

A Line Force Model to Measure the Strength of Polydimethylsiloxane (PDMS)-to-PDMS Bonding Using Blister Tests

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ABSTRACT

A method based on a blister test has been employed to study the strength of PDMS bonding. The blister consists of a PDMS membrane bonded to a PDMS substrate while a circular region in the center remains un-bonded as a blister. When the blister is pressurized, a force normal to the substrate pulls the membrane from the substrate and causes the delamination. The ruptured samples from the blister tests are examined to realize the failure mechanism. The bonding strength is defined not only by the critical delamination pressures but also the corresponding line forces, which provide us a more general means to characterize the PDMS bonding strength.

Keywords: Polydimethylsiloxane (PDMS), Bonding, Blister Test, Line Force.

1 INTRODUCTION

Micro-Electro-Mechanical Systems (MEMS) traditionally represents an approach using surface and bulk micromachining techniques to implement tiny but sophisticated devices. MEMS-based devices, by their nature are confined in simple two dimensional structures as they mostly are made by etching and thin film deposition processes. To create three dimensional structures and devices for broader applications, one of the important techniques is to bond two or more laminated structures to each other. Especially, bonding two pieces of polymeric material, polydimethylsiloxane (PDMS), to make micro devices has been widely used in micro fluidic sensor and actuator applications [1-3]. A robust bonding is usually required to sustain the internal pneumatic pressure from fluid in the devices, to prevent interfacial failures during operation, and to provide large pneumatic actuation forces. Thus, the capability and performance of the devices are highly dependent on the bonding quality.

Bonding quality conventionally are characterized using tensile test, shear test, and peel test, which are not suitable for characterizing the bonding of polymeric materials due to the flexibility of the samples [4]. A simple technique has been used by comparing PDMS bonding strength to optimize the bonding process conditions [5]. However, this

technique used an objective representation to quantify the bonding strength.

Meanwhile, blister tests provide a close approximation of the bonding failure modes experienced under field-used situations and have been extensively used for other bonding strength measurements. The blister test was first introduced by Dannenberg, which consisted of two adherends bonded to each other while a circular region in the center remains un-bonded as a blister [6]. The diameter of the blister remained the same until a critical pressure. Upon the critical pressure was approached, the boundary of the blister starts to dramatically propagate and the adhesive fractures occurred along the interface of the two adherends [4]. However, because the sample preparations are complicated and the major efforts are required in data analysis, only a few related research effort has been devoted to qualitatively and quantitatively characterize the PDMS bonding strength, particularly for the bonding which is used in making micro-fluidic devices [7]. This research effort solely characterized the PDMS bonding strength in terms of the delaminating pressures, which depends on not only bonding conditions but also blister sizes. When the characterization was performed in a certain size of blisters, they lacked dimensional analysis and could not be applied to general PDMS-based MEMS devices in different dimensions. A simple and generalized methodology, by which bonding strength measurement only depends on process conditions, therefore, is highly desirable.

2 EXPERIMENTAL DETAILS

Our bonding-strength measurements utilized a blister test, which consisted of a PDMS membrane, bonded to a PDMS substrate [8]. The substrate has a relatively thick thickness of 10 mm, compared to 75 μm in the membrane, to ensure negligible bending or deformation during blister tests. PDMS surfaces were treated by oxygen plasma that converted the methyl groups on the surface to hydroxyl (-OH) groups and allowed siloxane covalent (Si-O-Si) bonds to be formed when two plasma-treated PDMS surfaces were brought into contact. Adapted from the technique in [5], a circular PDMS slab, as a plasma mask, was placed upon the PDMS substrate to shelter the plasma during

plasma treatment process, so a corresponding circular unbonded area can be later created as the blister. The inherent adhesion between two PDMS surfaces was good enough to ensure hermit contact and prevent plasma exposure. No siloxane bonding therefore was formed in the circular area, which was inflated later when air pressure was gradually applied.

The experimental setup of the blister tests was illustrated in **Figure 1**. The pneumatic connection includes an air compressor, an electro-pneumatic controller and tubing with a pipette tip to the testing PDMS sample. A digital signal from a function generator was transmitted to the pneumatic controller and a LED beside the sample. The images of the blister and the LED were recorded from the top to show the blister deformation and the applied pressure simultaneously, which allowed real-time pressure monitoring in the following image process step.

