

SCS Parylene Deposition Uniformity

Roger Robbins

1/5/2011



The University of Texas at Dallas
ERIK JONSSON SCHOOL OF ENGINEERING

SCS Parylene Deposition Uniformity

Roger Robbins

1/5/2011

<C:\\MyDocuments\\CleanRoomGeneral\\Equipment\\ParyleneDepTool\\ParyleneDepManual.doc>

Table of Contents

INTRODUCTION.....	3
TOOL OVERVIEW	3
PROCESS OVERVIEW	5
PROCESS DETAIL FOR UNIFORMITY EXPERIMENT	5
<i>Control Parameters</i>	5
<i>Experiment Setup</i>	6
<i>Sample Placement</i>	6
<i>Process Profile</i>	7
<i>Process Flow</i>	8
Fault Conditions	8
THICKNESS UNIFORMITY DATA	9
<i>Wafer Uniformity</i>	9
<i>Analysis: Wafer Uniformity</i>	9
In-Wafer Uniformity	9
Wafer-to-Wafer Uniformity.....	11
<i>Analysis: Small Sample Uniformity</i>	11
Sample-to-Sample Uniformity	11
Shelf-to-Shelf Uniformity	15
CONCLUSION	15
APPENDIX A – PROCESS CONTROL PARAMETERS.....	17
PROCESS CONTROL PARAMETERS	17

SCS Parylene Deposition Uniformity

Roger Robbins

1/5/2011

<C:\MyDocuments\CleanRoomGeneral\Equipment\ParyleneDepTool\ParyleneDepManual.doc>

Introduction

The SCS (Specialty Coating Systems) Parylene Deposition tool is heavily used by researchers in the UTD Clean Room for conformal protective coating of devices or in some cases even using the material as an insulating layer in the device structure. The common assumption is that the deposited film thickness is highly uniform. However, to my knowledge, no one has actually measured the uniformity to verify the assumption. This paper presents the uniformity data characterizing the film thickness over 4" wafers as well as over small samples placed on all three of the shelves. Details of the tool operation are described in the tool instruction manual.¹

Tool Overview

The Parylene deposition system consists of a series of vacuum chambers that sequentially produce parylene vapor, pyrolyze it, deposit it as a polymer, and then capture its effluent. The Vaporizer chamber is a horizontal tube at the bottom of the tool behind the front panel. It has a hinged door that is held in place by a simple latch. This is where the foil boat with the Parylene Dimer pellets is loaded into the system. The Pyrolyzer furnace is a vertical tube connected to the back of the horizontal Vaporizer, and is the place that the Dimer vapor is broken into Monomers in preparation for deposition on the substrates in the Deposition Chamber. Figure 1 depicts the Parylene chemistry process and the vacuum system schematic.

¹ Robbins, Roger, "STS Parylene Deposition Tool Manual", <C:\Documents and Settings\rar011300\My Documents\Clean Room General\Equipment\ParyleneDepTool\ParyleneDepManual.doc>, (1/5/2011).

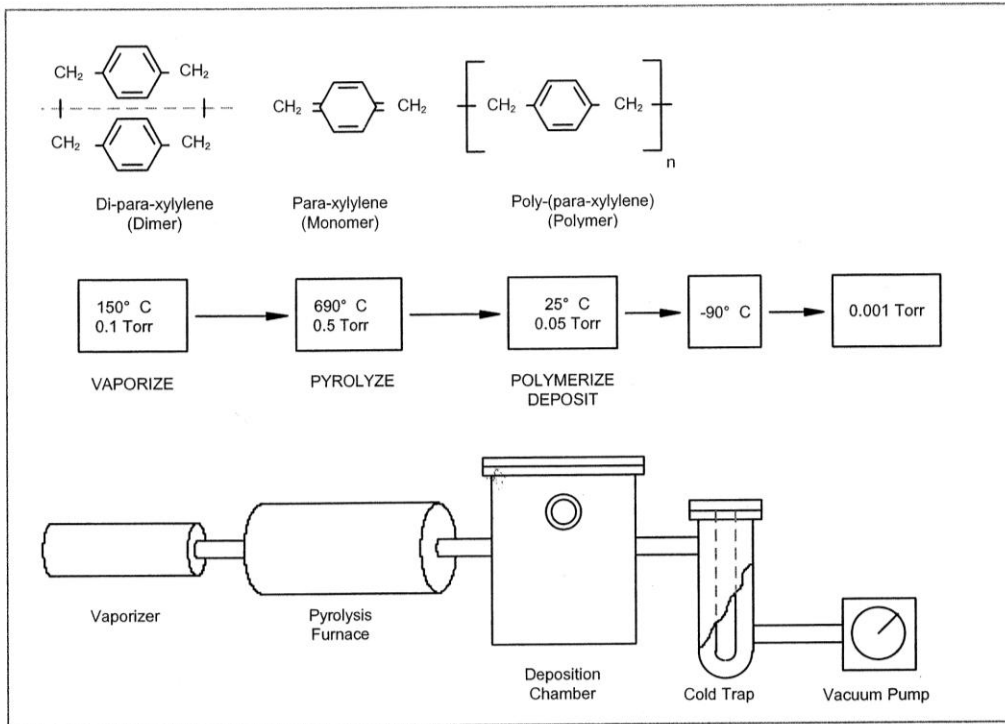


Figure 1. Generic Parylene chemistry, process concept, and vacuum system schematic from the STS Operation Manual. For the Parylene-C chemistry, the vaporization temperature is between 90 and 175 C, and in our system the cold trap is chilled by Liquid Nitrogen to 77 deg K, or -196 C (77-273=-196 C).

