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Evaluation of bonding between oxygen plasma treated polydimethyl siloxane and passivated silicon

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Abstract. Oxygen plasma treatment has been used extensively to bond polydimethyl siloxane to polydimethyl siloxane or glass in the rapid prototyping of microfluidic devices. This study aimed to improve the bonding quality of polydimethyl siloxane to passivated silicon using oxygen plasma treatment, and also to evaluate the bonding quality. Four types of passivated silicon were used: phosphosilicate glass, undoped silicate glass, silicon nitride and thermally grown silicon dioxide. Bonding strength was evaluated qualitatively and quantitatively using manual peel and mechanical shear tests respectively. Through peel tests we found that the lowering of plasma pressure from 500 to 30 mTorr and using a plasma power between 20 to 60 W helped to improve the bond quality for the first three types of passivation. Detailed analysis and discussion were conducted to explain the discrepancy between the bonding strength results and peeling results. Our results suggested that polydimethyl siloxane can be effectively bonded to passivated silicon, just as to polydimethyl siloxane or glass.

1. Introduction

In microfluidic applications, polydimethyl siloxane (PDMS) is a popular type of polymer used in the rapid prototyping of microfluidic systems [1,2]. There are several advantages of using PDMS including fast fabrication time, compatibility to a variety of biological and cellular applications due to the high gas permeability of PDMS, ease of installation of fluidic interconnects from the macroworld to the microfluidic device due to the elastomeric nature of PDMS, and so on [3]. In addition, a thin layer of PDMS can also be used as a capping layer for microfluidic devices as it is optically transparent down to 280 nm and thus suitable for a number of fluorescence or absorbance detection schemes [1]. The bonding quality of the PDMS capping layer is critical in microfluidic applications which in general are designed to process very small amounts of fluids. Consequently, this means that the fluidic pressure exerted in the fluidic channels are inversely proportional to its cross-sectional length scale by four orders [4]. Without a good seal, leakage of fluids will inevitably occur due to a breakdown of the bonding between the two layers.

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Oxygen plasma treatment has been used by many researchers to create an effective bonding of PDMS to PDMS or glass [5,6]. Plasmas can improve adhesiveness by removing surface contaminants, introducing roughened bonding surfaces and reactive chemical groups. In particular, the $–O\text{-}Si(CH_3)_2$- unit in PDMS can be converted to silanol group (–OH), thus the PDMS surface chemistry changes from hydrophobic to hydrophilic. However, insufficient reports exist for the optimum bonding conditions between PDMS to silicon-based substrates. In this paper, we describe the results on a study of the bonding of plasma activated PDMS to passivated silicon. The passivation layers include PECVD phosphosilicate glass (PSG), PECVD undoped silicate glass (USG), LPCVD silicon nitride (Si_3N_4) and thermally grown silicon dioxide (SiO_2). After the completion of the bonding process, the bond strength is evaluated qualitatively using a quick, manual peel test [7,8] as well as quantitatively using a mechanical shear test. Our results indicate the bond quality is generally enhanced by using low values of plasma pressure and power.

2. Materials and methods

2.1. Experimental materials

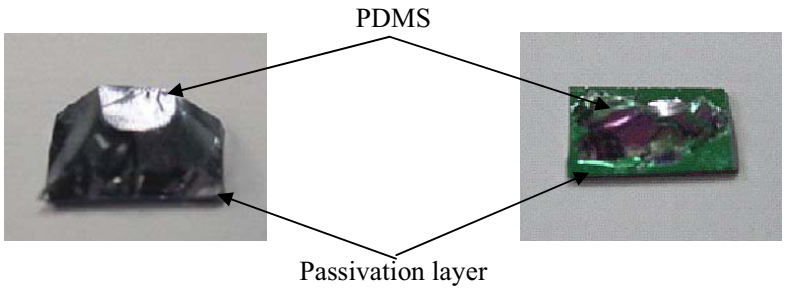
Samples (1×1 cm^2) were prepared with bare Si wafers that were coated with different passivation materials. The bonding surface of the silicon samples was cleaned for 10 min using Piranha solution (98 % H_2SO_4 : 30 % H_2O_2 at 6 : 1 v/v) that has been pre-heated to 100 ˚C and rinsed using de-ionized water before blow-drying using nitrogen.