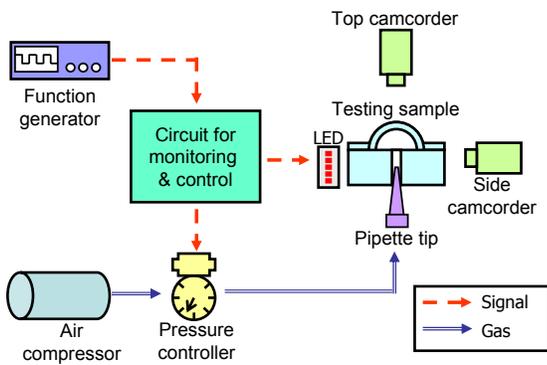


Figure 1: The Experimental Setup

2.1 Image Process

In order to understand how the blister was deformed and delaminated, an image process protocol has been developed. A software, ICARUS has been employed to calibrate and reconstruct the image sequences as well as to track the change of the blister [9]. To identify the blister boundary, four pixels were thus chosen around the blister boundary based on the RGB colors and luminance of the pixels as shown in **Figure 3**. Their coordinates were located via ICARUS frame by frame from the recording images to track the propagation of the blister delamination. The average diameter of the blister, meanwhile, can be accordingly estimated by using the least square approximation.

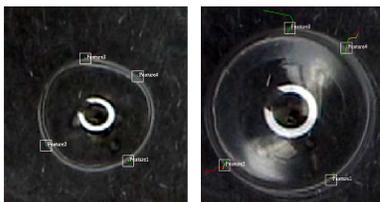


Figure 2: Four pixels were chosen along the blister boundary based on the RGB colors and luminance of the pixels. The images are the blister before delamination (left) and in delamination (right).

Sequences of blister images were therefore analyzed. The relationship between applied pressures and blister diameters was described in **Figure 3** for the sample in ideal and practical situations. Ideally, the blister diameter should remain constant when the pressure was applied from zero (point *A*) to the critical pressure (point *B*). Beyond the critical pressure, the delamination began and the interfacial failures propagated, resulting in the diameter increase of the blister until the delamination reached the edge of sample (point *C*). In practice, the blister diameter increased slowly even before the critical pressure was reached (from point *A'* to *B'*). It was because the blister deformations caused the change of light reflection on the blister, which made us difficult to determine the onset of the interfacial failure. A 5% increment of the original diameter, from points *A'* to *B'*, was employed as the criteria to define the beginning of delamination. Meanwhile, the corresponding pressure at point *B'* was considered as the critical pressure.

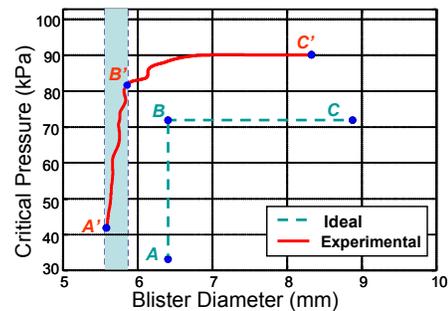


Figure 3: The relationship between blister diameter and pressure throughout a blister test. The blister was (*A* and *A'*) initially inflated, (*B* and *B'*) about to be delaminated at the critical pressure, and (*C* and *C'*) started to be delaminated.

2.2 Interfacial Fracture due to Delamination

Fractured samples from the blister tests, including PDMS membrane and substrate, were examined. A SEM picture in **Figure 4** revealed the fracture mechanism during the delamination in the blister tests. No fractures were found at the blister area. Only fracture patterns due to tensile stress were observed around the blister boundary. These fracture patterns suggested the PDMS membrane was initially pulled by a tensile force to detach from the substrate vertically, making the blister delaminated when the pressure reached the critical value. On the other hand, several shear ruptures were found further away from the

blister boundary; and they were on planes inclined at about 45 degrees to the radial direction of the blister, or the propagation direction of delamination. These shear fracture patterns showed the shear stress caused the delamination at the later stage when the blister failures propagated.

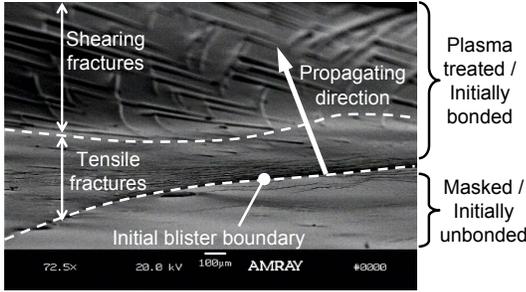


Figure 4: A SEM picture shows the interfacial fractures of PDMS bonding during blister tests.

