

Protocol: Parylene C Deposition with the SCS PDS 2010

Description: This document details the ASIC integration protocol for Parylene microelectrode arrays (MEAs).

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1 SETUP

1.1 RECORD KEEPING

Equipment: Internet-enabled PC

1. Fill out online log sheet (BioMEMS Google Drive > Master Cleanroom Logbook > Parylene Thickness tab).
 - a. If yours is the 5th run since the last oil change, please change the oil in the pump. Notify the superuser if you need assistance.
 - b. If your run will cause the number of grams since the previous baffle change (250 g) or pressure gauge change (1134 g) to exceed the limit, notify the superuser.
2. Determine from log approximately how much Parylene C should be used to obtain desired thickness and measure this out in lab.

1.2 PREPARATION

Materials: Parylene dimer

2% Micro-90 cleaning solution

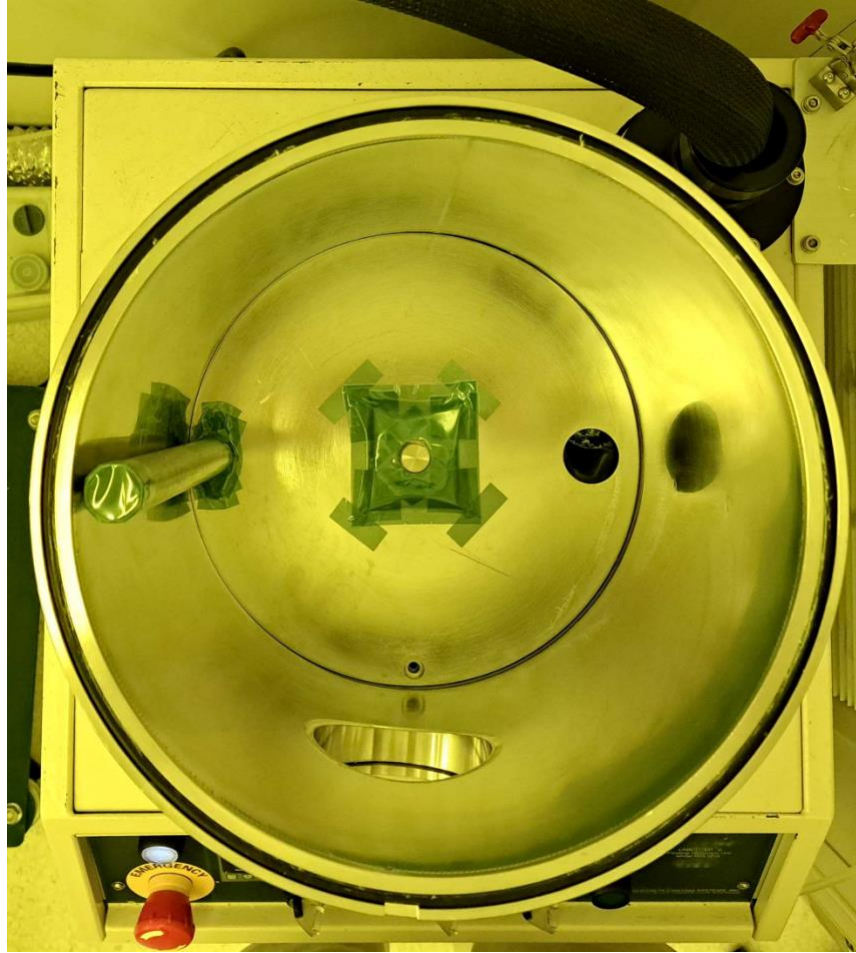
1" x 3" glass slides

Aluminum foil

Note: double-side polished wafers are recommended to reduce bubbles/weak adhesion of Parylene to the backside of the wafer

