# University of Minnesota Nano Center

## **Parylene Deposition System 2010-** Standard Operating Procedure

**Badger Name:** K3 Parylene Dep Labcoter SCS
**Model:** Parylene 2010 Labcoater 2
**Location:** Keller-Bay 3

**Revision:** 5
**Revisionist:** Laura Parmeter
**Date:** 3/26/2020

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# University of Minnesota Nano Center

## **Parylene Deposition System 2010**- Standard Operating Procedure

### 1. Scope

**1.1.** This document provides detailed instructions on how to properly operate the Parylene Deposition System 2010.

### 2. Tool description

**2.1.** Parylene, a polymer, deposits in a vapor form at room temperature under vacuum conditions. If forms a conformal coating on all exposed surfaces. Capable of thicknesses as little as a couple 100 nm, up to 100 µm.

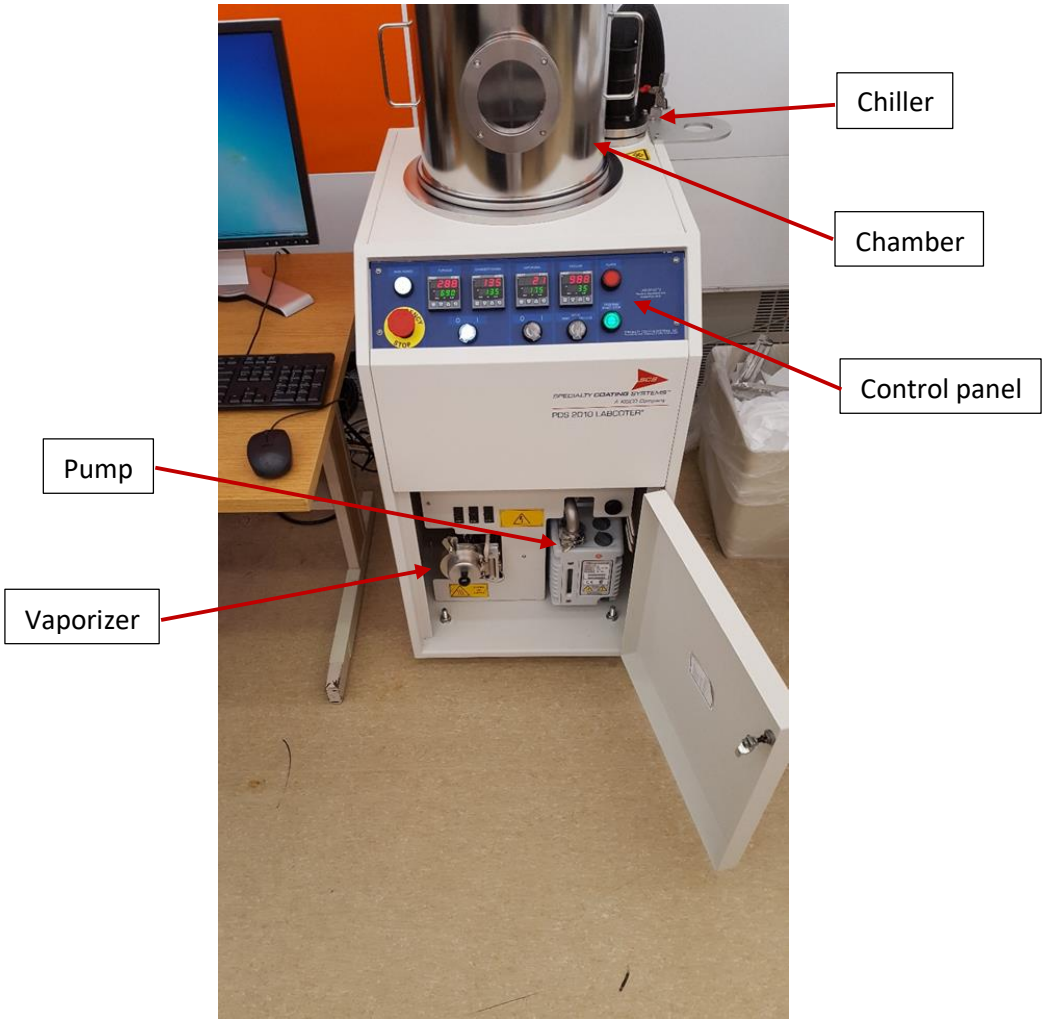



***Figure 1*** *Parylene Deposition System 2010*

## 3. Safety

**3.1.** Some areas of the system get very hot (up to 690 °C). Use caution and familiarize yourself with the location of hot surface areas.

