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Stiction of Parylene C to Silicon Surface Measured using Blister Tests

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***Abstract* – Micro-fabricated biocompatible check valves are integral parts of many implantable micro-fluidic devices. The cracking pressure of check valves is usually controlled by stiction between polymeric films and the underlying substrate. The following paper presents the first comprehensive study of stiction between parylene and silicon surfaces. The valves are fabricated using surface micromachining with parylene C as the structural material. Deep Reactive Ion Etching (DRIE) is used to create through holes in the wafer for the passage of fluids. Blister test is employed to calculate stiction. From experimental results, stiction between parylene C and silicon surfaces is found to be 2.59 J/m^2, which is comparable to the stiction between silicon and other polymeric thin films.**

Keywords – stiction; parylene; blister test; check valve;

I. INTRODUCTION

Micro or Nano electromechanical systems are employed ubiquitously in the field of micro-fluidics. In order to regulate the flow of fluids in such systems, check valves are required [2]. Implantable MEMS check valves often use parylene C as the structural materials because of the biocompatibility and flexibility of the polymer [5]. Parylene check valves are usually fabricated on silicon substrates and make up crucial parts of implantable micro-fluidic actuating devices [6]. The operation of a micro-fabricated biocompatible check valve is largely defined by its cracking pressure, usually controlled by stiction between polymeric thin films and the underlying silicon substrate. In order to understand their principles of operation and design parylene check valves with specific cracking pressures, study of surface stiction between parylene and silicon is necessary.

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Stiction is an attractive force that occurs between free standing surface micro-machined features and the substrate after the release of the sacrificial layer [9]. Stiction is often an undesirable phenomenon for surface micromachining because of its detrimental effect on free standing microstructures, causing them to be attached to the substrate (Fig. 1). Attempts have been made to reduce stiction. For example, a surface treated valve with Cr/Au coating and O_2 plasma showed cracking pressure of 20-40kPa [8]; the cracking pressure of a polyimide check valve with C_4F_8/Ar non-stiction coating changed from 210kPa to 59kPa [3]; and SAM (self-assembled monolayer) is also used to reduce stiction [7]. For parylene check valves, since stiction governs the cracking pressure of the valves, it must be controlled instead of blindly reduced. Parylene check valves are particularly sensitive to the effects of stiction because parylene is a soft polymeric material that is extremely non-rigid.

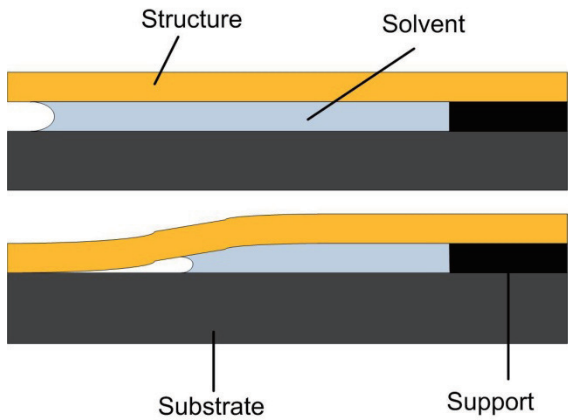

Fig. 1 | Stiction pulls free standing microstructures toward the substrate surface during release

The current study investigates stiction between parylene and silicon surface quantitatively using the blister test. Fabrication of the devices is done using standard MEMS surface micromachining techniques. For on-bench testing, blister test is employed because it allows direct testing on actual valve structures, making the experimental results more applicable to other devices when similar structures are involved. Different recipes for releasing and drying the parylene check valves are used to examine their effects on stiction.

II. THEORY

Blister test is usually performed on devices where a thin film covers the substrate. Through holes are required to allow pressure to be directly applied to the film from the back side (Fig. 2). For parylene check valves, when pressure is applied, plastic deformation occurs in the parylene film that causes it to bulge to a distance d that is dependent on the Young's modulus, Poisson's ratio, geometry of the substrate opening, and thickness of the parylene film.

