Studies on Surface Wettability of Poly(Dimethyl) Siloxane (PDMS) and Glass Under Oxygen-Plasma Treatment and Correlation With Bond Strength

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***Abstract*—An issue in microfabrication of the fluidic channels in glass/poly (dimethyl siloxane) (PDMS) is the absence of a well-defined study of the bonding strength between the surfaces making up these channels. Although most of the research papers mention the use of oxygen plasma for developing chemical (siloxane) bonds between the participating surfaces, yet they only define a certain set of parameters, tailored to a specific setup. An important requirement of all the microfluidics/biosensors industry is the development of a general regime, which defines a systematic method of gauging the bond strength between the participating surfaces in advance by correlation to a common parameter. This enhances the reliability of the devices and also gives a structured approach to its future large-scale manufacturing. In this paper, we explore the possibility of the existence of a common scale, which can be used to gauge bond strength between various surfaces. We find that the changes in wettability of surfaces owing to various levels of plasma exposure can be a useful parameter to gauge the bond strength. We obtained a good correlation between contact angle of deionized water (a direct measure of wettability) on the PDMS and glass surfaces based on various dosages of oxygen plasma treatment. The exposure was done first in an inductively coupled high-density (ICP) plasma system and then in plasma enhanced chemical vapor deposition (PECVD) system. This was followed by the measurement of bond strength by use of the standardized blister test. [1336]**

***Index Terms*—Advancing contact angle, bond strength, hydrophilicity, hydrophobicity, oxygen-plasma, poly (dimethyl) siloxane (PDMS), reactive ion etching (RIE).**

I. Introduction

SILICONE-BASED rubber poly (dimethyl siloxane) (PDMS) is primarily used in research laboratories all over the world for building of chip-based microfluidic devices fabricated using lithography and replica molding processes. These processes essentially use a variety of packaging techniques from spun on liquid PDMS acting as the adhesion layer [1] to chemical treatment of surfaces to make weak Vanderwaals forces that could hold fluid pressures up to around 5 psi. One technique most commonly used for getting irreversible seals is by exposing the surfaces to oxygen plasma. The changes in the surface texture and chemistry happening at such exposures have been widely studied by many research groups using a variety of techniques like fourier transform infrared spectroscopy (FTIR), X-ray photon spectroscopy (XPS), and atomic force microscopy (AFM), etc. [2]–[4]. Most of this research indicates that PDMS material in general comprises of repeated units of—$O - Si(CH_3)_2-$, which on exposure to oxygen plasma develops silanol groups ($-OH$) at the expense of methyl groups ($-CH_3$). As argued by Garbassi *et al.* [5], the oxidation of the surface layer increases the concentration of hydroxyl groups and this leads to the formation of strong intermolecular bonds [6], [7]. As the silanol groups are polar in nature, they make the exposed surface highly hydrophilic and this can be observed by a relative change in the advancing contact angle of deionized water [8]. These silanol groups then condense with those on another surface, when two such layers are brought into conformal contact. For both PDMS and glass, these reactions yield Si-O-Si bonds after loss of a water molecule. These covalent bonds form the basis of a tight irreversible seal between the layers [9]. Typically such seals can withstand 30–50 psi of air pressure and are practically inseparable. Although the various schemes for formation of these bonds and the chemistry behind it are very well estimated through hypothesis and experiment, there is no general rule of thumb, for predicting a set of good plasma parameter to obtain a reasonable level of bond strength for a beginner. As a result, for researchers venturing new into the field of microfabrication using polymer molding and plasma bonding processes, it may be necessary to try a lot of different combinations of exposure parameters for establishing the correct ones for any given plasma setup. This makes it difficult to achieve a reasonable level of wafer scale bonding between glass and PDMS and PDMS and PDMS while building microfluidic devices. As the fluid pressure in microflows never exceeds 5–6 psi [10], most of the fabrication research work focuses on obtaining a reasonably low level of adhesion, which can just hold together the flow path structure and does not look beyond that. The problem is faced in high pressure applications as in the case of certain peristaltic pump designs where an off chip compressed air supply is used to drive the fluids in micro channels created by a twin layer, one formed by bondage between glass with replica molded PDMS and another between PDMS and PDMS [11]. Also, in case of systems having pneumatic microvalves [12], a relatively high level of bonding particularly between two replica molded layers of PDMS becomes quite necessary. Another reason for establishing a good level of

Manuscript received May 4, 2004; revised August 19, 2004. This work was supported by the National Science Foundation. Subject Editor H. Fujita.

