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Keywords
Parylene-C, thinkness, uniformity, SCS Parylene Coater

Disciplines
Biotechnology | Nanoscience and Nanotechnology | Polymer and Organic Materials | Semiconductor and Optical Materials

Characterization and Optimization of Parylene-C deposition process using SCS Parylene coater

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Parylene-C has been deposited on bare Si wafers by physical vapor deposition using the SCS Coating Systems. Results show a 12 μm thick Parylene-C film with 10 g of dimer and negligible thickness variation across a wafer. We find a positive linear relationship between film thickness and mass of dimer at a range of 1 g to 18 g. However, the Al boat for dimer was burnt with 18 g of dimer, suggesting multiple depositions with 1 g to 10 g of dimer are recommended to achieve the Parylene-C film thicker than 12 μm.

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I. Introduction

Due to the increasing need for biomedical device and electronic coatings in nanotechology, the search and characterization for a biocompatible protective layer is an important area of research. Poly(p-xylylene), or "Parylene" shows hydrophobic surface property, relatively low gas permeability, transparency at wavelengths above 400 nm, relatively high tensile strength, high compliance and stability in the temperature range from -200°C to 200°C. These properties enabled Parylene to be an ideal barrier and protective layer for devices subject to moist or harsh environment such as in space, underwater, and inside the body.[1,2]

Parylene can be coated by physical vapor deposition process. Fig. 1 shows the SCS Parylene Coater tool and a schematic diagram of the machine. The depostion process begins by placing an amount of Parylene dimer into the sublimation chamber in front of the furnace. The dimer is then heated until it vaporizes, and the vapor moves into a higher temperature pyrolysis chamber where it is broken down into a monomer vapor. The monomer vapor migrates into a vacuum pressure, ambient temperature deposition chamber where it forms a conformal polymer coating as it comes into contact with surface area in the chamber[1].

The four types of commercially available Parylene are Parylene-N, Parylene-D, Parylene-HT and Parylene-C. Parylene-C differs in chemical structure from the other types of Parylene by containing one Cl atom in place of one of the aromatic hydrogen atoms in its benzene ring. Fig.2 shows the molecular phase changes of Parylene-C during the deposition process. This Cl atom in benzene ring leads to relatively lower permeablity to moisture and corrosive gases (such as O_2, N, and CO_2, and H_2) than other types of Parylene counterparts.[1] Common applications of Parylene-C are biomedical device coatings for stents, pacemakers, various implants, and multielectrode arrays.[3,4]

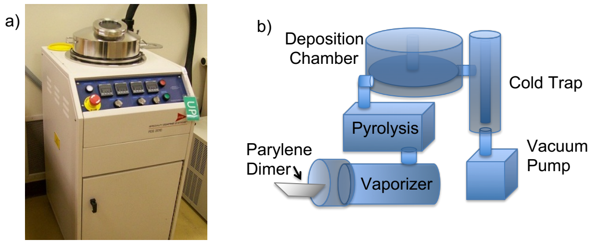

FIG. 1. a) The SCS Parylene Coater b) Simplified diagram of the SCS Parylene Coater (not to scale). The Parylene enters the vaporizer as a dimer and moves through the higher temperature pyrolysis chamber where is becomes a monomer vapor. The monomer vapor moves into the deposition chamber where it forms a polymer on the surface area of the chamber. A cold cylindrical "finger" traps excess vapor in the cold trap to prevent it from entering the vacuum pump.

While many studies carried out on electrical, mechanical and barrier properties of Parylene-C[1,2], the relationship between the amount of dimer and resulting coating thickness is less known. Robbins showed a positive linear relationship (exponent: 0.973) between dimer amounts and thickness for 0.1 g to 30 g of dimer for SCS Parylene Deposition.[5] However, the thickness-dimer amount relationship with different conditions and issues that can be concurrently happened during the process are still need to be studied. In this paper, we present the relationship between thickness and Parylene-C dimer amount and thickness uniformity, and suggest the maximum amount of dimer per batch.

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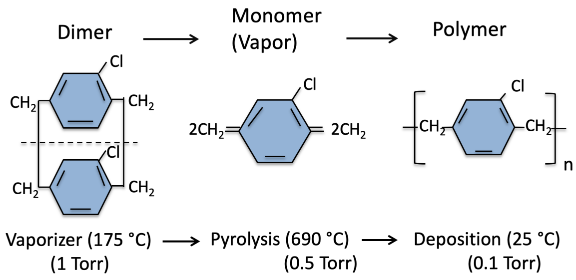

FIG. 2. Phase changes inside SCS Coating Systems as Parylene-C dimer changes to a monomer vapor and then a conformal polymer coating.