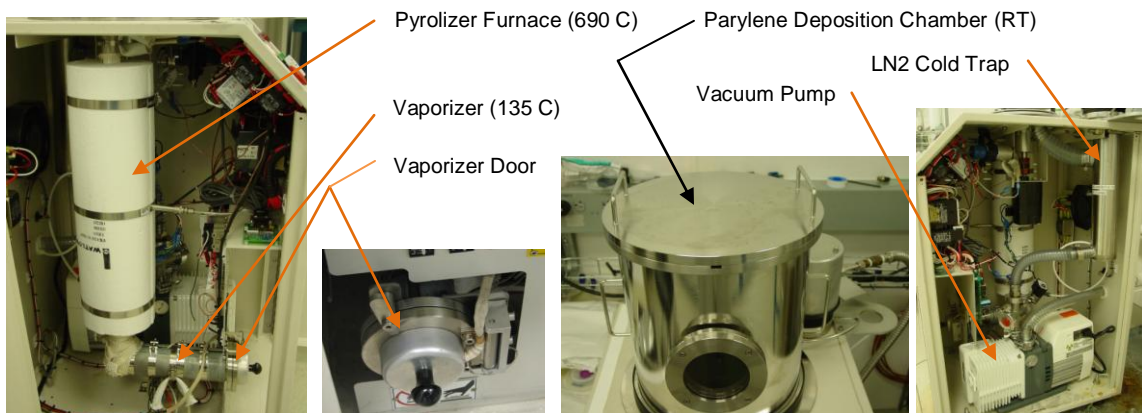


Figure 2. Elements of the Parylene Deposition vacuum system. 1) Vaporizer, 2) Pyrolyzer Furnace, 3) Deposition chamber, 4) Cold Trap, 5) Vacuum Pump.

Process Overview

The Parylene deposition process is fairly simple and consists of measuring a pre-determined mass of Parylene Dimer into an Aluminum foil “boat”, venting the system, and placing the dimer and carrying boat into the Vaporizer through its door behind the large front door of the system chassis. After the deposition system preparation is completed, processing parameters are set into the controller memory and the system is started by switching on all the front panel rotary switches and pressing the Green Start button. After about 2.5 hours, the process will self complete and flash the Green Start/Stop button. At this point, the user will vent the system and remove the samples.

Process Detail for Uniformity Experiment

Control Parameters

Before starting the run, control loop parameters based on previous experimental runs were input into the Vacuum and Vaporizer controllers to optimize the process control in our tool. These parameters are listed in detail in Appendix A, but the key parameters are listed in Tables 1 and 2 below. The objective of setting these parameters is to enable the controllers to manage the vaporization rate of the Parylene Dimer to control the vapor pressure in the deposition chamber to a value which determines the deposition and polymerization rate of the parylene polymer onto the samples. These parameters also control the overshoot and under shoot of the process pressure. Basically these parameters determine the quality of the film.

Table 1 - Key Vacuum Controller Parameters

Parameter	Description	Value
Pb_P	Proportional Band	0.5%
ArST	Auto Reset (Integral Time)	2.59 sec (Default=3.00)
rAtE	Rate (Derivative Time)	1.40 sec
PLA 1	Base Pressure	6 m Torr
SP	Process Pressure Setpoint	12 m Torr

Table 2 - Key Vaporizer Controller Parameters

Parameter	Description	Value
Pb_P	Proportional Band	5%
ArST	Auto Reset (Integral Time)	2.00 sec
rAtE	Rate (Derivative Time)	0.30 sec
PhA 2	Low Temp Setpoint	174 C
SP	Vaporizer Temp Setpoint	175 C

Experiment Setup

In order to obtain film thickness measurements, an appropriate film thickness must be used for the Clean Room ellipsometer to be able to measure the thickness. A film thickness of 300 nm was chosen. In order to obtain this thickness, 630 mg of parylene is needed in the Vaporizer². This amount was weighed out on the Metler balance and inserted into the Vaporizer using an Aluminum foil boat to save generating a mess in the Vaporizer with Parylene Dimer residue after the deposition cycle completes (Figure 3). The Dimer pellets were spread with a spatula into a single or double layer in the boat to facilitate repeatable sublimation rates.

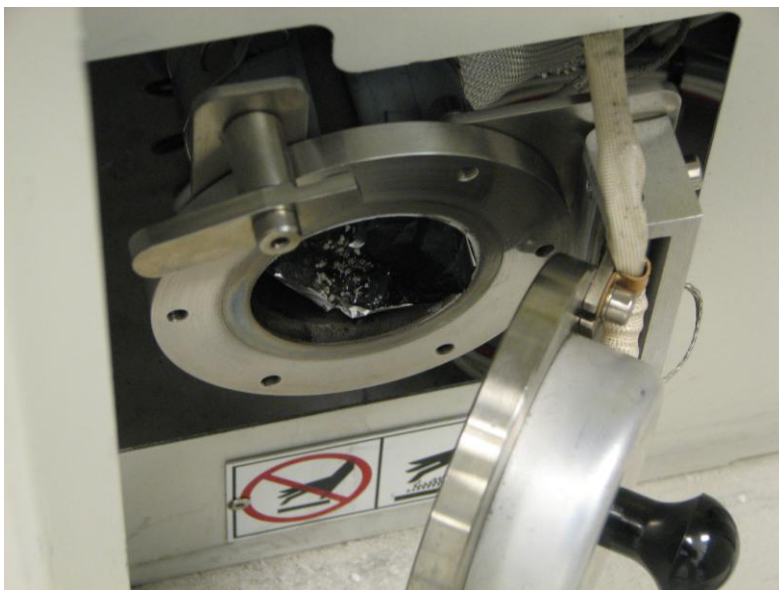


Figure 3. Parylene dimer spread into a single or double layer on an Aluminum foil Boat in the Vaporizer Furnace of the Parylene Deposition Tool.