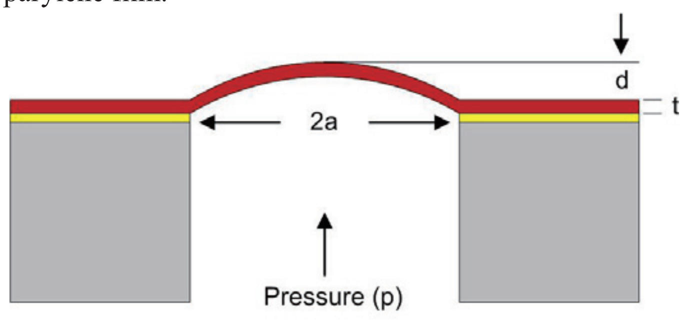

Fig. 2 | Side view of theoretical blister formed from parylene C film during experimentation

Due to the circular via in the silicon, it can be assumed that the blister has a semispherical profile. With this assumption, the stiction for can be calculated using equations (1) and (2) [1].

$$p_c = \frac{3.56Et}{a^4} {d_c}^3 + \frac{4\sigma_o t}{a^2} d_c \quad (1)$$

$$\gamma = 2.22Et\left(\frac{d_c}{a}\right)^4 + 2.00\sigma_o t\left(\frac{d_c}{a}\right)^2 \quad (2)$$

Where p_c is the critical debonding pressure, d_c is the maximum vertical displacement of the parylene film, E is the elastic modulus of parylene C (~ 4 GPa), t is the thickness of the parylene film (3 µm), α is the radius of the blister (100 µm), and γ is the stiction between parylene and silicon. The constant σ_o represents residual stress within the parylene film. For this particular experiment where parylene is annealed at 100°C, 37.8 MPa is used as the residual stress [4]. As pressure inside the blister exceeds the critical pressure p_c, parylene film debonds from silicon. When this event occurs, p_c is used to calculate γ.

III. DESIGN AND FABRICATION

Parylene check valves are fabricated according Fig. 3. First, 1 µm of oxide is grown on both sides of a silicon wafer. 300 µm diameter holes are partially etched on the back side using Deep Reactive Ion Etching (DRIE). Then, photoresist is applied to the front side and patterned to define the check valves' seat area. 3 µm parylene is deposited on the front side of the wafer using Chemical Vapor Deposition (CVD) and is patterned with oxygen plasma. DRIE is used again to complete the backside holes.

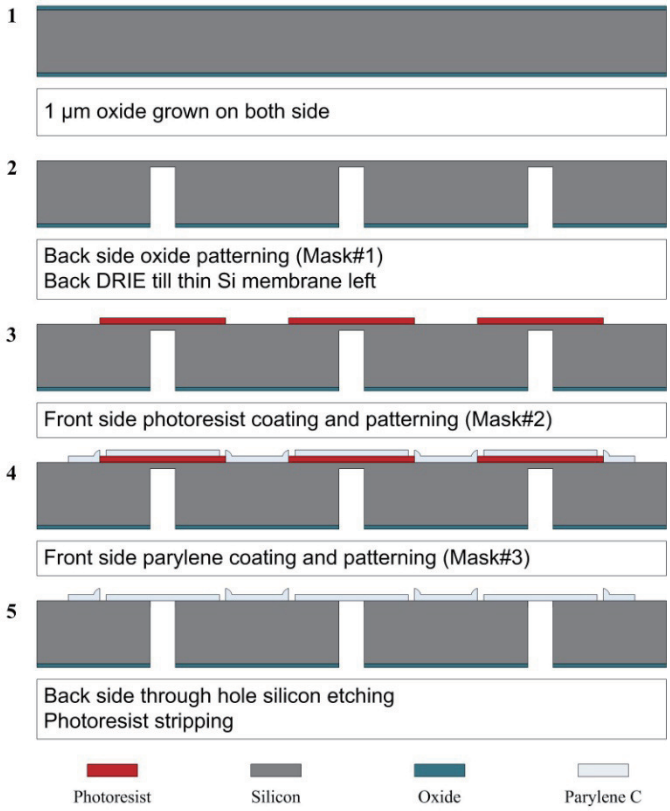

Fig. 3 | Process flow of micro-fabricated parylene check valve for stiction characterization using blister tests