PDMS (SYLGARD 184, Dow Corning Corporation, Michigan, USA) was prepared by thoroughly mixing the base and curing agent using a 10:1 weight ratio, degassed and then poured into a plastic mould. This was cured using a vacuum oven (Model 280A Vacuum Oven, Fisher Scientific, Pittsburgh, USA) for one hour at 80 ˚C. Once cured, the PDMS was peeled from the mould and divided into pieces using a razor blade for bonding to the silicon samples. The PDMS samples were cleaned in an ultrasonic de-ionized (DI) water bath for 20 min.

The PDMS layer was first treated by oxygen plasma (790 Series, Plasma-Therm, Inc., Florida, USA) and immediately brought in contact with the passivated silicon substrates for bonding. By far, the gas most commonly used for plasma activation is oxygen, but other substances like ammonia, nitrogen, and water vapor can also be used. The plasma power used was in the range of 20 to 140 W while the chamber pressure used was 30, 120, and 500 mTorr. The plasma exposure was maintained for 10 s for most tests, while some samples were plasma-activated for 40 s to study the time effect. After placing the two pieces together the specimens were treated in an oven at 150 ˚C for 2 h with a weight placed on it to improve the bond strength [2]. Similarly, some samples were treated at 100 ˚C to study the temperature effect.

2.2 Bond strength test

Tests of adhesion are essentially fracture tests and several possible tests can be performed qualitatively (peel test) [7] or quantitatively ((normal, peninsular, “inverted”) blister tests [6–10], tensile test [11,12], three-point bending test [13], and razor blade test [14].) As mentioned earlier, the amount of bond strength or adhesiveness was characterized by a manual peel of the passivated silicon from the underlying PDMS layer and through a mechanical shear test. Here, the main advantages of a qualitative test like peel tests are ease of implementation and it is quick way to justify if further quantitative tests are required. The bonding strength was taken as the percent area remained bonded after peeling the passivated silicon from PDMS. Figure 1 shows an illustration of the bonded area after a peel test. Each piece of PDMS (before peeling) has an average size of 4 cm^2 and a thickness of 2 mm.



(a) (b)

Figure 1. Samples after PDMS peel test (a) 100 % (b) ~50 % bonding of PDMS and passivation layer.

The shear test was carried out using a shearing machine (Dage Series 4000 DS100, Dage Group, Aylesbury, UK) commonly used in the electronics industry for shearing electrically connective solder balls.

Figure 2 shows a schematic of the shear test equipment. A sample of plasma activated PDMS bonded to passivated silicon was fixed using epoxy resin (Super Strong Epoxy-fix, Selleys, Australia) onto a piece of glass microscope slide (Eurotubo, Deltalab Group, Barcelona, Spain) which was in turn mechanically clamped (not shown in schematic) to the testing stage. The size of each PDMS piece was around 3 by 3 by 5 mm (L × W × H) while that of the passivated silicon remained at 1×1 cm^2. Using control joysticks, the shear cartridge was then pre-positioned as close as possible to the piece of PDMS. A pre-programmed recipe used for all tests was activated to run the shearing machine. Once activated, the shear cartridge will be automatically positioned at the pre-set shear height (distance between bottom face of shear cartridge and top face of the passivated silicon layer) of 50 μm before moving horizontally (indicated by white arrow) at a speed of 100 μm/s with a test load of 0.50 kg. The maximum load in kilograms exerted on the PDMS was noted and recorded as the failure shear load.