Equipment: SCS PDS 2010

1. Weigh out Parylene dimer using the scale in the cleanroom.
2. Make sure system was cleaned since last use. Report anything that was not properly cleaned to the superuser.
3. Check cold-trap, vaporizer door, and chamber lid O-rings to make sure each is clean and will form a good seal. If necessary, clean chamber lid O-ring by peeling off with tape. If you replace any O-rings, check for additional spares and order if necessary (see end of protocol for vendors). Also, notify superuser.
4. Do NOT remove chamber base L-gasket unless you are having leak issues or you see Parylene pieces sticking out. Frequent removal and handling make it crack sooner. Overtightening the c-clamp also makes it crack sooner.
5. Wipe all interior surfaces of machine (chamber, lid, rotating table, bottom plate, baffle, etc.) and the cold trap probe with Micro-90, even if it was wiped down after the last run. Do not wipe the shelf, as this leaves TexWipe fibers that get coated over time and cause an irregular surface.
6. Place the chiller cold trap probe into the coating system's cold trap.
7. Place the rotating shelf onto the base.
8. Cut 6 rectangular pieces of aluminum foil 8.5x4.5 in², and place a piece in every shelf (alternating directions) to avoid holes in the back of the wafers.
9. Place your wafers onto the foil lined shelves. Two 100 mm diameter wafers can fit on each shelf.
10. Place a 1x3" clean, labeled glass slide on each shelf for thickness measurements.
11. Make sure none of the samples extend beyond the edge of the rotating table, as overhanging samples may hit the baffle when the table rotates.
12. Carefully lower chamber around shelf. The chamber base should have a slight gap on the right side, so that the baffle flute holes are as close to the left side as possible. If using the shelf assembly, note that thickness can vary several microns between the different shelves.



13. Place chamber lid on top of chamber.
14. Use the metal cylinder and a 4"x10" piece of aluminum foil to mold an aluminum foil boat for the Parylene C dimer. The dimer boat must be no more than 7.5 inches long.
15. Pour Parylene C dimer pellets into the foil boat and load boat into vaporizer. Important: When loading boat, place it only as far into the vaporizer as necessary, do not push towards the back. Close and lock the vaporizer door.



2 OPERATION

2.1 RUNNING THE EQUIPMENT

Equipment: SCS PDS 2010

Timer

1. Twist "Emergency Stop" button until it pops up.
2. Push "Main Power" button. Wait for all the controllers on the display panel to come on and display current values.
3. Turn Vacuum knob to "Vacuum." Start timer now.
4. Initial Vacuum reading should be around 1000. It should reach ~100 in a couple of minutes. If not, your sample is off-gassing or there is a leak in the system.
5. Turn ON the chiller. Wait a minimum of 45 minutes before starting the deposition process.
6. Check that the vacuum level has reached at a level of 5 units (or lower). This may take up to 1 hour total if there are many items degassing. If the pump down takes much longer there may be a leak.
7. Common leaks are caused by cracks or Parylene residue along the chamber O-ring or gasket, leaks along the O-ring within the chamber viewing window, leaks where the pressure sensor attaches to the tool (if replaced recently).
8. "Enable" the furnace/chamber gauge and vaporizer switches.
9. Press the green button labeled "Process Start/Stop".
10. In order for coating to begin, **all** the following must happen: 1) the chamber gauge must reach 135 °C, 2) the furnace must reach 690 °C, and 3) the vacuum reading must be less than 11. Then the vaporizer will begin heating, and the coating process will start. If any of the conditions are not met, something is wrong, and you should contact the superuser.
11. The machine will run for several hours, depending on the coating thickness. The run will stop automatically and begin cool down when process is complete (green button will be blinking).

3 CLEANUP

3.1 COOLDOWN

Materials: 2% Micro-90 cleaning solution

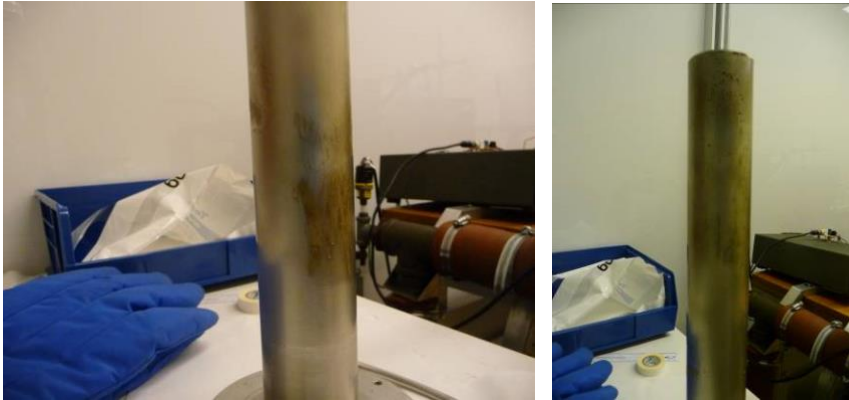
Scotch brite pad

Equipment: SCS PDS 2010

Allen wrenches

1. Let the system cool until the Vaporizer temperature is less than 90 °C to prevent harmful gases from escaping. Stop the timer.
2. Turn off the chiller and wait >5 minutes.
3. Press the green "Process Start/Stop" button.
4. Turn the left two switches to "Disable."
5. Turn the vacuum switch to "Vent."
6. Push "Emergency Stop" button down.
7. Carefully remove cold trap probe from machine and place in holder to defrost.