After dicing, dies are released using two different procedures. Sacrificial photoresist is stripped using ST-22 and cleaned with acetone. Then, some valves are soaked in isopropyl-alcohol (IPA) and water to remove all organic molecules. These valves are then air dried. Other valves are placed in a mixture of acetone and silicone oil overnight, after which the dies are air dried as well. The dried devices are then used in blister tests to determine its critical debonding pressure. Released devices (Fig. 4) are used for blister test.

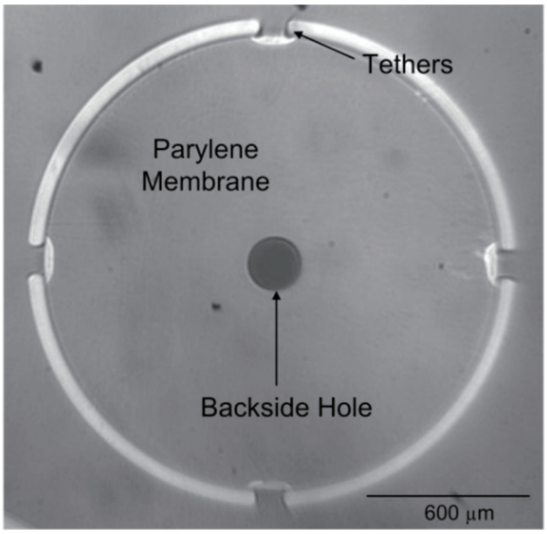

Fig. 4 | Top view of the finished parylene check valve ready for blister test

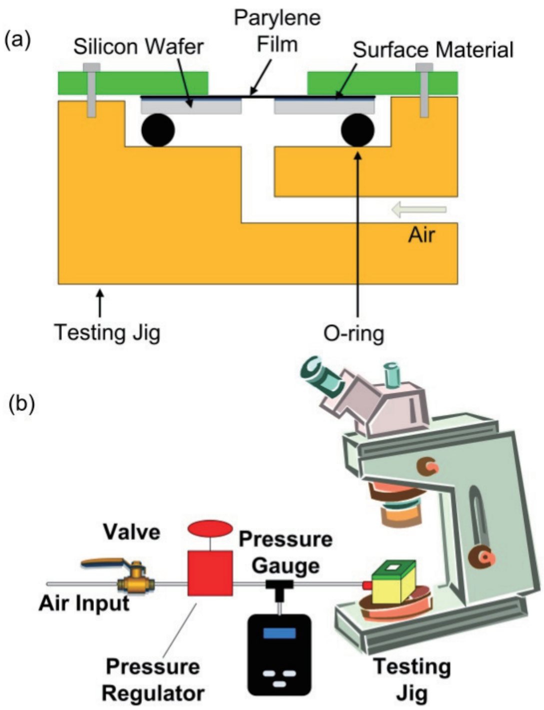

Fig. 5 | Experimental Setup (a) side view of the test jig (b) depiction of test instrumentation used

During the experiment, each die is placed in a testing jig that allows fluid (N_2 gas) to apply pressure to the parylene membrane. The jig is then connected to a fluidic setup consisting of a valve, a pressure regulator, and a pressure gauge (Fig. 5). The setup is placed under a microscope for observation. Pressure inside the tubing is gradually increased by adjusting the pressure regulator. The pressure gauge reads out the current pressure inside the blister. The critical pressure is recorded when debonding occurs.

