prior to beginning a deposition cycle to allow it to reach the proper temperature before deposition begins.

7. Check the cleanliness of the aluminum dimer boat for loose, dark brown or black particles. These boats may be reused if the boat is clean and free of loose, particulate material. If excessive dark brown or black particles are present in the bottom of the boat, a new one may be formed using clean aluminum foil. The size of this boat should be close in size to the
8. Load the boat with the type and amount of dimer to be used by placing the dimer boat on a scale, zeroing the weight of the boat, and then adding the appropriate amount of dimer. The maximum amount of dimer must not exceed 100 grams. (Dimer weight + boat are typically less than 10 grams.)

9. Place the boat with the dimer in the vaporizer furnace taking care not to place the boat too far forward in the furnace tube. It is only necessary to push the boat in until the door can be closed and latched. (This prevents pre-evaporation of the dimer into the vaporizer furnace before it can be taken into the pyrolysis furnace.)

10. Treat all surfaces inside the chosen vacuum chamber with MicroSoap Micro 90 to demote adhesion of the Parylene polymer to the parts of the chamber that will require cleaning after the deposition cycle. Dry those surfaces after treatment with lint-free tissues or allow to air dry before assembling the chamber and assorted parts to the machine. DO NOT APPLY Micro 90 to any of the subject materials to be coated with Parylene. (An adhesion promoter (gammamethacryloxypropyltrimethoxysilane or also known as SILANE (liquid)) may be directly applied or evaporated onto objects to which Parylene does not adhere well, including those with small values of surface roughness.)

11. If the large chamber is to be used, other internal parts may also be needed. The ported baffle, which looks somewhat as a crude flute constructed from an approximate 15" of electrical conduit pipe with evenly spaced holes, should be used to aid in the distribution of the Parylene vapor. The holes should directly face the inside wall of the chamber. Stacked trays, made of round, perforated aluminum may also be used to support subject materials. All subject materials should be anchored in some manner, usually with green polyester/silicone tape (3M #8403) to prevent displacement of items under high vacuum/venting conditions. In some cases, careful masking of areas of subject materials where Parylene deposition is not desired may be done using the same tape as above. It may also be necessary to perform a second coating cycle to completely encapsulate a subject where the subject's taped anchor site for the initial coating cycle prevented Parylene from being deposited. (It is not possible to use the distribution "flute" or the stacked trays in the small chamber.)

12. If the tumbler option is to be used, consult the Operator's Manual for instructions on installing the tumbler option on the large chamber. The large chamber port window must be removed.

13. Place the chamber to be used over the prepared chamber area and subject materials. The small chamber requires no other pre-cycle preparation. If the large chamber is used, make certain that no contact is made between the distribution flute and the rotating aluminum plate or stacked trays (if used.) It is recommended that the large chamber be shifted as far to the left
side of the machine as possible away from the holes in the flute. It is also recommended that a small piece of tape be used to secure the flute to the chamber wall such that it stands perpendicular to the horizontal floor of the chamber and parallel to the vertical wall of the chamber. Place the two-handled, metal lid on the large chamber.

14. Turn the vacuum main panel switch from VENT to VACUUM. This should begin the pump-down of the chamber. Watch the gage values to make certain the numbers are decreasing (it will increase for a few seconds, then, should begin to decrease.) If it does not, listen for sounds of a leak. It may be necessary to apply a small force to the top of the chamber downward to help create the vacuum seal. It is also helpful to apply a thin layer of silicone-based vacuum grease to the rubber gasket on the bottom of the chamber cylinder.

15. Once the vacuum gage values have begun to decrease, you may ENABLE both the furnace and vaporizer switches on the front panel. This should begin the rotation of the lower aluminum plate or the tumbler option, if used. Observe a complete rotation through the port window to make certain that no contact with the flute or inner chamber wall is taking place with the rotating plate or deposition subjects. If no port windows are available e.g., using the tumbler option, you may ENABLE the furnace and vaporizer switches before applying vacuum and placing the lid on the chamber to check the rotation of the internal components for a complete cycle.

16. Once the rotation has been checked, the PROCESS START/STOP button on the front panel may be depressed to begin the cycle. This button will now glow a solid green color. If at any time the cycle needs to be halted, you may use the red EMO button or the PROCESS START/STOP button depending on the required level of safety.

17. The process is automatic from this point forward until the cycle is complete. The vacuum continues to reduce to a base level (typically, less than 8 on the vacuum gage), while the Furnace and Chamber Gage values ascend toward their respective temperature set points. When these are reached, as much as an hour later, the Vaporizer begins to rise to its temperature set point. Then, automatically, the Parylene is released increasing the chamber pressure from its base pressure to a higher value indicating that Parylene coating has begun. When the Parylene supply begins to diminish, pressure in the chamber will once again reduce towards a lower base pressure. At this time, the green PROCESS START/STOP button will begin to flash signaling the end of the coating cycle.