8. Remove chamber lid.
9. Remove samples.
10. Peel Parylene C from the inside surfaces: chamber, lid, rotating table, bottom plate.
11. *if Parylene film is thin (< 3 microns, rainbow sheen) leave it until clean up after the next run.
12. Wipe inside surfaces with Micro-90 (except the shelf).
13. If necessary, clean chamber lid O-ring by peeling off with tape.
14. Open vaporizer and discard used foil boat.
15. **PERFORM THIS STEP IN THE MAIN LAB:** Once the probe is defrosted, use a green scotch brite pad to scrape off Parylene C residue from the cold trap probe (outside and bottom), wipe clean with a TexWipe and Micro-90. If it doesn't shine all over, you haven't cleaned enough yet. Any cloudiness or residue will lead to rust spots, which is good for neither the probe nor the pump.

<p>Bad - you will scrub the probe again and the Parylene room floor to make up for this!</p>	
<p>Good</p>	

16. Put probe back into holder (NOT cold trap) for storage in between runs. If you incorrectly leave the probe in the cold trap, condensation build up can drip to the bottom and leave rust spots that are impossible to clean and bad for the machine.
17. Use plastic razor blades to clean any vaporizer buildup. Avoid using metal if possible to prevent unnecessary scratching.
18. Update online logbook with samples, dimer used, and Parylene thickness for future reference.

3.2 THICKNESS MEASUREMENT

Materials: Parylene-coated glass slides
Razor

Equipment: Dektak profilometer

1. Use a new razorblade to cut a 1-2mm wide strip of Parylene off one of the 1x3" glass slides from each shelf.
2. Measure deposition thickness with Dektak profilometer and update log.

APPENDICES

A. MAINTENANCE

Every run:

1. Before:
 - a. Check the vaporizer door for any deposits or gunk, clean if there is any amount present. Wipe the cold trap probe with Micro-clean.
 - b. Wipe inside of the chamber, lid, rotating table, and the bottom plate with Micro-clean (not shelf).
 - c. If necessary, clean chamber lid O-ring (peel off Parylene, use only small amounts of Micro-clean and dry immediately afterwards to prevent damage to rubber).
2. After:
 - a. Check the vaporizer door for any deposits or gunk, clean if there is any amount present.
 - b. Clean the cold trap probe with Micro-clean and the green Scotch-brite pads.
 - c. Remove all Parylene from parts inside the chamber, or note in logbook that it is too thin to remove and will be done by the next user.
 - d. Clean chamber lid O-ring (peel off Parylene, use only small amounts of Micro-clean and dry immediately afterwards to prevent damage to rubber).
 - e. Do NOT remove chamber base L-gasket unless you are having leak issues or you see Parylene pieces sticking out. Frequent removal and handling makes it crack sooner.
 - f. Vacuum out any debris that has fallen into the cold trap.
 - g. Store cold trap probe in holder, not cold trap.
 - h. Log the total grams of dimer used
 - i. Notify superuser if total approaches 250 grams since the last baffle change.
 - ii. Notify superuser if total approaches 1134 grams since the last pressure gauge change.

Every 5 runs:

Material: Pump oil

1. Change the oil, regardless of the total amount of Parylene C deposited.
2. Check all O-rings and the chamber base L-gasket for cracks and ensure that they fit snugly with no gaps. Replace if necessary and notify superuser (may also need to order extra backup parts).
3. Let superuser know when only 2 bottles of pump oil are left.