**3.2.** The chiller on the system gets very cold (down to -90 °C). Use caution when working with the cold trap and thimble.

**3.3.** The use of a razor blade may be needed for cleaning the chiller thimble. Use caution to prevent cuts and lacerations.

## 4. Restrictions

**4.1.** Parylene will coat onto any material that is vacuum compatible.

**4.2.** No outgassing material allowed in the chamber (such as rubber, plastic, and fibrous materials). Check with MNC staff if you have questions about a particular material.

## 5. Tools and Equipment

**5.1.** Parylene Deposition System 2010 (see Figure 1)

**5.2.** Parylene C dimer

**5.3.** Aluminum foil

**5.4.** Boat former

**5.5.** A-174 adhesion promoter

**5.6.** Micro-90 soap

**5.7.** Razor blades, scoring pad, and other cleaning tools.

## 6. Procedure

### 6.1. Pre-deposition preparations

**6.1.1.** Check **Badger** for any reservations before starting. **Making a reservation is recommended** because of unpredictable run times and the adhesion promoter time sensitivity.

**6.1.2.** **Clean** substrate thoroughly.

**6.1.3.** If required, apply the **adhesion promoter** A-174. See appendix A for detailed instructions on how to prepare and apply solution.

**6.1.4.** **Dry** substrate thoroughly. **Note:** Any moisture in the chamber will cause pump down issues. If possible, consider a dehydration bake.

**6.1.5.** Create a **dimer boat** out of aluminum foil. Use the boat former to help shape the boat. Crimp one or both sides of the boats up so no dimer will fall out. The boat should **NOT** be longer than the boat former (see Figure 2).

***Figure 2*** *Use the boat former to mold the aluminum foil. The boat should not be longer than the boat form.*

**6.1.6.** Measure out the amount of dimer needed by placing the newly formed boat on the scale, zeroing the scale, and then adding dimer to one end of the boat. **See appendix B for recommended dimer amounts.**

**6.1.7.** Record dimer weight on the **log sheet**.

### 6.2. Deposition


***Figure 3*** *Control panel.*

**6.2.1.** Turn PDS 2010 on by turning the **emergency off** button clockwise and then push the **main power** button (see Figure 3). Wait until the numbers on the controls show up.

**6.2.2.** Press the green **process start/stop** button.

**6.2.3.** Turn the **furnace and chamber gauge** switch on. The furnace takes about 1.5 hours to warm up to its set point of 690 °C. Continue on to the next steps while the furnace is warming.

**6.2.4.** Vent the system by turning the **vacuum** switch to vent. Once the gauge reaches 1000 engineering units (E.U.), the system is at atmosphere. **Turn the vacuum switch back to hold once the chamber has reached atmosphere.** Neglecting to do this will damage the solenoid.

**6.2.5.** Remove the **chamber lid** and place it handle side down on the table (see Figure 4). Scratches to the sealing surface of the lid will cause problems with pumping down and make cleaning difficult. Use care.


***Figure 4*** *A designated working area is marked with green and white tape. Take care to place the chamber lid handle side down to prevent scratches to the sealing surface.*

**6.2.6.** Inspect the chamber platform and sidewalls for any flaking, peeling, or any separation of Parylene. If there's any flaking inside the chamber **do not continue** (see Figure 5). Notify MNC staff that cleaning is needed by reporting a problem in Badger.


***Figure 5*** *If Parylene is separating from the chamber sidewalls, do not continue. Cleaning is needed and should be reported to MNC staff via Badger*

**6.2.7.** Check the chamber and chiller **O-rings** for any debris or Parylene particles, clean if necessary.

**6.2.8.** Load your substrate into the chamber, making sure the substrate is not hanging off of the **rotating** platform and hitting the baffle. It's recommended to add a dummy slide or wafer to the chamber for later measurements. **Note:** Deposition rates at the center of the chamber are 8-10% less than the edges of the platform (see Figure 6).