3 ANALYTICAL MODEL

The thin PDMS membrane was assumed isotropic and uniformly deformed during the experiments. The ratio of membrane thickness to blister diameter was small (i.e. $\ll 0.1$) so that the thin shell theory can be applied [10]. The free body diagram in **Figure 5** showed the forces acting on the PDMS membrane and the PDMS substrate when the blister was inflated by the pressure (P).

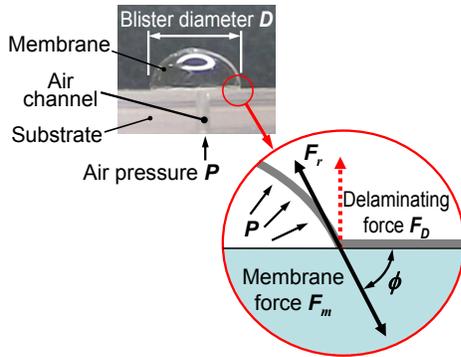


Figure 5: An analytical model: A force model described that the delaminating force, which was balanced by the membrane force, pulled the membrane to separate from the substrate and defined the bonding strength.

The force (F_r) due to the applied pressure was balanced by the membrane force (F_m); the equilibrium was expressed by:

$$P \cdot \frac{\pi D^2}{4} + F_m \cdot \sin \phi \cdot \pi D = 0 \quad (1)$$

The vertical component of F_r , denoted as F_D , pulled the membrane at the perimeter of the blister apart from the substrate. In other words, what determined the occurrence

of delamination were the force F_D and the blister perimeter. Thus, the bonding strength can be described by F_D in force per unit length (i.e. line force) rather than pressure.

$$F_D = F_r \cdot \sin \phi = -F_m \cdot \sin \phi = \frac{PD}{4} \quad (2)$$

Once the critical pressure was found, the corresponded line force ($F_{D,cr}$) could be calculated as follows:

$$F_{D,cr} = \frac{P_{cr} D_{cr}}{4} \quad (3)$$

The critical line force of the force per unit length was then used to define the bonding strength in our model.

4 RESULTS AND DISCUSSION

A series of blister tests under the same bonding conditions were conducted. PDMS samples were bonded using oxygen plasma treatment at 150 mtorr and 75 W for 10 seconds. All blister samples with a variety of dimensions were fabricated using the plasma-masking techniques. The experiments were recorded by a camcorder and studied using our image processing protocol, as described previously. The critical pressures and the critical line forces for every PDMS blister sample were obtained and calculated as shown in **Figure 6**. The data points in square marks represented the critical pressures, while the ones in cross marks represented the critical line forces. The critical pressures showed an inversely-proportional relationship to the blister diameters while the critical line forces had similar values. Thus, the critical pressure is a size-dependent quantity and can only describe the device performances at a particular dimension. However, the critical line force can represent the bonding strength, which is independent to the sample size and should be only affected by the parameters of bonding situations.

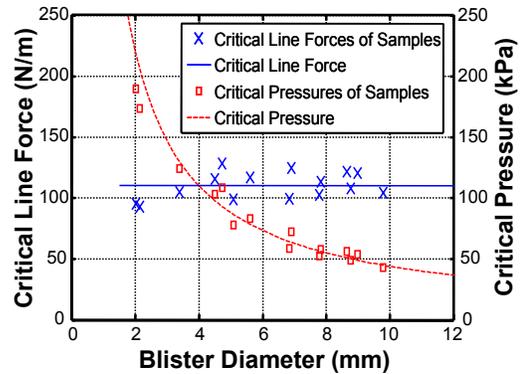


Figure 6: The relationship of critical line forces and pressures in different blister diameters. The bonding conditions for oxygen plasma treatment were controlled at 150 mtorr, 75 W for 10 sec.

5 CONCLUSIONS

We have developed a methodology using a blister test and a line force model to characterize the PDMS bonding strength. The PDMS samples were bonded using oxygen plasma and the blister areas were defined by the plasma-masking technique. An image process protocol and ICARUS have been adapted to realize the diameter change of the blisters. The fracture mechanism of blister delamination has also been studied, showing the initial failure was due to tensile stress. The relationship between critical pressure and critical line force versus blister size was obtained. It suggested that the line force can be a measurement parameter, which is sample size independent and only determined by the bonding conditions.

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