18. At the end of the cycle, depress the PROCESS START/STOP button (This is important step that prevents machine does not go into Alarm on the next startup). Shut OFF the
19. The cold-trap probe flex-line is very cold at this point. Allowing the unit to sit for about 5 minutes before moving the cold-trap probe gives the flex-line time to reach an ambient temperature on the perimeter of the hose so that it reduces stress in the hose. It requires EXTREME care when it is handled cold. Do not create a tight radius of curvature in this hose at any time. The refrigeration unit is placed a proper distance away to keep a large curvature radius in the hose. Grab the cold-trap probe with one hand and the middle of the flex-line with the other hand. GENTLY AND VERY SLOWLY, remove the cold-trap probe from the trap moving both hands to reduce stress on the hose as much as possible, then place the probe in the probe holder a few inches away at the far right-hand corner. A security clamp may be engaged with the cold-trap probe to keep it from moving in the holder. Most repair problems arising with the use of this machine are associated with the mishandling of the frozen cold-trap flex-line. The reason for not leaving it in the cold-trap until it completely warms up is that frozen condensation on the probe may enter into the vacuum system causing extensive damage to the pump, and at least, contamination of the vacuum system oil.

20. The final step is cleaning interior of the chamber and associated parts. It is not recommended that the cold trap probe be cleaned while it is still cold, however it must be cleaned prior to subsequent depositions. It is also not recommended that very thin deposits (less than 1 or 2 microns) of Parylene be removed as it is easily torn and will not easily remove. Subsequent deposit on thin layers will facilitate the removal of the thin layer as long as MicroSoap Micro 90 was used to demote the adhesion between the bare chamber wall and the first thin layer of Parylene. The vacuum pump oil life is increased if the vacuum system is left in a state of slight vacuum pressure. This is accomplished by placing a solid object over the cold-trap well and replacing a clean vacuum chamber (large or small) on the machine and starting the vacuum pump for a few seconds. When the vacuum gage reads about 900, the system is sealed and under vacuum. Set the vacuum switch to HOLD at this point for storage of the system.
Useful Parameters For Parylene Deposition

**Deposition rates:**
- Parylene N ~ 0.0003 inches/hour (0.762 um/hour)
- Parylene C ~ 0.002 inches/hour (5.08 um/hour)

**Maximum deposition thickness before cleaning chamber walls:** 0.001 inches (25.4 um)
(Clean yellowish deposit in pyrolysis heater after 400g of Parylene used)

**Typical Process Settings**

<table>
<thead>
<tr>
<th>Parylene</th>
<th>Vapor Heater SP</th>
<th>Pressure SP</th>
<th>Pyrolysis Heater SP</th>
</tr>
</thead>
<tbody>
<tr>
<td>Type N</td>
<td>160°C</td>
<td>Base +55 vacuum units</td>
<td>650°C</td>
</tr>
<tr>
<td>Type C</td>
<td>175°C</td>
<td>Base +15 vacuum units</td>
<td>690°C</td>
</tr>
</tbody>
</table>

**Original System Default Set points (for Type C)**

<table>
<thead>
<tr>
<th>Furnace</th>
<th>Chamber Gauge</th>
<th>Vaporizer</th>
<th>Vacuum</th>
</tr>
</thead>
<tbody>
<tr>
<td>690°C</td>
<td>135°C</td>
<td>175°C</td>
<td>25</td>
</tr>
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**The vacuum pressure controller** provides a displayed value that very nearly represents absolute pressure (in mTorr) for the process range of 10 to 100 units. After that point in becomes non-linear (i.e. display of 500 units is approximately 2.2 Torr)

For processing it has a factory set point of 15 units above base pressure. Increasing or decreasing this value will increase or decrease deposition rates, but too high of a deposition rate can lead to poor quality films.

**Typical foil size for boat form:** 11 x 5 inches (formed boat must be < 7.5 inches long)

**Vaporizer:**
- Temperature above which coating initiates: 90°C
- Temperature below which more Parylene can be added to boat: 60°C

**Surface area of chamber, baffle, fixture, and plate:** ~900 in²

**Minimum spacing between product in chamber:** 0.5 inches

**Machine considerations:**
- Chiller must run in the cold trap for 4S min before initiating the vaporizer heaters
- After turning the chiller off wait at least 5 minutes before turning it back on.