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Digital Object Identifier 10.1109/JMEMS.2005.844746

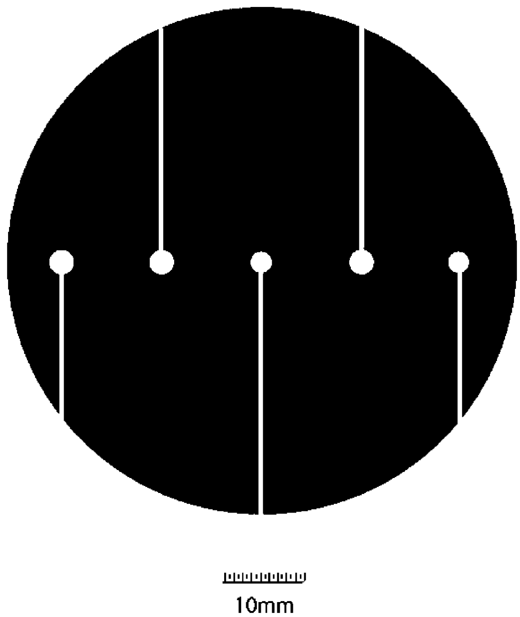


Fig. 1. Blister mask.

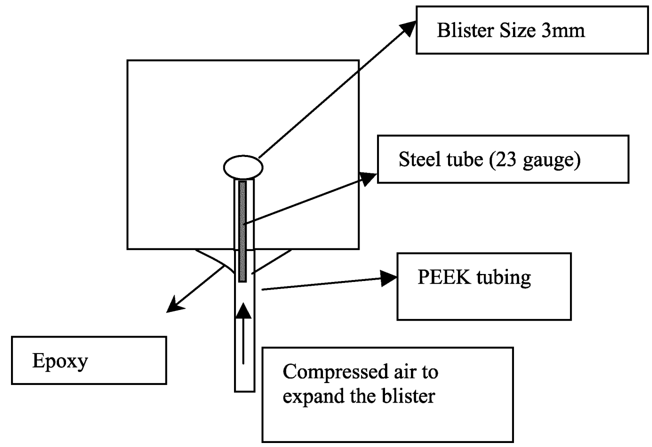


Fig. 2. PDMS blister.

irreversible bonding is the enhancement of the robustness of design during full scale manufacturing of the designs beyond research laboratories into real world applications.

This paper is intended to develop a general regime for the estimation of the bond strength using the differential advancing contact angle of glass and PDMS surfaces using the sessile drop method. We vary the plasma exposure dosages by controlling the parameters like time of exposure, reactive ion etching power and chamber pressure in a high density Trion inductively coupled plasma system. Such a technique promotes easy attainment of the required bond strength by observing the relative variation in surface wettability. Similar experiments are carried on a plasma enhanced chemical vapor deposition (PECVD) system. We get similar trends establishing confidence in the technique. We also measure the contact angle of chemically treated glass and PDMS surfaces and correlate it with the bond strength. As the bond is reversible in nature, we find the strength by using peel test.