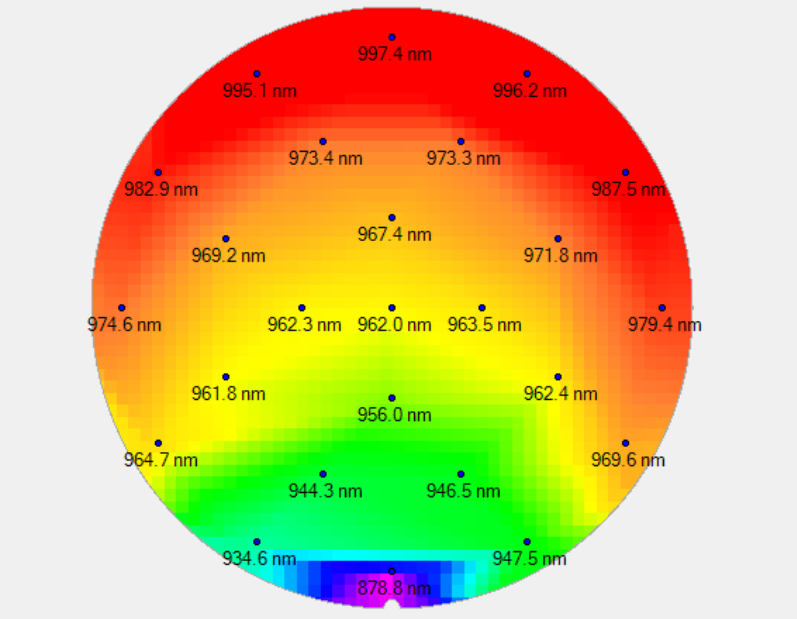



***Figure 6*** *Parylene thickness measurements, using the Filmetrics tool, of a 4 inch wafer are shown here. Deposition thickness varies from the center of platform to the edge by as much as 8-10%. Note: Purple (878.8 nm) measurement is from a shadowing effect of the center stage bolt.*

**6.2.9.** Load the dimer boat into the vaporizer with the dimer filled side closest to the door. Do not push the boat too far into the vaporizer. Keep the boat just inside the door (see Figure 7).

***Figure 7*** *Load the dimer filled boat into the vaporizer with the dimer filled side closest to the door.*

**6.2.10.** Prepare the **chamber lid** by cleaning off the O-ring grease and apply the Micro-90 soap to the whole sealing surface. Once the lid has air dried place it back on top of the chamber.

**6.2.11.** While holding the chiller thimble in place with one hand, turn the vacuum switch to **vacuum** with the other.

**6.2.12.** After 10 minutes turn the **vaporizer** switch on. **Note:** This switch is also tied in to the chiller control. It will first turn on the chiller, which has a 45 minute cool down time, then automatically turn on the vaporizer. If at any point you turn the vaporizer switch off and then back on, the 45 minute timer will start over.

**6.2.13.** If all other conditions are met but the vacuum pressure won't get below 15 E.U. then the process won't start. See Appendix C for troubleshooting.

**6.2.14.** At this point the process will run automatically. See Appendix D for details.

**6.3. Post-deposition cleaning**

**6.3.1.** Once the process is complete, indicated by the green blinking process button, **CLEANING IS NECESSARY**! Turn off the green blinking process button by pressing the button. **Note:** The heating elements will disable automatically when the process is completed but the chiller will remain on until you turn off the vaporizer switch.

**6.3.2.** Turn the **furnace** and **chamber gauge** switch off.

**6.3.3.** Turn the **vacuum** switch to **hold.**

**6.3.4.** Turn the **vaporizer** switch off. This will also turn off the **chiller**.

**6.3.5. After turning off the chiller wait 5 MINUTES EXACTLY before moving it.** Moving it too soon will cause fractures and damage the system. Moving it too late will allow moisture and chlorine from the Parylene C to drip into the cold trap.

**6.3.6.** After the 5 minute warm up vent the chamber and move the **chiller thimble** to the cleaning holder. Use care not to flex the line excessively (see Figure 8).