Every 10 runs or 250 g of Parylene:


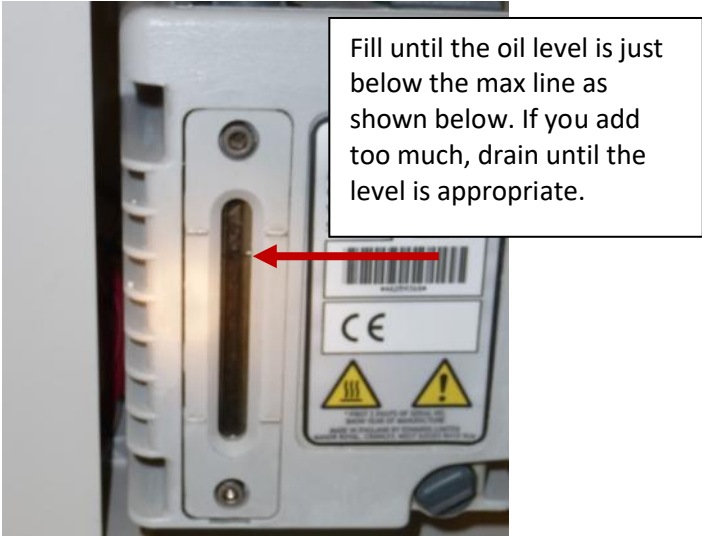
Materials: *Baffle*
 Polyethylene tape


1. Count the total number of grams of dimer used since 10-run inspection.
2. Notify superuser.
3. Check all O-rings and the chamber base L-gasket for cracks and ensure that they fit snugly with no gaps. Replace if necessary and notify superuser.
 - a. Vaporizer load door (brown O-ring)
 - b. Chamber lid (black O-ring)
 - c. Chamber base (black L-gasket)
 - d. Cold trap (red O-ring)
 - e. Pyrolysis tube (brown O-ring, unless you see a large gap, leave it alone). To replace, one must remove entire pyro pipe underneath the metal plate.
4. Remove baffle flute and check for clogs. This flute and the port it sits in can get clogged with burnt Parylene and block the flow of air towards the vacuum, preventing operation of the machine. Get down into the pyro pipe with a wire brush and clear this of any gunk as well. Vacuum thoroughly, preferably through the vaporizer door.
5. Change the oil.
6. Check all tubing leading from chamber to pump for excessive Parylene coating. Replace section(s) of tubing if necessary.

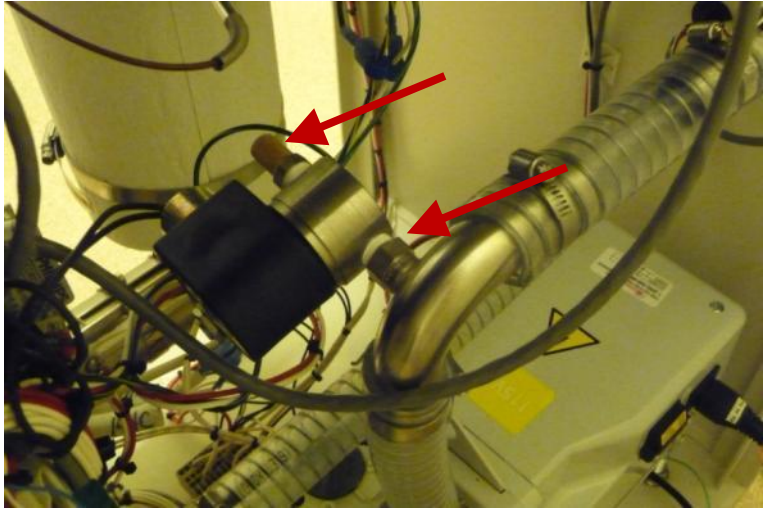
Baffle change:

1. Remove all polyethylene tape from baffle and stage rotator.
2. Use an adjustable wrench to pry the baffle out of its hole (using counterclockwise motion to prevent tape from binding)
3. Using wire brushes clean the pyrolysis tube and vaporizer chamber.
4. Use wipes on brushes to clean fine particulate from surfaces.
5. Vacuum all debris.
 - a. Apply polyethylene tape to the baffle approximately $\frac{1}{4}$ " below the threaded rod
 - b. Wrap the tape in a clockwise direction (when baffle is viewed from above).
6. Wrap the tape 11 times around the baffle.
7. Insert baffle into pyrolysis port with the holes facing away from the center
 - a. If the baffle is too tight, remove one revolution of tape.
 - b. If too loose, apply one revolution.
8. Eliminate gaps between the baffle and port by covering with polyethylene tape. Also cover the threaded rod.
9. Cover the top of the baffle with polyethylene tape.
10. Re-apply polyethylene tape to the stage rotator to eliminate gaps between the white spacer disk and the metal place underneath, but do not apply to the shaft.