II. EXPERIMENTAL

A. Fabrication of Blisters in PDMS for Bond Strength Measurement

The bond strength is measured using the blister test wherein a blister of 3 mm diameter is made in PDMS using photolithography and replica molding techniques. The masks for selective patterning are designed by Adobe illustrator and printed by using a high-resolution printer [see Fig. 1]. The fabrication of the blister is done in two layers. The negative photoresist SU8-2075 is spun onto a cleaned glass wafer of 63.5 mm diameter. The typical thickness of the resist in our experiment is about 200 micron after spinning. The SU8 is next patterned using the mask as shown in Fig. 1. This negative is used to cast the PDMS (GE Silicones RTV 615) up to 2.5 mm thickness. After curing the PDMS cast, pieces of size 12.7 mm $\times$ 12.7 mm are cut around the blister shapes. These are then bonded to pieces of plain PDMS, or cleaned glass slides of similar size by plasma treatment. For glass/PDMS bonding, the glass slides are thoroughly cleaned by boiling in piranha solution (5:1 ratio of concentrated H_2SO_4 and 30% H_2O_2 solution) for 3–4 min and then, repeatedly washed in DI water before plasma exposure. After fabricating the blister, an input port is attached to it using a steel pipe and a poly eukaryotic ether ketone (PEEK) (McMaster Carr) tubing, which is epoxied to one of the edges [see Fig. 2]. A regulated nitrogen supply is connected to the device. The pressure at which the blister starts to fail is noted down [13].

B. Description of the Plasma Tool

The first plasma equipment we use is a cluster tool designed with a state-of-the-art plasma etch and deposition capabilities. The inductively coupled plasma (ICP) is used as the primary plasma source to create plasma by inductively coupling the RF power via a copper coil. ICP is used to generate high-density plasma in a ceramic tube above the chuck. Then, RF power is supplied to the chuck to drive the ions to the sample, thereby increasing etch rate and anisotropy [14]. The parameters varied are chamber pressure, RIE power, and time of exposure for our present study. To test the repeatability of this data we use a capacitatively coupled PECVD system. In this system, a 13.56-MHz RF power supply with a maximum power of 300 W is used to support the plasma. The flow rate of each gas is regulated using mass flow controller before being admitted to the gas manifold. The pressure in the chamber is controlled using a butterfly valve [15] and the samples are placed on the cathode.

C. Contact Angle Measurement

Contact angle measurement is the ideal method to characterize surface wettability and widely used technique in studies of loss and recovery of hydrophobicity of silicone rubbers. So, this method can be used to accurately measure the hydrophilic characteristic of a surface for a polymer like PDMS, whose surface properties change speedily with post exposure time [16]. Thus, for accuracy of measurement, the contact angle measurement system used in this experiment is positioned close to the plasma exposure tool. This enables us to capture the image of a water droplet, dropped on the plasma treated sample within the first one minute of the plasma exposure. A CCD camera of the contact angle setup was used for this purpose. Simultaneously, a separate set of Glass-PDMS and PDMS-PDMS substrates exposed in the same run of the exposure tool are brought into conformal contact with each other after a similar span of time, as required to transit the exposed wafer and put a water drop over it. The surface hydrophobicity of a solid surface is determined by its free surface energy. Often, it is defined on the basis of the static contact angle between the surface and a water droplet. The fundamental equation for measurement of solid surface tension by contact angle measurements is described by the Young's equation [17].

D. Chemical Treatment of Glass and PDMS

The glass and PDMS surfaces are chemically treated using three different methods and the relative change in contact angle on the surfaces is measured. In the first method, pieces of PDMS and glass are immersed in 0.5 M (molar) boiling HCl for 10 min. The second method involves boiling the glass and PDMS in piranha for five minutes [9]. The third method for surface treatment begins by dipping the samples in acetone in an ultrasonic bath for five minutes. These are then rinsed with DI water and dipped in 30% Hydrogen peroxide for one hour. Rinsing is again done with DI water and Ethanol and then, compressed air is blown to dry the surface [18]. The chemically treated pieces are brought into contact to form a sealing. The force of adhesion of the chemically treated PDMS-PDMS and PDMS-Glass surfaces is observed qualitatively and the post treatment contact angle of the surfaces is measured using DI water. The bond strength is qualitatively checked by peeling of the two chemically treated PDMS surfaces or, the chemically treated PDMS and glass surfaces. The data obtained in the above experiment show that the contact angle level of PDMS does not change much and subsequently, the bond is of a reversible nature wherein the PDMS can be peeled off easily from a weakly bonded assembly. The quantitative measurement of bond strength was not possible due to the reversible nature of the weakly bonded surfaces. So, the blister test could not be used for estimation of bond strength.