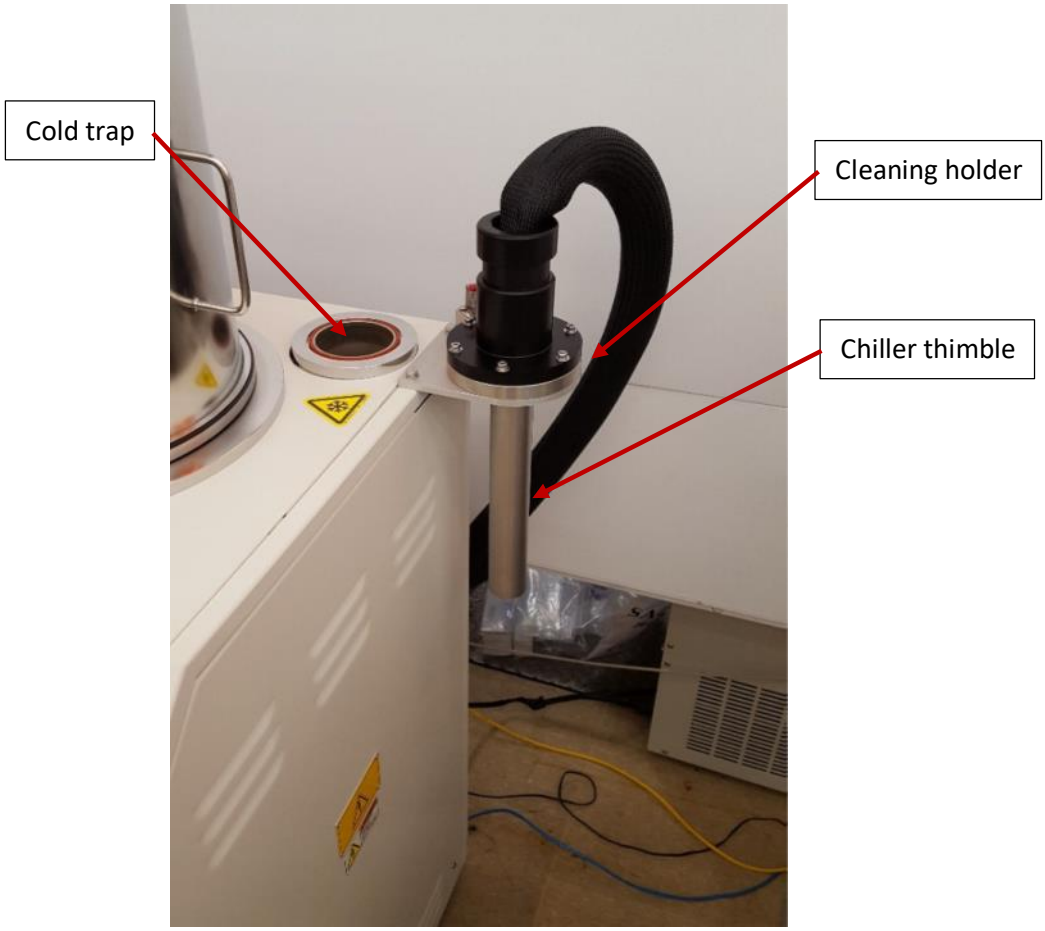



***Figure 8*** *A cleaning holder is provided for the chiller thimble. After turning off the chiller wait 5 minutes before moving it to the cleaning holder. After waiting an additional 10 minutes, or until the water crystals start to melt, clean the Parylene off of the chiller thimble.*

**6.3.7.** Wait an additional **10 minutes** for the chiller thimble to warm up outside of the chamber, or until the water crystals start to melt. **Note:** There is a balance between wanting the chiller thimble to warm but not wanting the water crystals to melt. If the water starts to melt then the chlorine in Parylene C will start to leach out and attack the metal of the chiller thimble. This will cause pitting and damage the chiller thimble.

**6.3.8.** Use a **razor blade** to slice the Parylene off of the chiller thimble. The Parylene should peel off easily once an edge is lifted up with the razor.

**6.3.9.** Use a **cleanroom rag** to get the rest of the Parylene off of the chiller thimble.

**6.3.10.** Use the scoring pad to remove the remaining Parylene from the chiller thimble. **Note: The scoring pad should only be used on the chiller thimble. Do not use it on any other surfaces of the system.**

**6.3.11.** Wipe the **chiller thimble** down once again with a cleanroom rag to clean the particles kicked off by the scoring pad.

**6.3.12.** Once the chiller thimble is **warm** and no more moisture is sticking to it, apply the Micro-90 soap, letting it air dry completely.

