Specialty Coating Systems PDS2010 Parylene Coater

Standard Operating Procedure

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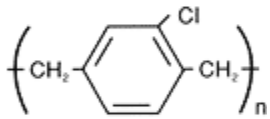
Please refer to the "Parylene Conformal Coating and Specifications" from Specialty Coating Systems for a complete description of the poly-para-xylene polymer family, the deposition process, and material properties. The document is posted in the microfab website. The following is an exerpt from the document

Parylene is the generic name for members of a unique polymer series. The basic member of the series, called Parylene N, is poly-para-xylylene, a completely linear, highly crystalline material.

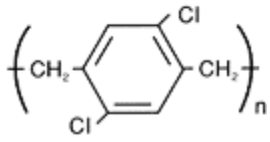
Parylene N

Parylene C, the second commercially available member of the series, is produced from the same monomer modified only by the substitution of a chlorine atom for one of the aromatic hydrogens. The structures are shown in Figure 1. Parylene N,C & D Chemical Structures (right).



Parylene C

Parylene D, the third member of the series, is produced from the same monomer modified by the substitution of the chlorine atom for two of the aromatic hydrogens. Parylene D is similar in properties to Parylene C with the added ability to withstand higher use temperatures. Parylene N is a primary dielectric, exhibiting a very low dissipation factor, high dielectric strength, and a dielectric constant invariant with frequency. This form has the highest penetrating power of all the Parylenes. Parylene C has a useful combination of electrical and physical properties plus a very low permeability to moisture and other corrosive gases. Along with its ability to provide a true pinhole free conformal insulation, Parylene C is the material of choice for coating critical electronic assemblies.



Parylene D

Due to the uniqueness of the vapor phase deposition, the Parylene polymers can be formed as structurally continuous films from as thin as a fraction of a micrometer to as thick as several mils.

Currently, only Parylene C is available in the Tufts PDS 2010.

Warnings:

The tool uses high temperatures and low vacuum during deposition. Do not attempt to open the chamber until the deposition cycle is complete.

Attempt to minimize generation of airborne particles when pouring the Parylene dimer. Under no circumstances breathe the Parylene monomer (which will be present inside the tool during operation… the only way this could happen is if there was a power failure during the deposition cycle).

The Parylene-C dimer (the solid form) and the microsoap are not harmful.

The A-174 silane adhesion promoter is a moderate skin, respiratory, and eye irritant. It is combustible, but not overly flammable.

Isopropanol is a highly flammable organic solvent.

Required Checkout: You must be formally tested by the faculty supervisor before you may use this tool unsupervised. When you are first learning procedures, work with experienced lab users (students, post docs, faculty or staff) to become familiar with the tool before requesting a formal checkout procedure.

1.0 Material Requirements:

1.1 Equipment: substrate, wafer tweezers, Microsoap spray bottle, baffle, aluminum foil boat
1.2 Personal Protective Equipment: nitrile gloves, safety glasses
1.3 Chemicals: Micro90 Microsoap, Parylene C dimer, Isopropanol, A-174 silane adhesion promoter (>98% Gamma-Methacryloxypropltrimethoxysilane)

2.0 Procedure:

Adhesion Promotion (not required, possibly not desirable)
If you are using the parylene layer as a flexible foundation – in other words, you intend to fabricate your devices on the parylene and, as the final step, peel the parylene away from the substrate – in that case the adhesion promoter is *not* recommended. The parylene will adhere too well and peeling it away in a single sheet will not be possible. On the other hand, if improved adhesion is desired, the following procedure was adapted from the Berkeley Microlab Laboratory Manual.

1. In a 1000 mL glass beaker, mix 400 mL of isopropanol, 400 mL of DI water, and 4 mL of A-174 adhesion promoter.
2. If the mixture is to be used immediately, stir continually for 5 min every 10 min for a period of 2 to 2.5 hours. Or else, leave the mixture overnight.
3. Remove wafer or substrate from mixture and allow to air dry for 30 mins.
4. Soak wafer in IPA for 5 min.
5. Dry wafer with air gun.
6. Bake in oven at 115ºC for 30 min.
7. Dispose of the waste in mixed organic solvents waste (HDPE bottle).
8. Rinse the beakers with DI water once and dispose of this rinse water in the organic solvents waste (HDPE bottle).