Sample Placement

The Silicon samples consisted of 3 full sized 4 inch wafers and a number of pieces of Si wafer spread around the perimeter of each shelf as depicted in Figure 4. The samples were marked according to their location so that correlation between film thickness and location could be established. These were brand new Prime 100 Si wafers from the shipping boat.

² Robbins, Roger, "SCS Parylene Deposition Tool Manual," (1/5/2011).

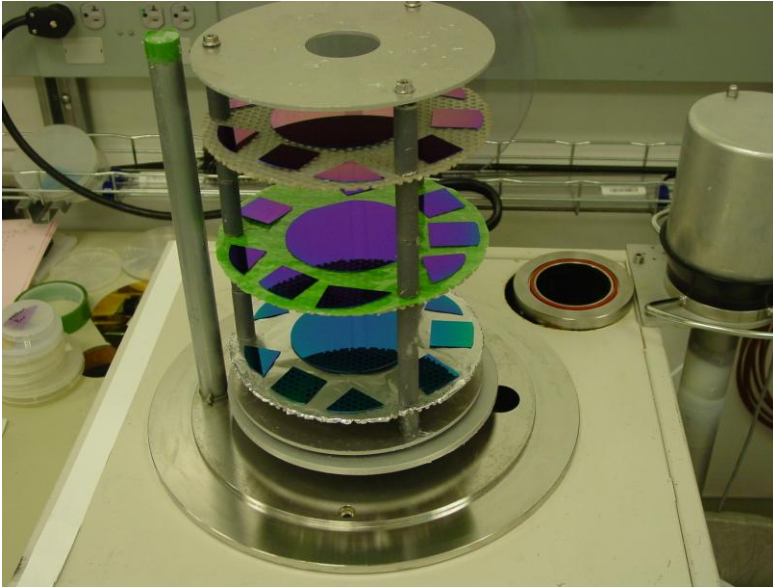


Figure 4. Photo of Si sample arrangement and placement on rotating triple shelf of the substrate platen used for Parylene film thickness uniformity determination.

Process Profile

During the process, all the system parameters were measured as a function of time. This data is displayed in Figure 5.

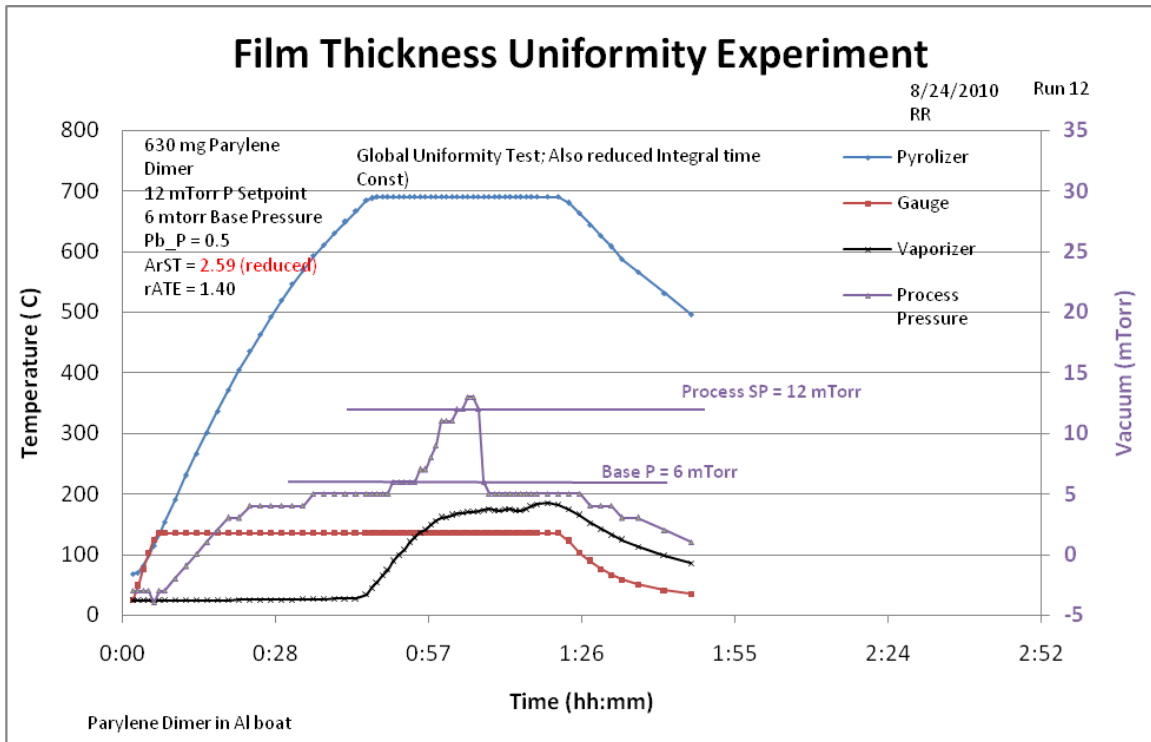


Figure 5. Process parameters vs time.