**6.3.13.** Peel off the Parylene from the **chamber lid**, clean off the O-ring grease, and apply the Micro-90 soap, letting it air dry completely.

**6.3.14.** Inspect the chamber platform and sidewalls for any **flaking, peeling, or any separation** of Parylene (see Figure 5). If any flaking is noticed notify MNC staff, via Badger, for required cleaning.

**6.3.15.** Remove the **dimer boat** from vaporizer. **Cation:** The vaporizer and the dimer boat will be **HOT**. Only remove the boat once the temperature has reduced significantly.

**6.3.16.** Place the **chamber lid** on the chamber.

**6.3.17.** Place the **chiller thimble** into the cold trap.

**6.3.18.** Hold the chiller thimble in place and turn the vacuum switch to **vacuum** to pump down the chamber. Wait until the chamber pressure reaches **10 E.U.** (this should only take 10 minutes).

**6.3.19.** Turn the **vacuum** switch to hold.

**6.3.20.** Press the red **emergency stop** button to turn off the system.

## 7. Film Characteristics

### 7.1. Properties

| **Typical Mechanical Properties** | **Parylene C** |
|---|---|
| Tensile Strength, psi | 10,000 |
| Tensile Strength, Mpa | 69 |
| Yield Strength, psi | 8,000 |
| Tensile Strength, Mpa | 55 |
| Tensile Modulus, Mpa | 3,200 |
| Elongation at break, % | 200 |
| Yield elongation, % | 2.9 |
| Density, g/cm$^3$ | 1.289 |
| Coefficient of friction: | |
| Static | 0.29 |
| Dynamic | 0.29 |
| Water Absorption: % (24hr) | 0.06( 0.029") |
| Index of refraction | 1.639 |
| **Typical Electrical Properties** | |
| Dielectric strength, short time (Volts/mil at 1 mil) | 6,800 |
| Volume resistivity 23°C, 50% RH (Ohm-cm) | 6x10$^{16}$ |
| Surface resistivity, 23°C, 50% RH (Ohm) | 10$^{15}$ |
| Dielectric constant: | |
| 60Hz | 3.15 |
| 1,000Hz | 3.1 |
| 1,000,000Hz | 2.95 |
| Dissipation factor: | |
| 60Hz | 0.02 |
| 1,000Hz | 0.019 |
| 1,000,000Hz | 0.013 |
| **Typical Barrier Properties** | |
| Gas Permeability | |
| cm$^3$ - mil/100in$^2$ - 24hr - at(23°C) | |
| Nitrogen | 0.95 |
| Oxygen | 7.1 |
| Carbon Dioxide | 7.7 |
| Hydrogen Sulfide | 13 |
| Sulfur Dioxide | 11 |
| Chlorine | 0.35 |
| Moisture Vapor Transmission | |
| g – mil/100in$^2$ - 24hr, 37°C, 90%RH | |
| 1mil = 1/1000in = 25.4 microns | 0.14 |
| **Typical Thermal Properties** | |
| Melting Temperature (C°) | 290 |
| Linear Coefficient of expansion (10$^{-5}$/°C) | 3.5 |
| Thermal conductivity, @ 25°C watts/Meter. Kelvin | 0.082 |

***Figure 9*** *Parylene C properties.*

### 7.2. Film thickness measurements

**7.2.1. Profilometer** – Add a dummy slide or wafer to the deposition process. Do not apply the adhesion promoter. After deposition, cut and peel away some of the Parylene from the dummy slide or wafer. Use the profilometer to measure the Parylene edge. Keep in mind that the deposition rate inside the chamber varies by as much as 8-10% from the center to the edges.

**7.2.2. Ellipsometer** – Only use if the average film thickness expected is known.

**7.2.3. Filmetrics** – Can only be used to measure film thickness. The n and k values cannot be obtained using this measuring tool.

### 7.3. Removing from substrate

**7.3.1.** If you intend to remove Parylene from the substrate at any point in the process, do not use the adhesion promoter A-174.

**7.3.2.** Parylene is soluble in chloronaphthalene and benzyl benzoate (not provided by MNC) at temperatures above 150 °C.