II. Experiment

Fig. 3 shows the process flow for coating each wafer and measuring thickness. First, a 2% micro-90 solution was spun on the bare Si wafer to create an anti-adhesive layer underneath the Parylene. This allows sections of the Parylene coat to be cut and peeled off after the deposition in order to measure the step height with the profilometer stylus. The wafer was placed in the deposition chamber of the SCS Parylene coater as shown in Fig. 4. Parylene-C dimer was weighed out on a high vacuum Al foil boat. The dimer amount was varied from 2 g to 18 g. The boat was then placed in the sublimation chamber.

The chamber was pumped down to vacuum pressure, a process that can take up to 15 minutes depending on how clean the cold finger is. The deposition process was run with vaporizer temperature at 175°C and pressure of 1 Torr, furnace temperature at 690°C and pressure of 0.5 Torr, and the deposition chamber at 25°C and pressure of 0.1 Torr. The process runs automatically once the “process start” button is pressed, and the furnace temperature automatically returns to room temperature when the process is finished. The chamber was vented and the sample taken out once the furnace temperature returned to below 100°C. Details of operating procedure are fully described in PENN nano SOP.[6]

Rectangular sections were carefully cut through the Parylene layer in the center and four edges of the wafer using a scalpel. Each section was carefully peeled off using tweezers under a microscope to check that excess pulling or stretching did not occur at the edges of the peel. Step height was measured using a KLA Tensor 2D/3D/Stress Profilometer. Five measurements of thickness (one from each cut section) were taken using 2D normal operation with an applied force of 1 mg, stylus radius of 2 μm, scan speed of 200 $\mu m/s$, and sampling rate of 100 Hz. The mean and standard deviation of the thickness measurements was calculated for each wafer.

III. Results and Discussion

A. Dimer amount - Parylene thickness relationship

Fig. 5 shows the mean thickness measured for each dimer amount. A positive linear relationship was found between dimer amount and coating thickness. The equation that best fit the data was $T = 1.1d{+}0.44$ ($R^2 = 0.98$) where T is Parylene-C coating thickness and d is amount of Parylene-C dimer. Whereas blue data points were obtained from recent deposition by us, red data points (3g, 3.5g, and 16g) were taken from previous depositions by other researcher for comparison.[7] The experimental conditions were identical with the exception of 16 g deposition. The 16 g was done in two steps of 8g depositions.

The positive linear relationship between dimer mass and thickness makes sense given that increasing dimer mass into the machine causes an increase in the amount of Parylene molecules in the vapor entering the chamber. Therefore, more Parylene is able to come into contact with surface area in the chamber leading to a thicker coating on all surfaces inside the chamber.

The thickness of Parylene coating at 10 g of dimer was 12.3 μm. This is almost two times thicker than the thickness Robbins obtained with the same amount of dimer.[5] Such a high deposition rate is presumably related to the actual surface area of loaded sample. Whereas we loaded a wafer without the wafer stand as shown in Fig. 4 (b), Robbins used a triple-story stand with full of samples in each story[5].The wafer stand increases the amount of surface area in the deposition chamber which might cause a decrease in coating thickness because more surface area is available per volume of monomer vapor for the vapor to polymerize onto.

B. In-wafer thickness uniformity

The variation in thickness of Parylene-C across the surface of each wafer was the order of sub-μm as listed in Table. I. The ratio of thickness variation to thickness decreases as the film thickness increases. This thickness variation is caused by relatively thicker section which is located close to the vapor entry hole for the vapor. As shown in Fig.4, the chamber contains a circular platform on which the samples are placed, and the opening where the vapor enters the chamber is near the outside edge of this platform. The platform rotates during the deposition process, causing one end of the wafer to always be closer to the entry hole for the vapor.

C. Optimum amount of dimer

A subsequent result of the experiment was that large dimer amounts (greater than 10g) created complications for the system. Two issues were observed during the 18g deposition. First, the bottom of the Al boat containing the Parylene dimer was burnt during the heating processes. This may lead to contamination of both the Parylene vapor and the polymer coating. Second, the vacuum pump had trouble reaching vacuum pressure during the next deposition. This might be caused by excess vapor failing to become trapped on the cold finger and entering