B. TROUBLESHOOTING

Vacuum Issues	Action/How to Test
Is your sample outgassing?	Run the chamber empty to verify.
Has the oil been changed recently?	<p>If yes, drain a little oil to verify that it is still clean. If no, change the oil. Drain used oil into a plastic tray and transfer to a plastic waste bottle. Flush with about 1/3 bottle of oil before plugging the drain outlet.</p>  
Is the vaporizer door clean?	If present, scrape away residue and smooth out with a Scotch Brite pad. Then vacuum up AND wipe with TexWipe.
Is the interior of the vaporizer clean?	If present, scrape away black residue with stainless steel brush or metal tool. DO NOT use Scotch Brite pad, as this leaves particles behind. Thoroughly vacuum debris.
Are all o-rings/L-gasket intact?	Replace as necessary.

<p>How many grams have been processed since the last baffle port cleaning?</p>	<p>If more than 10 runs have passed, remove the baffle flute and check for clogs. Replace baffle flute if necessary and scrape out the port with a wire brush. After several years, you may need to chip away at residue built up between the baffle port wall and the outside of the baffle flute. Vacuum thoroughly.</p>  <p>Baffle flutes after several uses (left), and after many uses and clogging (right).</p>  <p>Baffle flute openings after several uses (bottom) and after many uses and clogging (top).</p>
<p>Is the inlet filter to the pump blocked?</p>	<p>Clear out the mesh cone filter at the pump inlet for debris. Open the clamp pictured below for access.</p> 

Is there a leak after the chamber?	Use a rubber stopper (in the Parylene room) with the spare gauge tube to block the outlet port of the chamber.
Is there a leak after the furnace?	Use the spare sight glass and o-ring to block the port between the furnace and the chamber base.
Is there a leak in the probe?	Remove the probe and place in holder. Use the spare sight glass and o-ring to block the cold trap probe opening.
Is there a leak in the cold trap?	Spray acetone around the base of the cold trap and watch pressure reading.
Is there a leak at the gauge tube port?	Spray a small amount of acetone at the joint and watch pressure reading.
Is there a leak at the solenoid valve?	Spray a small amount of acetone on either the gold mesh filter or the elbow pipe connection and watch pressure reading.
	
Is the oil cap closed?	Make sure the cap is not loose.
Is the vacuum in the correct modes?	Gas ballast should be on "0". Mode (dial facing the vaporizer) should be all the way to the right for High Vacuum (small droplets).
Other Issues	
Fixture not rotating	Check Fuse F7 (for fixture rotation motor) and replace if necessary. It's located above the vaporizer and is accessed through the side panel.
Does the pump slow down in the 100's and up?	You probably have a leak somewhere. Check all connections and chamber seating.
Does the pump slow down below 50 vacuum units?	Something could be outgassing. Check your sample. Check the baffle port for residue buildup between the baffle port wall and the outside of the baffle flute.

C. MATERIAL SOURCES

Note: Standard materials (e.g. acetone, DI water, cleanroom wipes, etc.) are not listed

Material	Supplier
Parylene dimer	Specialty Coating Systems, Indianapolis, IN
Micro-90 cleaning solution	Specialty Coating Systems, Indianapolis, IN
Baffle	Specialty Coating Systems, Indianapolis, IN
Glass slides	VWR
Edwards Ultragrade 19 pump oil	VWR
Chamber L-Gasket, 12", Buna-N	Kurt J. Lesker Co., Jefferson Hills, PA (part #LG12B)
Lid O-ring, Buna-N, No. 380	McMaster-Carr (part #9452K436)
3M Polyester tape 8403	R.S. Hughes Co., Inc, Pacoima, CA

D. EQUIPMENT MODELS

Note: Standard equipment (e.g. tweezers, microscopes, N₂ gun, scale, etc.) are not listed

Equipment	Model #	Supplier
Parylene PVD	PDS 2010 Labcoter	Specialty Coating Systems, Indianapolis, IN
Profilometer	DektakXT	Bruker, Billerica, MA