### 7.4. Etching

**7.4.1.** Recommend etching with $O_2$ plasma.

**7.4.2.** Recommended parameters: 100 sccm, 100 mT, 400W

**7.4.3.** Etch rates in µm/min:

| | STS etcher | AV | Oxford |
|---|---|---|---|
| **Parylene C** | 0.40 | TBD | TBD |
| **AZ9260** | 0.50 | TBD | TBD |

***Figure 10*** *Etch rates, µm/min, for Parylene and AZ9260 photoresist on the STS etcher.*

# Appendix A

## Adhesion promoter A-174

********The shelf life of the adhesion promoter solution is only ***24 hours***. Plan ahead!*

**1. Preparing solution**

To prepare the solution, mix equal parts isopropyl alcohol (IPA) and deionized water (DI). Then add 0.5% A-174 (100:100:1 of DI:IPA:A-174). Stir for 30 seconds. To allow for adequate chemical reaction the solution must rest for at least 2 hours.

**2. Applying solution**

**2.1** Submerge substrate in the solution for 15-30 minutes.

**2.2** Remove substrate and allow to air dry for 15-30 minutes.

**2.3** Submerge substrate in IPA for 15-30 seconds, agitating the beaker during process.

**2.4** Remove substrate and drain adequately (30-60 seconds).

**2.5** Dry parts thoroughly. If possible consider a dehydration bake.

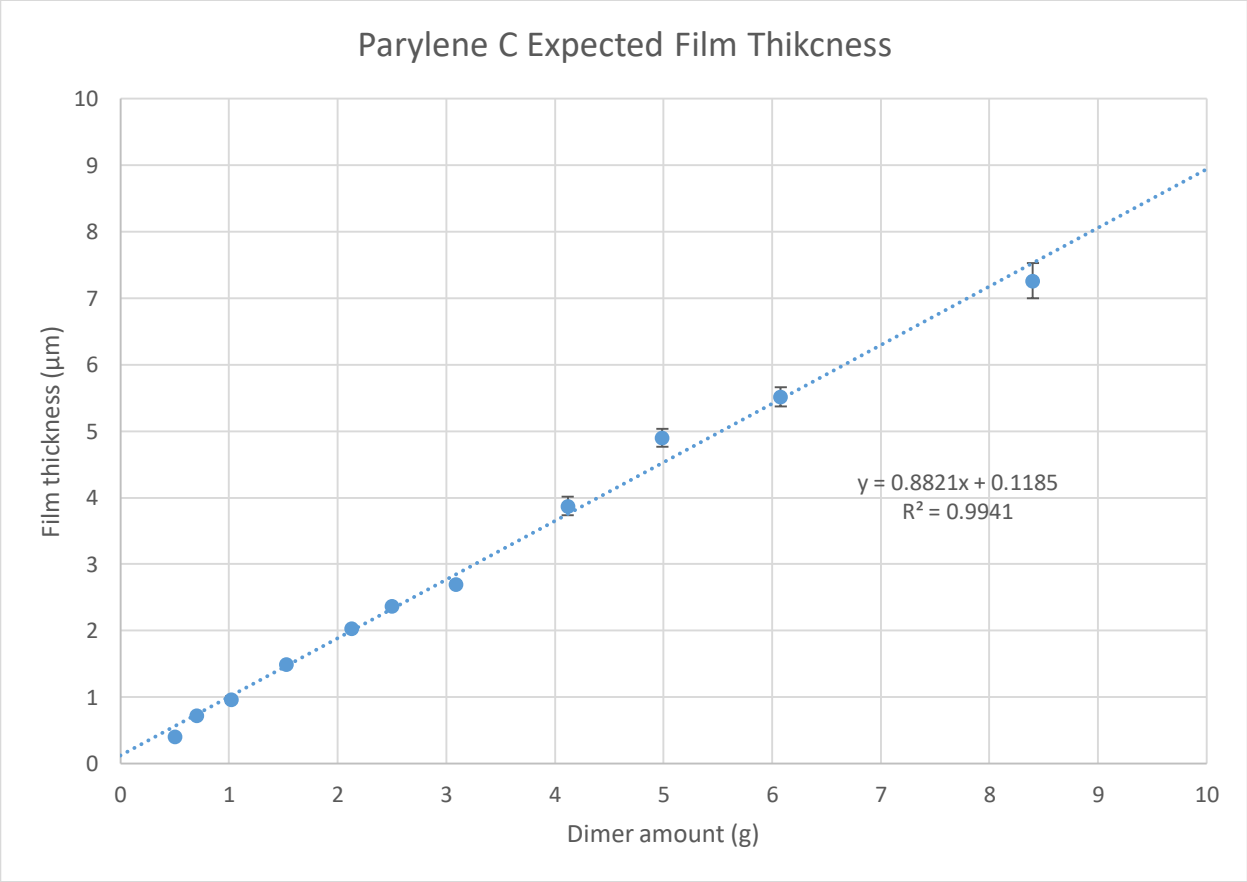
***Substrates should be coated with Parylene within 30 hours of adhesion promoter solution being applied.*