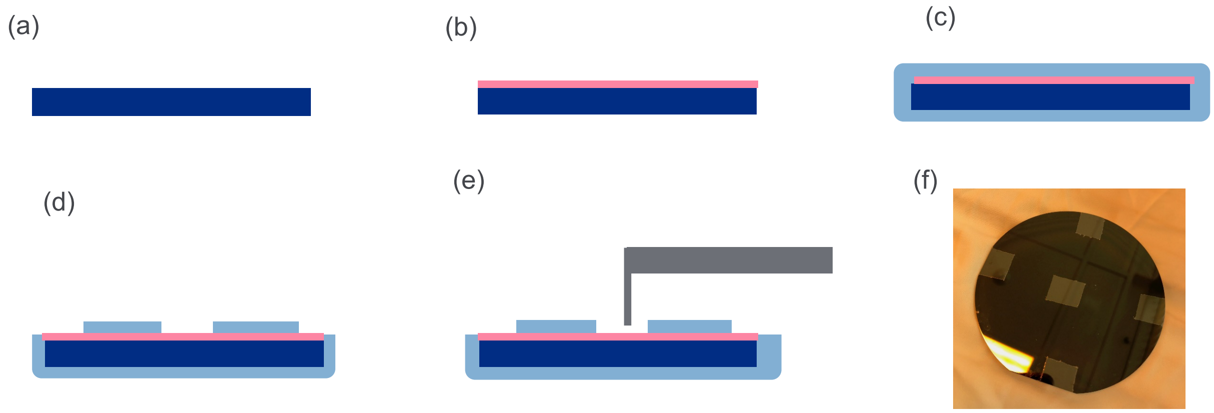

FIG. 3. (a) Blank Si Wafer, (b) Spin Coat 2% Micron90 solution, (c) Coat in Parylene-C, (d) Cut and peel of sections, (e) Measure the profile height at multiple (n=5) points across the wafer and (f) Deposited Parylene-C film.

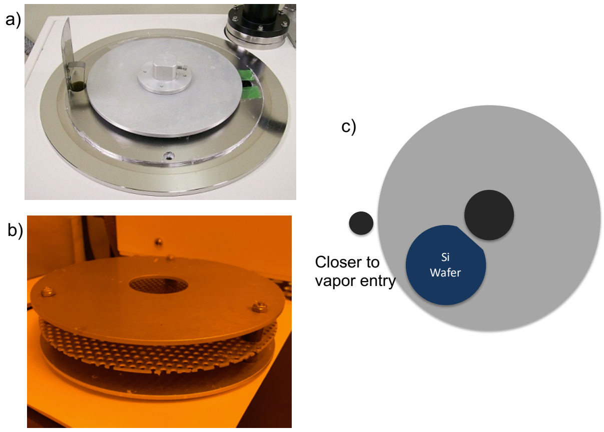

FIG. 4. (a) Rotating platform in the deposition chamber, (b) three tiered wafer stand which can be placed on top of the rotating platform in the deposition chamber to accommodate additional wafers, and (c) diagram of the deposition chamber platform; wafers were places with the flat edge facing the center of the platform.

TABLE I. Difference in thickness between sides of wafer facing inside the rotating platform and outside of the platform

Amount of Dimer (g)	4	6	10
Thickness (μm)	4.3	6.8	12.3
Thickness variation (μm)	0.39	0.55	0.20

the vacuum pump. Large depositions are recommend to be done in steps of smaller amounts (1 g to 10 g) to avoid these issues.

IV. Summary

This experiment found a positive linear relationship between Parylene-C dimer amount and thickness of the Parylene-C coating in the SCS Coating System. The thickness variation across the wafer surface was negligible. Subsequent results show that depositions requiring dimer amounts greater than 10g are recommended to be done in steps of smaller amounts (1 g to 10 g) to avoid complications caused by excess Parylene and contamination in the system.

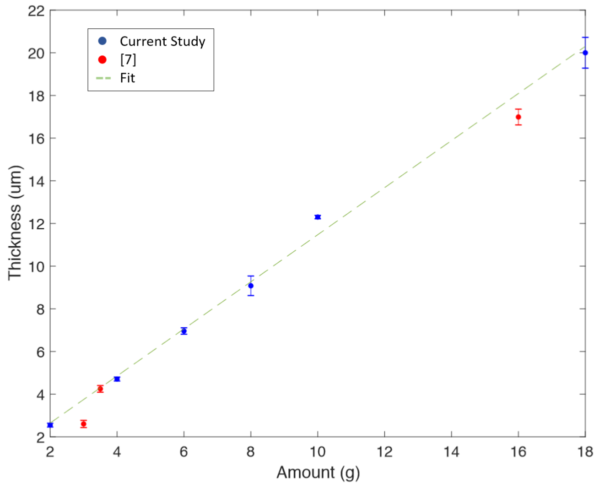

FIG. 5. Parylene-C coating thickness (μm) versus Parylene-C dimer amount (g). Blue data points show mean thickness of coating from experimental data. Red data points show mean thickness of coating collected from previous coatings.[7] The data has a linear fit with $R^2 = .98$

V. Acknowledgements

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