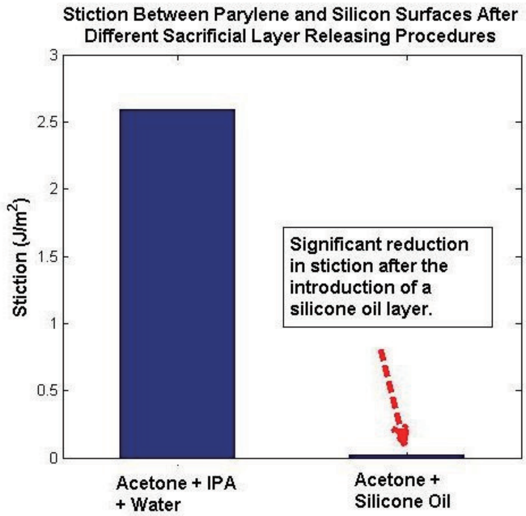

Fig. 6 | Average stiction measured from blister tests

Release Procedure	Acetone + IPA + water	Acetone + silicone oil
Critical Pressure (kPa)	424	30.9
Stiction (J/m^2)	2.59	0.019

Table 1 | Table outlining critical pressure and stiction between parylene and silicon surfaces after different releasing procedures

IV. RESULTS AND DISCUSSION

Figure 6 and table 1 illustrates the results of the stiction experiment. When parylene check valves are released using ST-22, acetone, IPA, water, and allowed to air dry, results from blister tests show that significant stiction exists between the parylene membrane and the silicon surface. Each die tested contains eleven valves. After testing several dies from different parts of the wafer, the stiction between parylene film and the silicon substrate surface is determined to be 2.59 J/m^2, corresponding to an average critical pressure of 424 kPa or 62 psi. Variations in stiction across different samples can be attributed to the non-uniformity of the fabrication process. The relatively large stiction can be explained by possible surface chemistry between water and the two materials. When sacrificial photoresist is removed via wet etching, water occupies the cavity previously occupied by the photoresist. Water molecules tend to hydrolyze the dangling bonds on the surfaces of silicon and parylene. As water dries, hydrogen bonding pulls the parylene film toward the silicon surface. After drying, due to the proximity of the parylene and silicon surfaces, Van der Waal's force cause the two materials to remain attached.

When valves are soaked in a mixture of silicone oil and acetone after photoresist removal, both acetone and silicone oil occupies the area previously occupied by sacrificial photoresist. When these devices are air dried, acetone vaporizes. However, silicone oil, which does not easily vaporize at room temperature, is trapped between parylene and silicon. Experimental results show that the stiction associated with these devices is much smaller, usually on the order of 19 m J/m^2, corresponding to a critical pressure of only 30.9 kPa or 4.9 psi. The significant reduction of stiction can be attributed to two factors. Air drying after soaking in an acetone mixture prevents contact of the surfaces with water. Very few of the dangling bonds on the surface may be hydrolyzed. In addition, due to its hydrophobic character, silicone oil most likely forms a thin layer separating the two surfaces. Thus, parylene and silicon surfaces never come close enough during drying to result in permanent attachment.

The data shows that the magnitude of stiction that results from drying has to do with how close the parylene and silicon surfaces approach each other during the drying process. Mechanisms that can reduce their proximity will likely reduce stiction. An additional note is that the stiction between parylene and silicon is small compared to the adhesion strength between the two materials after direct vapor deposition. Such a high surface bonding strength is a desirable characteristic since it implies that a large pressure can be applied to the parylene valve membrane without risking debonding of the anchors.

V. CONCLUSION

Stiction between thin film parylene and silicon substrate has been studied. Devices with check valve configurations were fabricated and released using different procedures. After performing blister tests, stiction was quantified. The results are consistent with other studies performed on thin polymeric films such as polyimide [3]. Experiments show that mechanisms that can reduce the proximity between parylene and silicon surfaces during drying will likely reduce stiction since stiction results from the hydrophobic interaction between silicon and parylene surfaces due to proximity. The results of this study can be used to design parylene check valves with different cracking pressures by simply changing the proportions of silicone oil in acetone in the procedure.

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