Process Flow

When the process is started, all the switches on the front panel are Enabled and the green Start button is pressed. This causes a chain of events that the graph of Figure 5 charts. First, the Pyrolizer and the Vacuum Gauge heaters start heating. The Vacuum Gauge heats up rather quickly to prevent any parylene from depositing inside the gauge tube. After a few minutes, the vacuum in the deposition chamber climbs due to out-gassing from the dimer as the vaporizer warms a bit by conduction from the Pyrolizer furnace as it rises toward 690 C. When the Pyrolizer temperature reaches about 670 C, the Vaporizer begins to heat. By the time the Pyrolizer reaches its steady state process temperature of 690 C, the Vaporizer temperature is rising significantly. As this temperature approaches 90 C, the Parylene Dimer begins to sublime and stimulates the process pressure to rise rapidly. As the Process pressure approaches its set point at 12 m Torr, the Vaporizer temperature begins to stabilize. During the controlled deposition, the process pressure has stabilized at the Set Point and remains there until the Parylene Dimer has all sublimated and then the process pressure suddenly drops to baseline. After about 5 minutes at baseline while the Vaporizer temperature rises to maximum to drive out any remaining dimer, the controller times out and all the heaters are turned off to end the process. The cooling takes about 30 minutes until the temperature is low enough to safely open the system to retrieve the samples.

Fault Conditions

There are some fault conditions that affect the progress of the process but do not create a fault notification. These subtle conditions stem from the deposition chamber pressure not achieving pressures during the process that meet set point values set in the controller. Listed below are the 3 circumstances I know that will stall the process:

1. The Deposition chamber vacuum pressure must achieve a pressure less than the "Base Pressure" value set into the chamber pressure controller when the pyrolizer and chamber pressure gauge temperatures are at programmed temperatures before the Vaporizer is enabled to heat the Parylene Dimer (thus actually starting the deposition process).
2. The process pressure must rise above the process Set Point before it will recognize the end-of-run conditions and shut down properly.
3. Chamber pressure must also drop below the set base pressure at the end of the process before the system will recognize the end-of-process state and shut down the system (after a 5 minute delay at process conditions).
 - a. If, at the end of the process when the chamber pressure drops below the process set point, the chamber pressure does not drop below the base pressure value, the system will maintain all temperatures for 30 minutes before shutting down the system.

After considering the three conditions above that create blind "process faults" you can see why the base pressure set point is so important. For example, if a leak is created by not having good o-ring seals, then the system can stall the process without telling you. Thus you must make sure that the o-rings and gaskets are clean before you put the lid, chamber, or LN2 cold trap back together after loading your samples.

Thickness Uniformity Data

Wafer Uniformity

Wafer uniformity was measured by two methods – Ellipsometry in 5 locations (center and four edges) around the wafer, and via the NanoSpec tool in 49 points over the wafer which produced a 3D graph of the thickness profile. The target film thickness was 300 nm. Table 3 records the Ellipsometry data for the top, middle and bottom shelf wafers. Wafer-to-Wafer Uniformity is calculated in Table 4 via the formula $(\text{Max}-\text{Min})/(\text{Max}+\text{Min})\times 100$ (%).

Table 3
Parylene Film Thickness – Internal Wafer non-Uniformity

Location	Parylene Film Thickness (nm)		
	Top Wafer	Middle Wafer	Bottom Wafer
1	270.32	293.00	304.17
2	271.79	293.37	305.38
3	271.45	293.40	305.26
4	271.59	293.42	305.01
5	271.89	293.42	304.82
Average (nm)	271.408	293.322	304.930
Sigma (nm)	0.6318	0.18	0.476
Range (nm)	1.57	0.42	1.21
Non-Uniformity	0.290%	0.072%	0.199%

Table 4
Top to Bottom Wafer-to-Wafer non-Uniformity

Thickness Average =	289.886 nm
Thickness Sigma =	14.39 nm
Average Range =	33.52 nm
Non-Uniformity =	6.09%

Analysis: Wafer Uniformity

In-Wafer Uniformity

Thickness data taken from the ellipsometer were collected in the interior space that would have been used to make devices. This data shows a very high degree of in-wafer uniformity for a single deposition run – less than 1.5 nm variation in thickness across the device area of the 4 inch diameter wafers. In percentage terms, the film thickness variation on these three wafers was less than 0.3% for each of the sample shelves in the Parylene tool.