Alternatively, you may spin HDMS (photoresist adhesion promoter) onto your substrate but we have not tried this and cannot comment on the resulting level of adhesion.

Prepare Chamber and System:
Parylene deposition is conformal, in other words, it will deposit on all surfaces including those where deposition is undesirable. If left unchecked, parylene will eventually clog the system and render it unusable. Thus, it is critical to slow down the build-up and remove the parylene from these areas.

1. Prepare the system by coating everything you want to stay clean with "microsoap" – there is a spray bottle on the shelves next to the coater - which will reduce Parylene adhesion and allow you to clean the chamber afterwards. This includes the chamber walls, the baseplate and any fixture that will be in the chamber. The spray bottle should be a mixture of 2% microsoap to 98% DI water by volume. Spray it onto a lab wipe and wipe the target surfaces. **Avoid spraying Microsoap on other surfaces**, if you do happen to get it on other surfaces or objects use a fab wipe wetted with DI water to wipe away.
2. Place the baffle flute in the chamber. Put tape on baffle flute, below the threaded stud for a snug fit. Also place tape over the top of the flute. Make sure holes are facing the chamber wall, not the parts being coated.
3. Load your specimens. Often it is desirable to load a glass slide(s) to measure the parylene thickness post-run.
4. Check all O-rings, gaskets, and their matching surfaces for cleanliness.
5. Put the chamber lid into position – it is heavy, be careful – it should sit on the base plate precisely.

Load Dimer:
Here you are loading in the Parylene precursor… the dimer form of Parylene, small white particles.

1. Make a dimer boat out of aluminum foil. To do this, cut an 11x5 in. rectangular piece of aluminum foil, and form the sheet along the outside diameter of the boat form provided. Fold in the ends of the foil so that the dimer will not spill out.
2. Weigh out the desired amount of dimer on the digital scale. This will determine your final Parylene thickness. The maximum dimer load cannot exceed 125g, though actual needed loads will probably utilize far less than this amount. Check the log to determine a dimer-weight to layer-thickness conversion. Note that this conversion will change over time. Deposition rates are on the order of 5 microns per hour.
3. Load the dimer (in the aluminum foil boat) into the vaporizer chamber load door, located in the lower front compartment of the parylene coater. The dimer boat may be reused until a moderately thick amount of dark residue has formed along the inside of the boat.
4. Close and lock the vaporizer door.

Setting up the Deposition:

1. If needed, release the emergency stop by turning it clockwise; if it was engaged it will pop out.
2. Push the Main Power button on the left side of the control panel, all gauges should illuminate.
3. Check all set point values, and if all are correct, enable the furnace/chamber gauge and vaporizer switches. The setpoints should be as follows: 690ºC furnace, 135ºC chamber, 175ºC vaporizer.
4. Put the chiller wand in place in the well on the back right of the tool.
5. Turn on the mechanical chiller using the switch on the front of the chiller (the chiller is the rectangular box to the left of the main PDS2010 unit). It will take 5-10 minutes for the chiller to cool to the appropriate temperature, continue with steps 6 & 7 while waiting

6. Turn the "Vacuum" switch to "vacuum," and the pump will power up. The chiller wand should stick in place (you may need to hold it down for a few seconds) as the pressure begins decreasing.
7. Enable both heaters by turning the furnace/chamber gauge and vaporizer switches to "enable".
8. Once the chiller has achieved the appropriate temperature the green "cool light" will come on, it is now OK to proceed with the next steps. The chiller is an essential part of the system, without it some parylene will find its way to the vacuum pump and deposit there, destroying it.
9. With the chiller green OK light on, push the Start/Stop button on the tool, this will initiate all the heating systems. The process is underway. You may leave the lab during the run.
10. Once the controller values are reached (see step 3) the deposition will run automatically.
11. For Parylene-C, it will take approximately 1.5 hours for every 10 grams of dimer charge loaded. Once complete, the green Start/Stop button on the control panel will begin flashing.