III. Results and Discussion

The bond strength and contact angle are tabulated [see Table I] and plotted as a function of chamber pressure (mtorr), RIE power (W) and time of exposure (s) for PDMS-PDMS and PDMS-Glass bonding, respectively. In case of PDMS-Glass bonding, the contact angle was measured on the glass surface. The contact angles of piranha cleaned glass and PDMS before the exposure are 20° and 109°, respectively. In the first set of experiments, the bond strength is measured by varying chamber pressure and keeping ICP at 150 W, RIE power at 20 W oxygen flow rate at 20 sccm and the time of exposure at 30 s. Bond strength is found to increase with an increase in chamber pressure. In the second set of experiments, the RIE power is varied at a constant chamber pressure (1000 mtorr for Glass-PDMS and 700 mtorr for PDMS-PDMS) and all other parameters remaining same as before. In the third experiment, the time of exposure is varied and other parameters kept same as before. The variation in RIE power and time of exposure indicates maximum bond strength at a certain optimum value of power and time. This observation is true for both PDMS to PDMS and PDMS to glass bonding. In the following subsections we will describe these effects in details.

A. Effect of Chamber Pressure Variation

Fig. 3(a) and (b) illustrates plots for contact angle and bond strength versus chamber pressure for a fixed RIE power and exposure time for Glass-PDMS and PDMS-PDMS bonding, respectively. Bond strength is measured as the value of pressure at which interfacial separation of the pressurized blister starts occurring. As our measurement setup is not capable to accurately measure below five degree contact angle, all angles below 5° have been replaced by an average value of 2.5°. Physically, such a situation can only occur as the surface silanol bond density reaches its maximum thus inducing in the surface highest degree of polarity. The error bar for bond strength is ± 2 psi. Error bar for the contact angle is found to be $\pm 2.5°$. The error bar has been computed on the basis of results obtained by repetition of experiments at sixteen different observation points among a total of around 50 points.

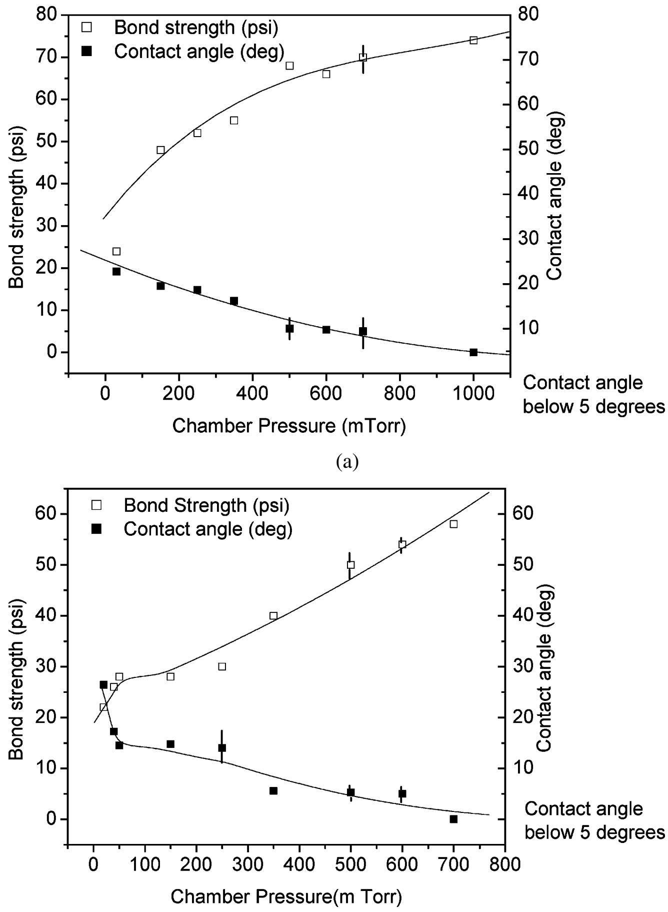
The maximum bond strength obtained for glass to PDMS bond is found to be 74 psi. This corresponds to a contact angle of less than 5°. There is a decrease in bond strength below 100 mtorr pressures. Normally, at a chamber pressure of 100 mtorr or less the plasma etching becomes highly directional and anisotropic [3]. Here, we would like to hypothesize that the high level of anisotropy in etching may lead to damage in the siloxane backbone causing a change in the overall bond chemistry and a reduction in bond strength. The change in contact angle measured on the glass surface does not show a sharp decrease with increase in chamber pressure like that of PDMS as will be explained later. For PDMS-PDMS bonding the maximum bond strength is found to be 58 psi. The corresponding contact angle is found to be less than 5°. The bond strength curve in the low pressure region (<100 mtorr) is similar to the curve for glass to PDMS bonding. In the high pressure region (>100 mtorr) there is a gradual increase in the bond strength and decrease in contact angle with an increase in pressure. The behavior of the data in the high-pressure region can be explained in the following way. As the chamber pressure increases, the mean free path of the gas molecules reduce and the plasma becomes more and more isotropic. With an increase in pressure, the sheath of charged particles formed near the electrode move closer to the substrate [3]. So, newly formed ions near this sheath have smaller distances to travel before striking on to the substrate resulting in less momentum transfer. To explain the increase in bond strength, we would like to hypothesize that the less energetic oxygen ions generated in this way may remove methyl groups from the surface without damaging the siloxane backbone.