# Appendix B

## Dimer amount vs film thickness

*Thickness acquired during the process depends on the amount of dimer used and the total surface area being coated. In equation 1, T is the film thickness in µm and D is the amount of dimer in grams:*

$$T=0.8821D+0.1185 \quad \textit{(eq. 1)}$$




***This tool was characterized using a flat silicon wafer on the edge of the rotating platform. Measurements were taken using a profilometer.*

# Appendix C

## Troubleshooting

### 1. Pump down issues

**1.1** Pump down issues will cause the pump to work too hard causing the oil to become frothy (see Figure 9).


***Figure 9*** *Frothy oil in the pump is a telltale sign the pump is working too hard.*

### 1.2 Chamber lid

**1.2.1** Debris/Parylene on the lid or O-ring can cause a leak. Clean off the lid and O-ring.

**1.2.2** Make sure any excess grease on the lid is wiped off.

**1.2.3** Make sure there is a thin layer of grease on the O-ring. Too little will not be effective and too much will cause a virtual leak.

**1.3** Moisture in the system caused by either the chiller being turned on before vacuum is established, or from substrate not being dried properly. Remove the chiller thimble, wipe away any moisture, and allow the thimble to warm completely. Remove your substrate and dry thoroughly. Consider a dehydration bake.

**1.4** Some materials have high outgassing, like rubber, plastic, and fibrous materials, and are not allowed in the chamber. Check to make sure your material is vacuum compatible.

**2. Cloudy film**

**2.1** Cloudy film can be caused by too high of a deposition rate. Deposition rate is controlled by the vacuum pressure. When the deposition process starts the vacuum pressure is likely to go over its setpoint of 35 E.U.

**2.2** It is possible to lower the vacuum pressure setpoint. The benefit of this is only at the initial rise of the vaporizer temperature. The overshoot will still happen but the idea is to keep the overshoot below 35 E.U. A consequence of lowering the vacuum pressure setpoint is the deposition time will increase, i.e. runs will take longer. Contact MNC staff if you would like to decrease the vacuum pressure setpoint.