Remove Samples and Power Down:

1. **When the dimer is all gone, the Start/Stop button on the control panel will start flashing.**
2. It is advisable to let the system cool for ~1 hour before continuing
3. Turn off the chiller. Set the switches – Chamber and Furnace, Vaporizer and Vacuum to disable. You will hear the vacuum chamber venting.
4. Gently bring the chiller wand out of the housing and hang it on the bracket so condensation does not get into the instrument. Put some fab wipes on the floor under the chiller wand to catch any water. Move the wand gently, the pipe is very cold and can be damaged by excessive flexing.
5. Press the green main push button to turn off the blinking green light. Press the emergency stop button to power down. Twist the button clockwise to return to its proper position
6. Lift off the chamber cover and place it down carefully. Remove your samples.
7. If you loaded any glass slides to measure parylene thickness, with a razor blade carefully slice across the parylene and remove a portion. You may now measure the height of the parylene in the DekTak profilometer.
8. In the log, note the amount of dimer used and the resulting layer thickness.

Clean up:

Parylene removal and clean up after a deposition is essential to the operation of the coater, if you are not willing to clean up properly, please don't use the instrument. Make use of the vacuum cleaner and tools found next to the coater to remove parylene from the various surfaces.

1. Using the scraper, remove the white residue from the chiller wand. I recommend scraping directly into a non-hazardous trash container.
2. It may be possible to peel the parylene from the chamber in one sheet. Clean out the inlet flute with the wire brush to remove residual Parylene ash from the inside. You will notice that the parylene may have some static charge and be very "sticky" – take care to not spread this throughout the lab, it can contaminate other processes. All parylene can be thrown away in the general trash bin, it is not hazardous.
3. Peel/scrape off excess Parylene film from other surfaces. Use microsoap and cleanroom wipes to assist in cleaning as needed.
4. Try not to scratch the surfaces of the chamber walls or the chiller wand.

Storage

1. A-174 adhesion promoter is stored in the solvent cabinet
2. Micro90 Soap and Micro90 Soap solution are stored next to the coater.
3. Parylene C dimer is stored in the dessicator next to the coater.

3.0 <u>Waste Disposal:</u>

3.1 Parylene C Dimer and Parylene C Polymer

3.1.1 Solid waste can be disposed of in the general trash. This is not hazardous waste.

3.2 Micro90 Soap

3.2.1 Solid wipes contaminated with Micro90 soap can be disposed of in the general waste trash can (non-hazardous).

3.2.2 There should be no reason to generate liquid Micro90 Soap liquid waste, but if such waste is generated, it should go in an HDPE bottle in the hazardous waste accumulation area. This bottle should be labeled "Micro90 Soap" and should not be mixed with other chemicals. The Micro90 soap is not considered a hazardous waste by federal regulations.

3.3 A-174 Adhesion Promoter

3.3.1 Solid waste contaminated with A174 adhesion promoter should go in the solvent trash bin.

3.3.2 Liquid A174 waste should go in the mixed solvents waste bottle in the hazardous waste accumulation area.

4.0 <u>Accident Procedures:</u>

4.1 Contact: Read MSDS prior to working with any chemical to familiarize yourself with the symptoms of exposure and recommendations for treatment.

4.1.1 A174 Silane Adhesion Promoter

4.1.1.1 Skin contact: If this material contaminates the skin, begin decontamination with running water. Recommended flushing is for 15 minutes if any sign of skin irritation develops. Victim should seek immediate medical attention if any adverse exposure symptoms develop. **Tufts Emergency Medical Services are at x66911.**

4.1.1.2 Eye contact: If this product enters the eyes, open victim's eyes while under gently running water. Use sufficient force to open eyelids. Have victim "roll" eyes. <u>Minimum</u> flushing is for 15 minutes. Do not interrupt flushing. Victim must seek medical attention. **Call Tufts Emergency Medical Services are at x66911.**