The morphology of the non-uniformity is shown in the graphs of film thickness vs location taken by the NanoSpec film thickness tool, (Figures 6 & 7). Basically it shows that the film may be uniform in the central portion of the wafer, but at the perimeter, the film thickness is slightly thicker. In fact, the total thickness range is on the order of 40

nm. This apparently is a fundamental characteristic of the parylene deposition process. In a later section of this document we also find that the edges of small samples show the same characteristic thickness increase. Figures 6 and 7 show a 3D panorama of the effect on the top and bottom wafers in the parylene Deposition tool (could not get the NanoSpec tool to measure the middle shelf wafer)

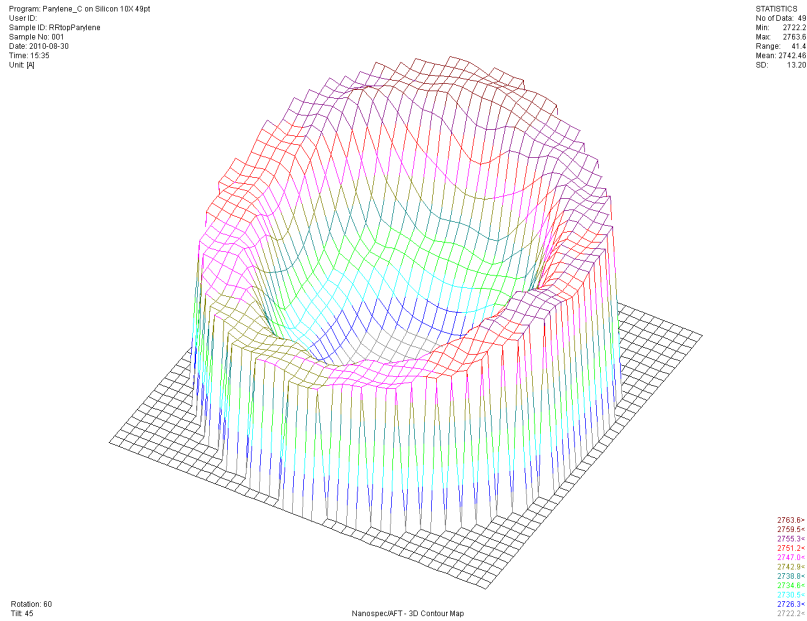


Figure 6. 3D graph of the thickness of Parylene on the top wafer in the SCS parylene deposition chamber.

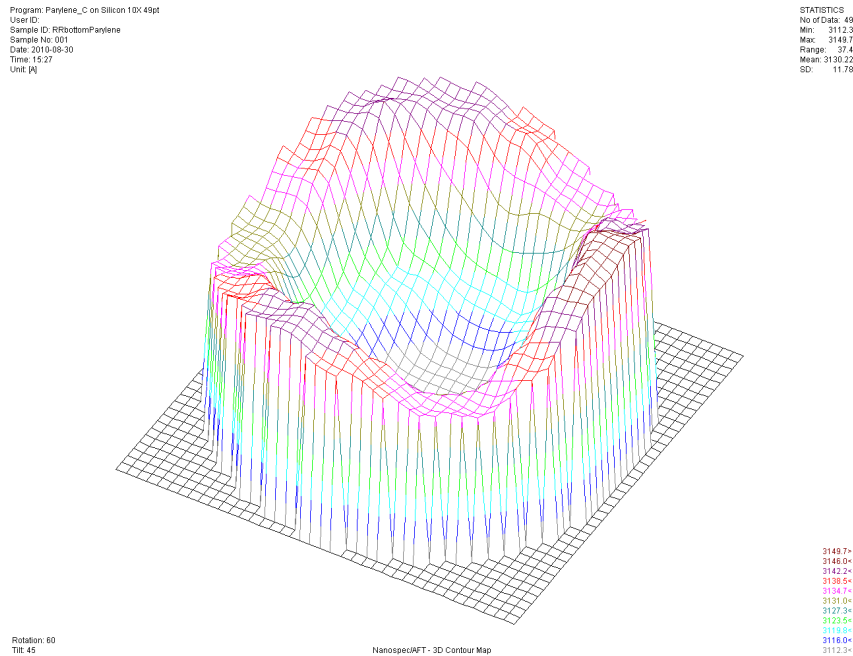


Figure 7. 3D graph of the Parylene film thickness on the bottom shelf wafer.

Wafer-to-Wafer Uniformity

Film thickness variation between the wafers on the three shelves of the sample platen showed a considerable variation – 6.8% non-uniformity. The total thickness variation range between wafers is on the order of 40 nm for a film of 300 nm thickness (13%). The top shelf produces thinner films and the bottom shelf produces thicker films.

Analysis: Small Sample Uniformity

Nine small samples were arranged around the perimeter of each of the three substrate shelves in the parylene deposition tool as previously shown in Figure 4. The general size of the small samples is on the order of 1x1 inch, which appears to be the about the norm for user sample sizes. The numbering of the sample locations was identical on all three shelves and started at Post #1 which is marked on the rotating platen. This record was intended to allow the determination of any possible rotation bias in thickness, since the platen rotation is off center by about 1.2 inch.

Sample-to-Sample Uniformity

The ellipsometer was used to measure the thickness at the center of each small-sample for thickness comparisons between samples. Data is shown in Table 5.

