For principles of parylene deposition see the following scheme:

Parylene Deposition Process



For more details visit: http://www.scscoatings.com/parylene_knowledge/specifications.cfm Our home made (good for basic research) system with optimized procedure is described below.



The whole system assembled: you have to be able to reach ~ 5×10^{-3} Torr in the clean assembled system after about 1 h pumping and filling the cold trap with liquid nitrogen (w/o the dimer loaded).



Parylene-N deposition procedure:

Prepare your samples. The samples with contacts and wire leads have to be attached to substrates. We use thin gold wires (25 μm-thick) as contact leads and thin glass or plastic slides for the substrates. Make sure that the leads are long enough (about 1 inch), and the substrates are smaller than the inner diameter of the quartz tube used for parylene deposition (*ID* = 19 mm in our system). Using a razor blade, acetone and tissue clean old parylene deposit off the aluminum plate used as a sample holder. Carefully attach the samples to the aluminum plate (small pieces of scotch tape can be used, or you can design something more decent, like screws or clips). Beside each sample, place a small (0.5 - 1 cm²) piece of thick glass or silicon wafer (affixed by a small piece of double-side Scotch tape) for thickness monitoring: you will be looking at reflection of ambient light in these pieces to estimate the thickness of growing parylene film. After the samples are attached to the aluminum plate, inspect them carefully under a microscope to make sure that wires are ok.



- 2. Clean the two silicone stoppers with acetone and tissue, making sure that there are no dirt, fibers, debris or small parylene particles on their surface.
- 3. Wash the quartz tube for parylene deposition (ID = 19 mm) with acetone for three times with both ends sealed with the silicone stoppers cleaned in step 2; wash the tube again with isoproponal for three times. Dry the tube with a heat gun. Although polymerized parylene does not dissolve in acetone or any other solvents, nevertheless cleaning as described will help to get rid of loosely attached small pieces of old parylene deposit and remove other contaminants.
- 4. Load the aluminum plate with the samples into the tube (it should be positioned close to the end that will be connected to the LN₂ trap the right end of the quartz tube in our system). Make sure by visual inspection that there is no particulate or debris on the silicone stopper/QF-25 adapter and on the inner wall of the quartz tube, where the silicone stopper will come in contact with the quartz, otherwise you will not achieve the desired level of vacuum.
- 5. Attach the tube to the pumping system (in our case, it is a two-stage mechanical pump, an in-line oil back-stream filter and a LN₂ trap) using a silicone stopper-QF-25 adapter.



- 6. Clean the ceramic boat used for the parylene dimer powder (commercially available parylene precursor); make sure to mechanically remove old parylene build-up and particles using a spatula or a razor blade. Fill the cleaned boat with enough fresh parylene dimer powder using a clean spatula.
- 7. Carefully load the boat into the opposite end of the tube, using marks on the tube for precise positioning. Try to minimize spill and particles flying due to static electricity.
- 8. Clean up the spilled dimer particles around the loading end of the quartz tube (left end) and seal it with a clean silicone stopper. This step is critical: make sure by visual inspection that there is no particulate on the inner wall of the quartz tube, where the silicone stopper comes in contact with quartz, otherwise you will not achieve desired vacuum.



- 9. Assemble the LN₂ cold trap.
- 10. Start pumping and wait for around 1~1.5 hours. Note: at this moment, the sealed quartz tube is not yet in the main tube furnace (Blue-M). It is sitting in air at room temperature supported by a couple of mounts. The height of the tube above the table is adjusted, so that an opened Blue-M hinged tube furnace can be shifted under the quartz tube.
- 11. Around 20 minutes before reaching 1 hr pumping, turn on the main furnace and set temperature to 700 ^oC (this furnace must have an independent temperature control and monitoring).
- 12. Attach a thermocouple (TC) to the marked position on the outer surface of the tube right above the boat with the dimer powder; the TC should be attached with a piece of heat resistant sticky yellow tape – this is important to prevent the TC from moving or detaching from the surface of the quartz tube which could result in incorrect temperature readings. Put the TC wires through the small tube heater that we'll be using for dimer evaporation. This removable heater will be later put on the left end of the tube, right above the boat with dimer powder.



- 13. Connect the thermocouple to the temperature controller/power supply of the small heater.
- 14. Turn on this power supply and set temperature at around room temperature (~ $20 \, {}^{\circ}C$).
- 15. Add liquid nitrogen to the cold trap and wait for around 10 minutes. The cold trap should be refilled during the following deposition process from time to time.
- 16. When the main furnace reaches 700 °C, open it and carefully shift it toward the quartz tube until the quartz tube becomes right above the open grove of the furnace heater. Carefully place the quartz tube into the furnace grove (be careful: the furnace is hot, and the tube is under vacuum) and after making sure the tube is sitting along the grove nicely close the furnace. Use marks on the quartz tube for precise positioning of the heating zone of the furnace.
- 17. Put on the small tube heater for parylene evaporation on the left end of the quartz tube, so that it covers the TC and the boat area.
- 18. After the main furnace reaches 700 ^oC again, set the temperature of the small heater in the following sequence to avoid overshooting: 60 ^oC -> 80 ^oC -> 100 ^oC -> 110 ^oC -> 120 ^oC. The purpose here is to achieve a steady 120 ^oC in the evaporation zone, without overshooting.



19. While doing step 18, keep an eye on the deposition. Watch for color change of the control glass/Si substrates. Keep a record of changing color. As the thickness of deposited parylene increases, its color also changes due to the optical interference effect. It usually takes 6-7 min before you can see the appearance of coloration on your substrates and the walls of the quartz tube. In the reflected light, glass substrate's color starts changing in the following order:

Yellow (faint) Pink (faint) Violet-grey (faint) Pistachio green Yellow (brighter) Pink (brighter) Pistachio green (brighter) ...and then, this cycle repeats itself.

One such cycle corresponds to the parylene thickness of approx. 0.36 μ m. Therefore, to deposit about 1 μ m thick parylene film you need to go through 3 cycles like this.

- 20. When the desired film thickness is reached, quickly turn off the small heater and remove it by sliding it off the quartz tube (be careful not to disturb the silicone stopper and the TC).
- 21. Quickly cool the evaporation zone (where the boat is sitting) by rubbing it with a piece of paper towel wetted with water to stop evaporation of parylene quickly (be careful not to get water into the heater of the main furnace). At this time, the main furnace should still be kept at 700 ⁰C and the pump is running.
- 22. Turn off the main furnace; open it (be careful: everything is very hot!), reposition the quartz tube on the supporting mounts, and then remove the main furnace away by carefully shifting it backwards; right after that close the main furnace and let it cool.
- 23. Wait for the deposition tube to cool down to room temperature (when the tube is in air, it takes about 15 min).

- 24. Shut down the pump and disconnect the QF-25 flange between the quartz tube and the LN₂ trap slowly, so that the air is let in as gently as possible.
- 25. Disconnect the cold trap and place it under a ventilation hood (it has to be cleaned off the parylene deposit later).
- 26. Seal the pumping line by a blank flange.
- 27. Remove the silicone stopper/QF-25 adapter from the sample loading end of the tube (the right end) and collect the samples by pulling the aluminum plate out with tweezers. Be very careful with the gold wires, as they sometimes stick to the inner wall of the quartz tube.
- 28. The control glass or silicone substrates can be used for precise measurement of the thickness of the parylene film. If a conducting substrate was placed in the chamber, it can be used to make metal/parylene/metal capacitors for measurements of the capacitance per unit area, C_i.