4.1.1.3 Ingestion: If this material is swallowed, CALL PHYSICIAN OR POISON CONTROL CENTER FOR MOST CURRENT INFORMATION IMMEDIATELY. If fully conscious, give victim two glasses of water. DO NOT INDUCE VOMITING unless directed by medical personnel. Never induce vomiting or give diluents (milk or water) to someone who is <u>unconscious, having convulsions, or unable to swallow</u>. If vomiting occurs naturally, have victim lean forward to reduce risk of aspiration. If medical advice is delayed or if the amount swallowed is significant (a few ounces), give victim three to four ounces of alcohol, such as whiskey (proportionally smaller amount if victim is a child). **Get immediate medical attention. Call Tufts Emergency Medical Services are at x66911.**

4.1.1.4 Inhalation: Remove to fresh air. Resuscitate if necessary. Take care not to inhale any fumes released from the victim's lungs. **Get immediate medical attention. Call Tufts Emergency Medical Services are at x66911.**

4.1.2 Isopropanol

4.1.2.1 Skin contact: Rinse affected area with water for 15 minutes, removing contaminated clothing during the rinse. If burning and irritation persist, contact Tufts health services.

4.1.2.2 Eye contact: Immediately flush with water for 20 minutes while holding the lids open. Get immediate medical attention. Call Tufts Emergency Medical Services are at x66911.

4.1.2.3 Ingestion: Do not induce vomiting. Get immediate medical attention. Call Tufts Emergency Medical Services are at x66911.

4.1.2.4 Inhalation: Remove to fresh air. Get immediate medical attention. Call Tufts Emergency Medical Services are at x66911.

4.1.3 Micro90 Microsoap Solution

4.1.3.1 Eye contact: Immediately flush eyes with plenty of water. Get medical attention if irritation develops or persists.

4.1.3.2 Skin contact: Remove contaminated clothing. Wash skin with soap and water. Get medical attention if irritation develops or persists.

4.1.3.3 Inhalation: If exposed to excessive levels of fumes, remove to fresh air. Get medical attention if cough or other symptoms develop.

4.1.3.4 Ingestion: Swallowing less than an ounce will not cause significant harm. For larger amounts, do not inducevomiting, but give one or two glasses of water to drink and get medical attention.

4.1.4 Parylene Dimer or Parylene Polymer

4.1.4.1 Skin contact: This is a mild irritant only. Wash with soap and water. If irritation persists, contact Tufts Medical.

4.1.4.2 Eye contact: Rinse eyes with running water for 15 minutes. Contact Tufts Medical.

4.1.4.3 Ingestion: Call Tufts Medical. Do not induce vomiting. Have victim rinse mouth with water.

4.1.4.4 Inhalation: Remove to fresh air. If coughing, sneezing, headache, nausea or other symptoms persist, contact Tufts Medical.

4.2 Spill:

4.2.1 If a small, contained spill of **A174** and/or **isopropanol** occurs, such as inside the hood, wipe it up with fab wipes and dispose of in the solvent trash container.

4.2.2 If a large spill of **A174 and/or isopropanol** occurs that you are not comfortable cleaning up, evacuate the lab and notify the Tufts emergency services (x66911) immediately. Clean up should only be performed by authorized personnel according to MSDS guidelines. Notify the faculty advisor.

4.2.3 If a spill of **Micro90 soap solution** occurs, wipe it up with fab wipes and dispose of in the general trash (this is a non-hazardous waste).

4.2.4 If a spill of **Parylene Dimer** occurs, pick it up with fab wipes and dispose of in the general trash (non-hazardous waste).

4.3 Fire:

4.3.1 If a fire starts, use the fire extinguisher to put it out. Evacuate the lab and contact the faculty supervisor.

4.3.2 If you are not able to quickly extinguish the fire using the fire extinguisher, immediately evacuate the lab and call **Tufts Emergency Services at x66911.**

If at any time you feel unsure about how to use the tool, please stop work and contact a qualified user or faculty advisor. Please don't forge ahead when you are unsure, you may end up damaging the tool.

Report all accidents or tool issues to James Vlahakis at james.vlahakis@tufts.edu