

Plasma pretreatment of polymers to optimize the adhesion of coatings

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Abstract— Parylene-C is used for various biomedical devices because of its high conformity and biocompatibility. However, delamination could occur because of low adhesion between Parylene-C and other materials. For this experiment 4-inch Si-wafers are used. Parylene is deposited by chemical vapour deposition (CVD). Silane A-174 as adhesion promoter is added between Parylene-Parylene layers. The samples are soaked at different duration 1 h, 24 h and 120 h in PBS solution at 37 °C. A peel test is further performed to investigate the adhesion properties of the samples.

I. INTRODUCTION

Poly(para-xylene) better known as Parylene is a semi-crystalline, hydrophobic polymer. Thin conformal and pinhole-free layers are achieved with chemical vapour deposition (CVD). The chemical structure of Parylene C (Figure I) has a chlorine atom on its benzene ring and is known for its chemical inertness, resistivity, low moisture permeability and biocompatibility. Further properties are optical transparency, mechanical flexibility, and compatibility with a number of standard micromachining processes. Parylene is often used in combination with rigid substrates such as silicon or glass. More and more usage in the promising field of polymer-based biomedical microelectromechanical systems (MEMS), is becoming increasingly popular as a flexible structural material [1].

For avoiding foreign body response, it is necessary to prevent water permeation into the implanted electronic devices and avoiding diffusion of corrosive products into the tissue. That is why it is crucial to encapsulate the devices, which are implanted into the body. A commonly used material is Parylene because of good encapsulation properties. It is also known for high biocompatibility and good barrier property, which makes it ideal for encapsulating active implants. However, Parylene is suffering from poor adhesion properties. Weak adhesion in implants can cause tremendous failure such as electrical shorts [3,4,7].

In the course of this work, the pre-treatment method with silane A-174 on Parylene-C is used to investigate the adhesion properties at different durations in PBS solution. The adhesion properties of the samples are measured and evaluated with the peel test. In the pre-treatment, the standard adhesion promoter silane A-174

is used to improve adhesion. This chemical linker binds to the OH coupling group of the oxidized substrate by forming hydrogen bonds and subsequent covalent bonds.[5]

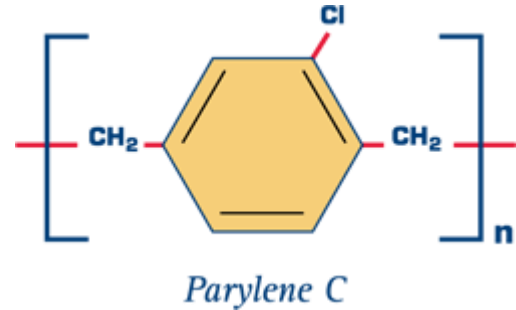


Figure I: Chemical structure of Parylene-C [2].

II. MATERIALS AND METHODS

In the following chapter, equipment, optimizations, the preparation of the samples and the peel test are described.

A. Equipment

For measuring the adhesion properties of the samples, the tensile testing machine FMT-313.B5 from the company Alluris is used. It is capable of measuring loads up to 50 N, which is enough for our purpose of use.

B. Optimization of the setup

The setup for the 180° peel test was optimized. For this purpose, a slightly modified and optimized fixture for the tensile testing machine was designed (Figure II). This newly designed fixture allows a more comfortable clamping than the previous one. To achieve this, a fixture was realized by means of 3D printing. It has a bolt which is used to open and close the clamping jaws. The fixture is easy to unscrew from the tensile testing machine, which allows easy clamping of the samples.

C. Preparation of wafer

For this experiment 4-inch Si wafers are used as substrates. Cleaning of the wafers is essential, before these can be coated. This ensures that there are no residues on the wafer that could later affect the evaluation. The wafers must undergo a specific cleaning process. First, the wafers are placed in acetone, then in propanol and in the last step in deionized water for 15 minutes each in an ultrasonic bath. The excess liquid on each wafer, is blown dry with nitrogen gas.



Figure II: Tensile testing machine with optimized fixture.

D. Coating and pre-treatment

The cleaned wafer is then coated with the first layer of Parylene-C by using approx. 15 g of Parylene from the company Plasma Parylene System GmbH. The amount of Parylene allows the deposition of 13 μm on the wafer. After the first Parylene-C layer, the Kapton DuPont tape is placed using a special technique, which will be explained in more detail in the next section. Then the second Parylene layer is deposited. The adhesion promoter is applied between the two Parylene layers by CVD. For this purpose, 0.1 ml of Silane A-174 is used.

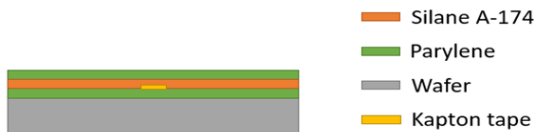


Figure III: Schematic order of the coating.

E. Kapton tape attaching technique

Kapton tape has excellent chemical resistance with consistent mechanical properties besides having a very good dielectric strength even at high temperatures. This makes it suitable for these experiments.

In the first tests, Kapton tape with thickness of 60 μm was used, which caused the samples to break off at the

transition point during testing. When using a thinner Kapton tape with a thickness of 12 μm , the sample did not break off at the transition point at any time.

The Kapton tape is placed after the first layer of Parylene-C. In order to separate the layers from each other, there is a special application technique for the Kapton tape. The Kapton tape is attached to the wafer with a specific arrangement, by using two different sized Kapton tapes.