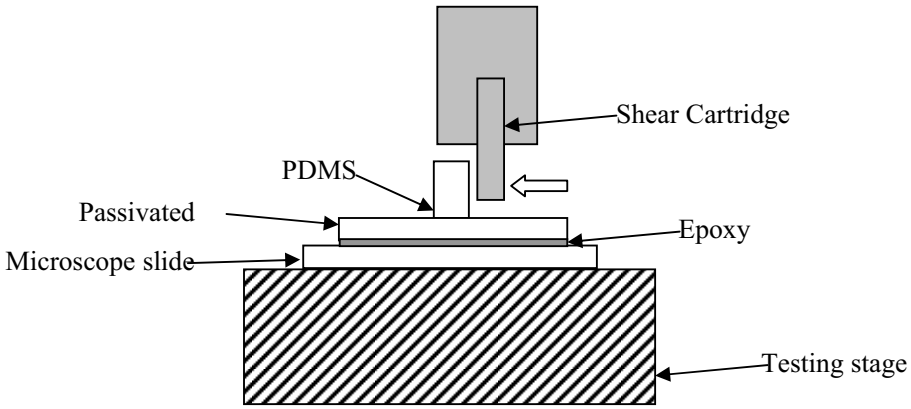
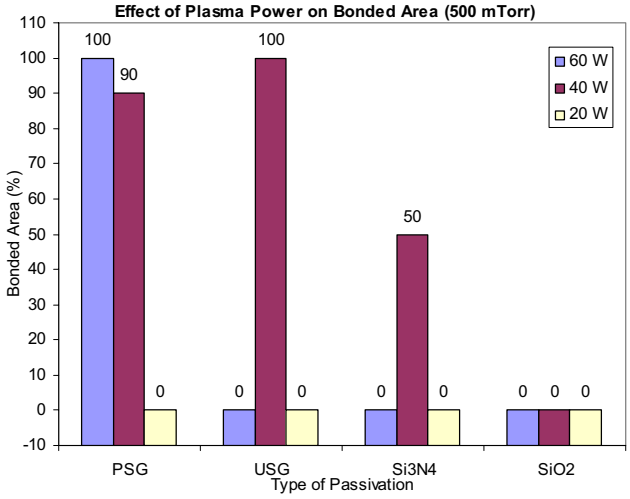


Figure 2. Schematic of the set-up for the quantitative evaluation of bond strength (not drawn to scale).

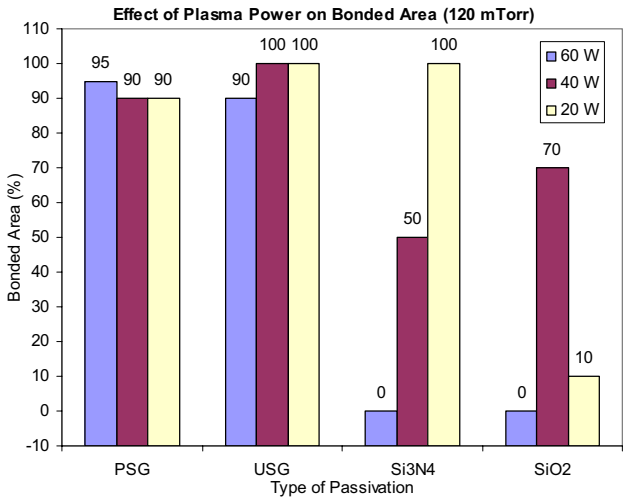
3. Results

3.1. Peel test results

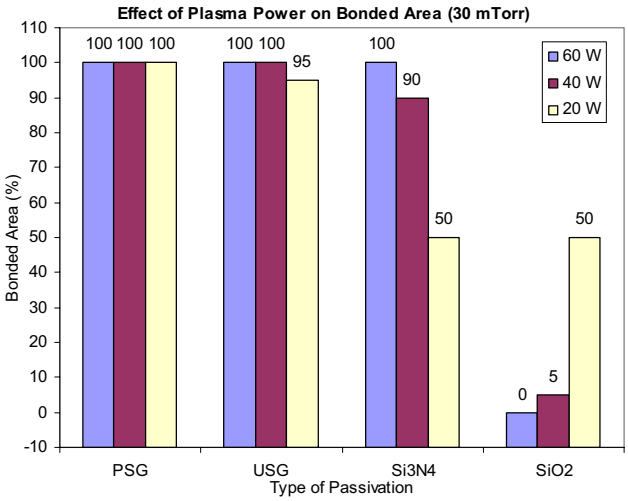
Figure 3 shows how the bonded area varied under different plasma power and pressure. For each set of passivation type, pressure and power, the peel test was performed once. Results from the peel test suggested that reducing the plasma pressure from 500 to 30 mTorr helped to improve the bond quality for PDMS bonding with PSG, USG and Si_3N_4 samples. This was substantiated by a reasonable and consistent amount of bond area for these observed samples. Among the passivation materials under investigation, PSG and USG always demonstrate best bonding quality despite different power conditions, which was in sharp contrast to the worst bonding of SiO_2 to PDMS. The reason for this inferior bonding is not clear, but it may be partially attributed to the different surface chemistry and topology as well. For example, the PECVD oxides demonstrated higher micro-roughness than SiO_2 as a result of the different deposition techniques, and this in turn might have manifested itself as a bonding mechanism in addition to the chemical interface bonding.