# Appendix D

## Deposition process

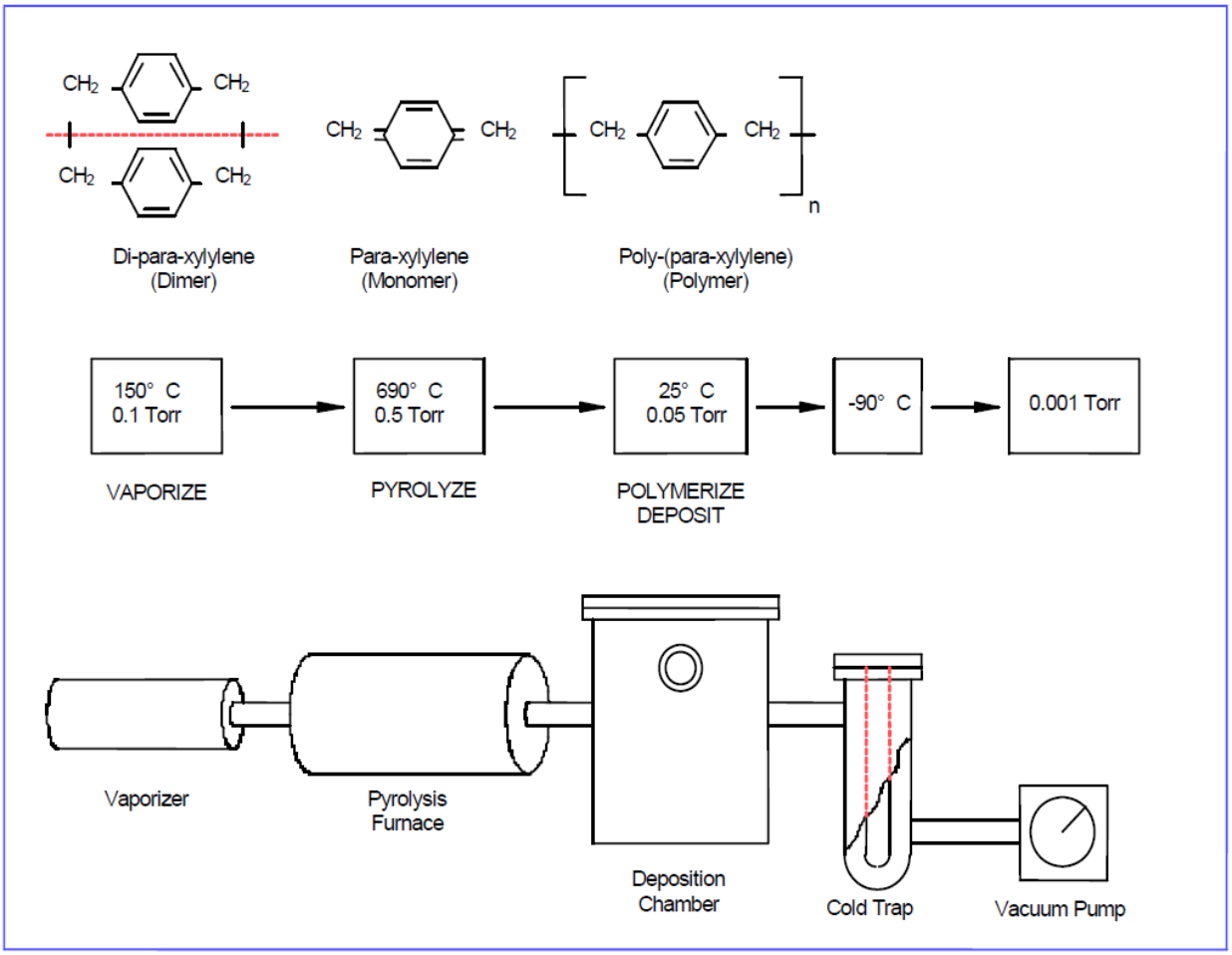
*Parylene, a polymer, deposits in a vapor form at room temperature under vacuum conditions. It forms a conformal coating on all exposed surfaces.*

**Deposition occurs in 3 steps:**

**Step 1: Vaporization** – Application of heat under vacuum vaporizes the Parylene dimer (in a solid powder form) into a gas.

**Step 2: Pyrolysis** – At high temperature, the gaseous dimer is cleaved into a monomer.

**Step 3: Polymerization** – At room temperature, the gaseous monomer deposits as a polymer onto the substrate in the vacuum chamber.



# Appendix D

*The system checks for three conditions before the deposition process will start. First the chiller needs 45 minutes to cool down to less than -90 °C, second the furnace needs to get to its set point of 690 ±20 °C, and third the vacuum pressure needs to get to its base point of 15 E.U. If there are pump down issues the vacuum pressure may never get to its base point and the process will never start. Once the vacuum pressure has met its base point of 15 E.U. then the vaporizer temperature will start to rise. Once the vaporizer temperature gets above 90 °C the dimer has started to vaporize, subsequently causing the vacuum pressure to rise, and coating has begun. The vaporizer temperature will continue to rise until the vacuum pressure rises to its set point of 35 E.U. If there is an overshoot of the vacuum pressure, the vaporizer temperature will decrease until the vacuum pressure falls back to 35 E.U. The system will keep the vacuum pressure at 35 E.U. by slowly increasing the vaporizer temperature until all the dimer is consumed. Parylene that is not deposited in the chamber will be "caught" by the cold trap. This will protect the vacuum pump. The vaporizer temperature will continue to rise, trying to keep the vacuum pressure at 35 E.U., until it reaches its set point of 175 °C. If the vaporizer temperature reaches its set point and the vacuum pressure starts to drop below 35 E.U. the system has entered its bake out mode. The vaporizer temperature will continue to rise until the vacuum reaches its base point once again, upon which the temperature will hold for 5 minutes. This step ensures that any leftover Parylene is removed from the system. The green process button will start to blink indicating that the process is complete.*