First, the wider Kapton tape (thickness: 12 μm , width: 15 mm) is placed in the centre of the wafer with the sticky side down, then the smaller Kapton (thickness: 12 μm , width: 8 mm) is placed in the centre of the wider Kapton tape with the sticky side up, after this step, two Kapton strips are placed with the sticky side down so that the small Kapton tape is enclosed, by overlapping of the two Kapton tapes. This technique is important to separate each Parylene layer to clamp it in the tensile testing machine later.

F. Cutting out samples

The fully coated wafer is placed in a template to prevent the wafer from slipping when cutting out the samples.

First, it is cut longitudinally to the Kapton tape, exactly where the thin Kapton tape is located. The second cut is made horizontally to the Kapton tape in 2-centimetre-thick strips (Figure IV). 10 samples are obtained from one wafer. It is important to cut around the edges of the wafer for easier peeling later. Since the thin Kapton was placed with the sticky side up, it is now possible to pull the two Parylene layers apart at this point.

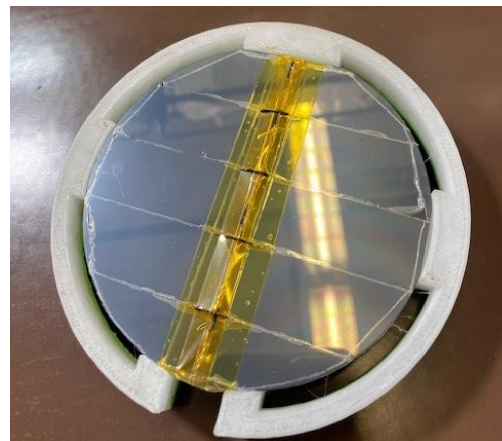


Figure IV: Coated wafer in the cutting template.

G. Detaching samples off the wafer

The wafer is soaked in distilled water for approx. 1-

2 hours to make it easier to pull off the samples from the wafer. The samples can be then pulled off more easily with the assistance of tweezers.

H. Soaking in PBS

The samples taken from the wafer are then placed in phosphate buffered saline (PBS) for soaking. The solution has a PH of 7.4 and is stored on a hot plate at a constant temperature of 37 °C, as in the human body. This allows the samples to be tested for biostability and flexibility as in the body.

I. Peel test

The adhesion strength is determined with the 180° peel test. One end of the sample is connected to the holder on the table, the other end to the load cell which is pulled at an angle of 180 ° [4]. The samples are slightly pulled apart at the Kapton so that they can be clamped in the holder. The other half of the samples is clamped into the holder on the tensile testing machine. The peel speed is 10 mm/min, and 9121 points are recorded per sample.

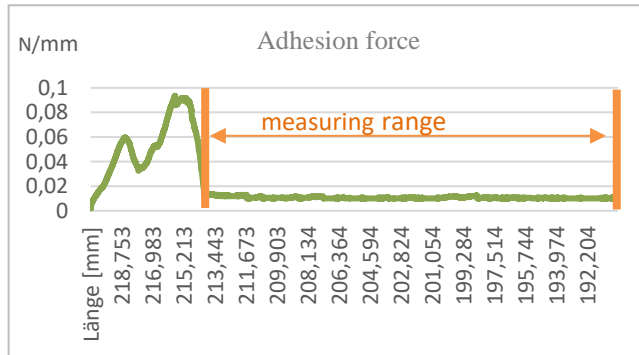


Figure V: Diagram of the adhesion strength of Parylene-Silane Parylene Coating. Showing the measuring range. The X-axis shows the length that the tensile testing machine has travelled.

III. RESULTS AND DISCUSSION

The samples are analysed at different soaking times in PBS solution, and were evaluated once for 1 h, 24 h and after 5 days. In comparison with the results of S.Strohe and J.Buda [6], the transition point Kapton-Parylene is not cracked at any time of the experiment. This means that the difference in thickness between Kapton and Parylene in the test was too high. With the use of the thin Kapton tape, the problem is no longer present. The Kapton tape did not break at any time at the Parylene- Kapton transition point. Measurements were possible after 1 hour duration in PBS. When evaluating the measurements, two peaks are visible (Figure V), this represents the force from the Kapton to

the Parylene and is excluded from the calculations. The output of the adhesive strength is given in N/mm.

As shown in the table the adhesion strength Parylene-Silane-Parylene is highest after 1 h duration in PBS with 20.3 mN/mm. The adhesive strength is decreasing with a longer duration in PBS, after 24h the mean adhesion force is 19.95 mN/mm and less after 120h, it is 15.0 mN/mm.

Table 1: Average adhesion force and standard deviation of Parylene-Silane-Parylene at different duration in PBS solution.

Parylene-Silane-Parylene		
Duration in PBS	Average Adhesion Force	Force
1 h	20.3 ± 2.0	
24 h	19.95 ± 0.9	
120 h	15.0 ± 0.8	

IV. CONCLUSION & OUTLOOK

In this work, the adhesion strength was investigated with silane pre-treatment, at different exposure in PBS solution. By using a thinner Kapton tape, the samples did not crack at the Kapton-Parylene interface. An overview of the values can be seen in Table 1. A decrease in adhesion with time in PBS solution can also be seen. These values are as expected. With longer PBS duration, a decrease in adhesion is observed. The difference between 1h and after 24 h in PBS solution is not so large compared to 24 h and 120 h exposure time. Nevertheless, further measurements with longer exposure times in PBS should be performed and evaluated to obtain a reliable result.

A perspective for future work could be the investigation of different adhesion promoters, such as titanium oxide, or Aluminium oxide. In order to be able to make an informed comparison as to which adhesion promoter is most suitable.

V. REFERENCES

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