Table 5
Small Sample-to-Sample Non-Uniformity

Location	Parylene Film Thickness (nm)			Deviation from Average (nm)
	Top Shelf	Middle Shelf	Bottom Shelf	
1	272.39	294.63	311.7	-0.57
2	273.63	296.59	314.7	1.49
3	273.63	293.6	310.26	-0.98
4	273.29	294.8	308.5	-1.28
5	274.23	296.49	313.28	1.19
6	277.6	294.8	311.15	1.04
7	274.93	295.79	309.47	-0.08
8	274.59	-Visiting Sample-	312.8	0.21
9	274.84	294.28	308.52	-0.93
Average (nm)	274.35	295.12	311.15	
Sigma (nm)	1.47	1.07	2.17	
Range (nm)	5.21	2.99	4.78	
Non-Uniformity	0.95%	0.51%	0.99%	

The sample-to-sample film thickness variation looks reasonably good with an apparent film thickness range of 3 – 5 nm, which is less than a 1% variation. This data was taken with the ellipsometer roughly in the middle of each sample. However the color appearance to the eye suggested that there might be a some in-sample variation which could have entered into the single point data in Table 5. After examining the small samples when they were arranged as in Figure 8, the eye noted that the outer rim of the samples in the circle had a lighter color than the inner rim. This observation stimulated a closer examination of the film thickness uniformity inside individual small samples. This data is shown in Table 6. Note that location 2 (edge of sample facing the perimeter of the rotating sample platen) in both samples has the thickest Parylene film. This suggests that a film thickness gradient exists at the outer rim of the rotating platen. To improve the uniformity of small samples, place them about $\frac{3}{4}$ inches inside the outer rim of the platen shelf.

This data shows that small sample interior film uniformity may have thickness variations up to the range of 12 nm if the edges are considered. (4%)

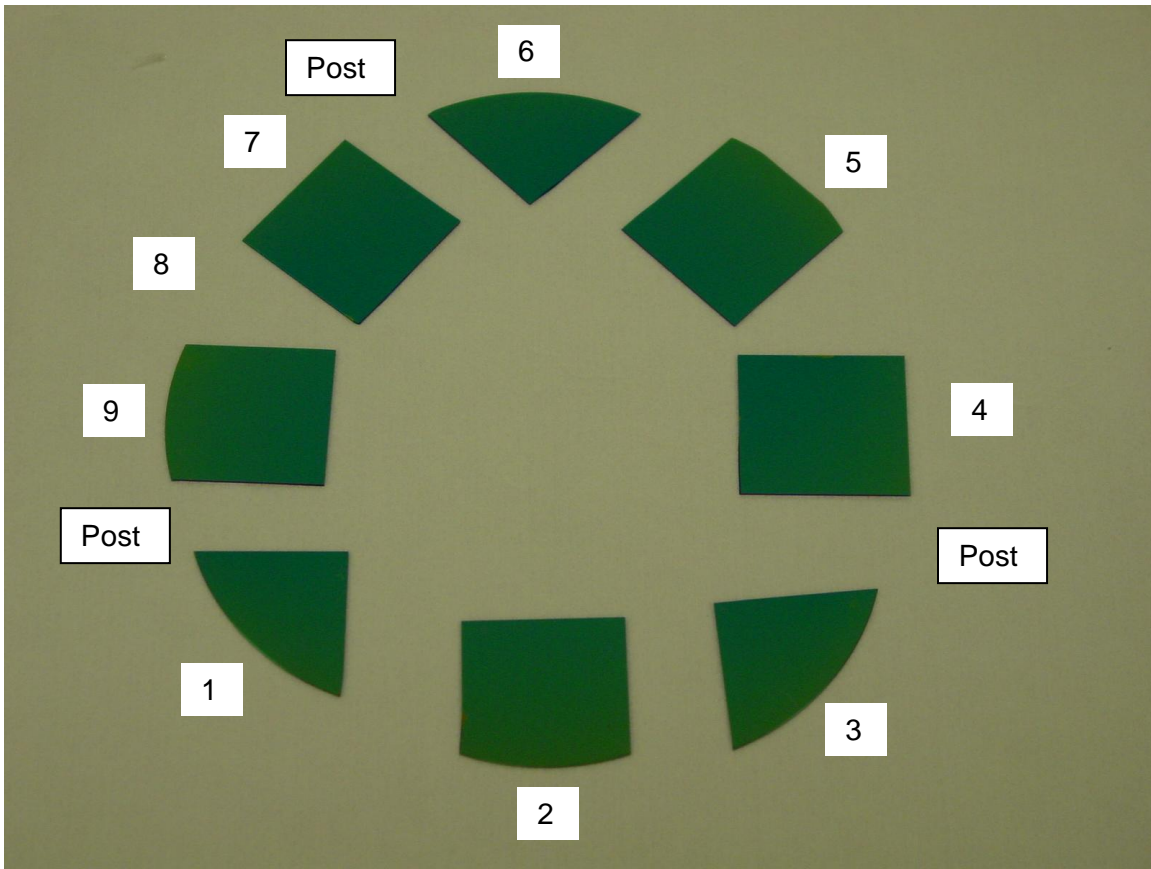


Figure 8. Location numbering for sample locations on parylene substrate rotating platen showing location of platen support posts. These samples came from the middle shelf of the substrate platen and are laid out in the photo in the order in which they were placed on the platen. Note the location of the platen support posts.

Table 6
In-Sample Uniformity of Small Samples

Location	Sample 1	Sample 2
1	308.69	296.49
2	313.58	307.15
3	311.20	296.57
4	305.58	294.72
5	306.32	296.86
Average (nm)	309.07	298.36
Sigma (nm)	3.35	4.99
Range (nm)	8	12.28
Non-Uniformity	1.29%	2.07%

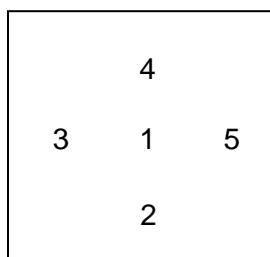


Figure 9. Ellipsometer measurement locations on small samples. Location 2 is on the rim of the platen shelf where the thickness is higher than the interior.