(a)



(b)



(c)

Figure 3. Salient peel test results. One sample was used for each test condition.

Figure 4 shows the effect of power variation on the bonded area at 120 mTorr. The high power effect has been tentatively attributed to the damage of the PDMS backbone, and the over-exposure effect has been linked to various observations on the PDMS surface, including cracked silica (SiO_x) and reduction of the effective silanol group due to the back biting scission reaction between excessive silanol groups. Besides higher plasma power, an over-exposure to plasma is detrimental to the adhesiveness of PDMS to all the passivation materials, which has also been reported in previous literatures [5,6]. We observed that USG to PDMS bonding area through peel test increased from 0 to 80 % when the plasma power was fixed at 80 W and the exposure time decreased from 40 to 10 s. Finally, it was also observed that the oven treatment temperature (100 °C vs. 150 °C) did not affect the bonding quality.

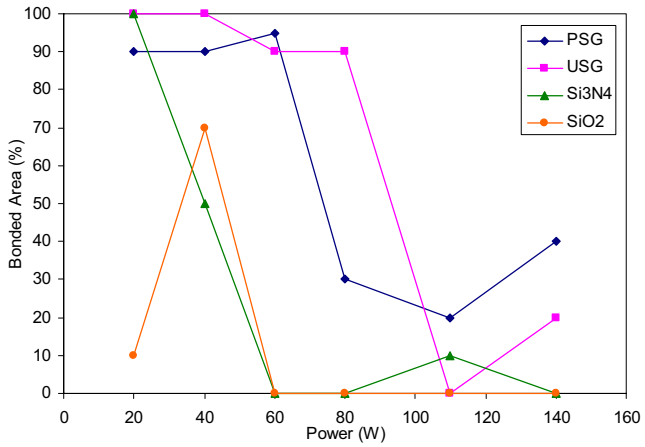


Figure 4. Effect of power variation on bonded area at 120 mTorr.

3.2. Shear test results

A trial shear test was performed on a batch of 18 USG samples and we found the failure shear loads, ranging from 0 to 1.12 kg, are randomly scattered among the samples as shown in table 1. There was no clear evidence of any behavioral trends for the bonding strength similar to what was seen from the results of the peel test. A possible reason could be the lack of a consistent and reproducible self-alignment technique to ensure that the PDMS always remains flat in contact against the shear cartridge during the shear process. This may have resulted in the introduction of background noise and consequently, scattered results.

Table 1. Results from shear tests performed on USG samples.

Pressure (mTorr)	Power (W)	Shear failure load (kg)
500	140	NA
500	110	NA
500	80	0.56
500	60	0.38
500	40	0.08
500	20	0.08
120	140	0.26
120	110	0.02
120	80	1.12
120	60	0.00
120	40	0.70
120	20	0.86
30	140	0.24
30	110	1.02
30	80	0.44
30	60	0.42
30	40	1.10
30	20	0.62

4. Conclusions

The bonding of oxygen plasma activated PDMS to passivated silicon was evaluated. The use of 30 mTorr plasma pressure and plasma power ranging from 20 to 60 W with 10 s of exposure time helped to improve the bond quality for PSG, USG and Si_3N_4. The bonding quality deteriorated with higher plasma power and longer plasma activation time, while the post-bonding oven treatment temperature showed no obvious effects. A simple shear test can be employed to characterize the bonding strength of PDMS to passivation materials, however the test set-up and procedure can be further refined to obtain more consistent results.

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