There might be another aspect to the uniformity of these small samples from the location of the small sample in relation to the support posts for the platen shelves. Table 5 has a column that calculates the deviation of the average film thicknesses for small samples placed at the same angle on each of the platen shelves from the full data average thickness. This is plotted in the chart in Figure 10 as a function of the placement angle. Note that the film thickness average of all samples at the same location angle is less on either side of the post location compared to the film thickness midway between the posts. This effect appears to be most prominent on the middle and bottom shelf compared to the top shelf (Figure 11). This is probably due to the gas flow characteristics around the support posts during the deposition as the platen rotates. Note that the gas is introduced into the deposition chamber with a pipe at the side of the chamber with its port holes facing the wall of the chamber so that the posts both approach the source and recede from it in the same rotary motion. This would explain why both sides of the post could see flow shadows that slightly inhibit the deposition rate just behind the post. To improve uniformity, place samples about 1 inch away from posts.

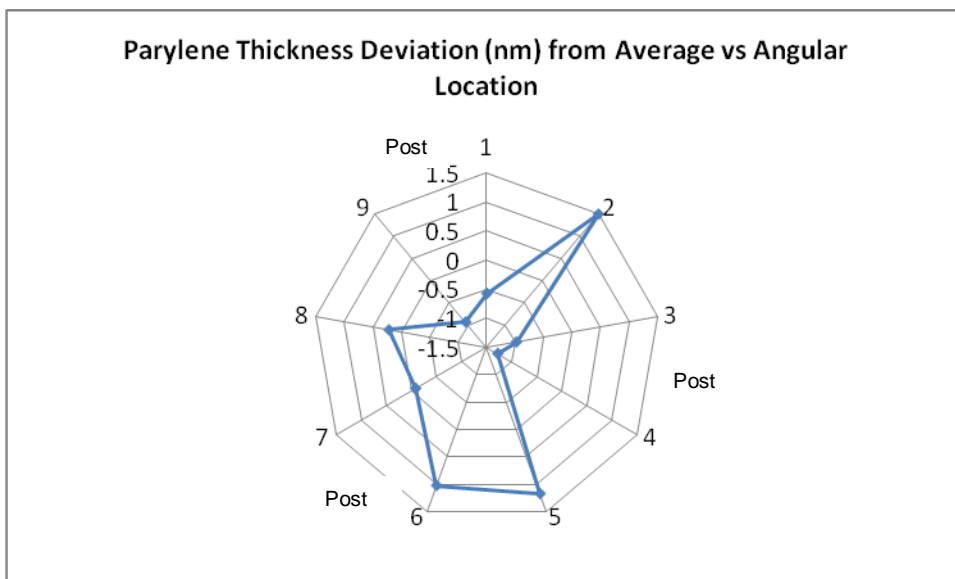


Figure 10. Magnitude of thickness deviation from total average film thickness of small samples on all three platen shelves. Note that the film thickness is generally lower next to the post locations.

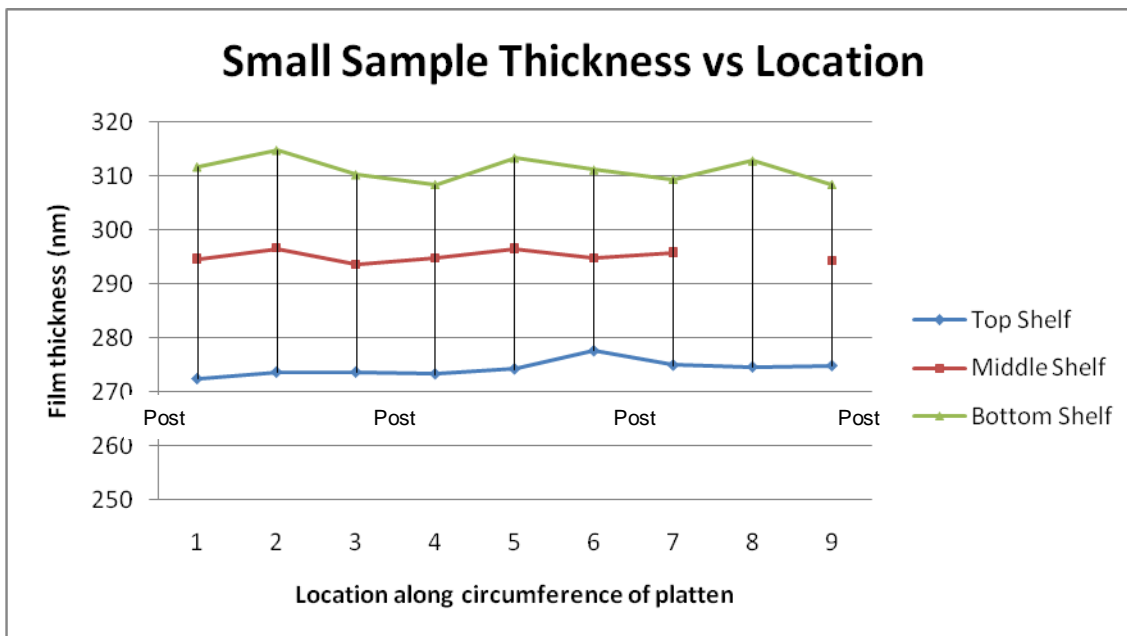


Figure 11. Plot of film thickness as a function of angular location of the small samples. Note the low thickness values next to the post locations. This effect is greater on bottom shelves compared to the top shelf.

Shelf-to-Shelf Uniformity

The thickness data in the small-sample trial appear to reproduce the shelf-to-shelf variation noted with the wafers – 6.3% non-uniformity compared to the wafer-to-wafer non-uniformity of 6.1%. The Non-Uniformity data range is 37.15 nm vs 35.06 nm for the wafer shelf-to-shelf spread. The difference in uniformity between the full wafers and the small sample set is very small. This is most likely due to the monomer gas flow pattern inside the deposition chamber stemming from the gas input nozzle pipe and the rotating platen as well as the location of the exhaust port at the opposite bottom of the chamber from the input pipe.

Conclusion

This paper has presented non-uniformity data for a single parylene run loaded with 3 wafers at the center of each shelf of the sample platen and 9 small (1x1 inch) sized samples ringing each shelf at the outer edge. The film thickness target was set at 300 nm so that the ellipsometer could easily measure the film thickness. This required 650 mg of Parylene-C Dimer starting material to accomplish. The experiment used the process parameters listed in the tables in Appendix A. The experimental results are summarized in Table 7.

Table 7
Final Results: Non-Uniformity of Parylene Film Thickness
Thickness Target = 300 nm

Test Type	Sample Type	Non-Uniformity	Thickness Range
In-Wafer Unif.	Top Shelf Wafer	0.29%	1.57 nm
	Middle Shelf Wafer	0.07%	0.42 nm
	Bottom Shelf Wafer	0.2%	1.21 nm
Shelf-to-Shelf Wafer Unif.	Wafer	6.1%	35 nm
Small Sample to Sample Unif.	Top Shelf Small Sample	0.95%	5.21 nm
	Middle Shelf Small Sample	0.51%	2.99 nm
	Bottom Shelf Small Sample	0.99%	4.78 nm
Shelf-to-Shelf Small Sample Unif.	Small Sample	6.3%	37.15 nm
In-Sample Non-uniformity	Small Sample, Middle Shelf	2.07%	12.43 nm

The bottom line is that the Parylene deposition process produces a very uniform film. There are, however slight differences between deposition rates between the three sample shelves. We also noted that there is a fundamental footprint of the film thickness pattern that shows slightly thicker film at the boundaries of substrates and also at the rim of the round sample platen. There is also a slight shadowing effect from the three posts that hold the sample shelves together if the sample is placed at the rim of the shelves.

Appendix A – Process Control Parameters

Roger Robbins

1/5/2011

Process Control Parameters

The following table lists all the parameters that were set into the controllers to produce the uniformity data for this paper.

Table IA
Vacuum Controller Parameter list

Parameter	Description	Value
FiLt	Input Filter Time Constant	2 sec
OFFS	Process Variable Offset	0
PPLJ	Primary (Heater) Power	Read Only
Pb_P	Proportional Band	0.5%
ArST	Auto Reset (Integral Time)	2.59 sec (default=3)
rAtE	Rate (Derivative Time)	1.4 sec
biAS	Manual Reset (Bias)	25
SPuL	Setpoint Upper Limit	100 mTorr
SPLL	Setpoint Lower Limit	-4 mTorr
OPuL	Output Power Limit	100%
Ct 1	Output 1 Cycle Time	8 sec (Norm=32)
bAL 1	Band Alarm 1	-
PhA 1	High Alarm 1	-
PLA 1	Low Alarm (Base Pressure)	6 mTorr
AHY 1	Alarm 1 Hysteresis	1
PhA 2	High Alarm 2	-
AhY 2	Alarm 2 Hysteresis	1
Apt	Auto Pre-Tune	Disabled
PoEn	Auto/Man control Selection	Disabled
SPr	Setpoint Ramping	Disabled
rP	SP Ramp Rate value	OFF
SP	Process Pressure Setpoint	12 mTorr
SLoc	Setup Lock Code	10

Note: Key parameters are listed in Red

Table 2A
Vaporizer Controller Parameter list

Parameter	Description	Value
FiLt	Input Filter Time Constant	2 sec
OFFS	Process Variable Offset	0
PPLJ	Primary (Heater) Power	Read Only
Pb_P	Proportional Band	5%
ArST	Auto Reset (Integral Time)	2.00 sec
rAtE	Rate (Derivative Time)	0.3 sec
biAS	Manual Reset (Bias)	25
SPuL	Setpoint Upper Limit	180 C
SPLL	Setpoint Lower Limit	0 C
OPuL	Output Power Limit	100%
Ct 1	Output 1 Cycle Time	8 sec (Norm=32)
bAL 1	Band Alarm 1	-
PhA 1	High Alarm 1	200
PLA 1	Low Alarm (Base Pressure)	-
AHY 1	Alarm 1 Hysteresis	1
PhA 2	High Alarm 2	174 C
AhY 2	Alarm 2 Hysteresis	1
Apt	Auto Pre-Tune	Disabled
PoEn	Auto/Man control Selection	Disabled
SPr	Setpoint Ramping	Disabled
rP	SP Ramp Rate value	OFF
SP	Process Pressure Setpoint	175 C
SLoc	Setup Lock Code	10

Note: Key parameters are listed in Red