One more important behavior of the trend is reflected at pressures below 100 mtorr, where the contact angle of PDMS rises faster than that of glass. This can be explained by the fact that glass is more rigid structurally. So, at a low pressure and greater mean free paths, the ionic momentum transfer suddenly increases. This situation is sufficient to damage the flexible siloxane backbone in PDMS leading to a change in

TABLE I
CONTACT ANGLE AND BOND STRENGTH DATA FOR VARIOUS CHAMBER PRESSURES, RIE POWER VALUES AND TIME OF EXPOSURE FOR VARIOUS PLASMA SYSTEMS

S.N.	Type of Plasma Etcher	Chamber pressure (mTorr)	RIE power (Watts)	Time of Exposure (secs)	Surface Hydrophilicity measured by contact angle		Bond Strength measured by separation pressure	
					Surface type	Contact angle error ± 2.5 deg.	Nature of bondage	Pressure error ± 2 psi
1		20				26.45		22
2		40				17.22		26
3		50				14.5		28
4		150				14.8		28
5	ICP	250	20	30		14.04		30
6		350			PDMS	5.59	PDMS-PDMS	40
7		500				5.26		50
8		600				5.02		54
9		700				2.5 (avg. val.)		58
10	PECVD	900	10	30		8.3		36
11		100				19.19		23
12		30				19.2		24
13		150				15.77		48
14		250				14.85		52
15	ICP	350	20	30	Glass	12.21	PDMS-GLASS	55
16		500				5.68		68
17		600				5.41		66
18		700				5.03		70
19		1000				2.5 (avg. val.)		74
20			5			19.6		14
21			10			8.1		48
22			20			2.5 (avg. val.)		50
23			30			8.8		44
24	ICP	700	50	30	PDMS	10.0	PDMS-PDMS	38
25			75			15.2		26
26			100			22.1		14
27			125			23.8		12
28			150			28.5		10
29	PECVD	900	10	30	PDMS	7.1	PDMS-PDMS	37
30			30			12.2		30
31			10			16.32		50
32			15			15.5		54
33			20			2.5(avg. val.)		68
34			30			7.05		57
35	ICP	1000	50	30	Glass	10.06	PDMS-GLASS	52
36			75			14.55		50
37			100			17.27		46
38			125			18.74		42
39			150			27.91		40
40				5		28.16		26
41				10		26.79		30
42				15		18.44		37
43				20		5.18		51
44	ICP	700	20	25	PDMS	2.5 (avg. val.)	PDMS-PDMS	50
45				30		5.23		43
46				40		8.13		25
47				45		10.95		23
48				50		10.97		20
49				60		17.13		20
50	PECVD	900	10	35	PDMS	9.32	PDMS-PDMS	32
51				60		12.15		35
52				10		18.31		48
53				20		5.05		72
54				30		2.5 (avg. val.)		70
55	ICP	1000	20	40	GLASS	10.14	PDMS-GLASS	50
56				45		15.05		46
57				50		15.45		44
58				60		16.01		35

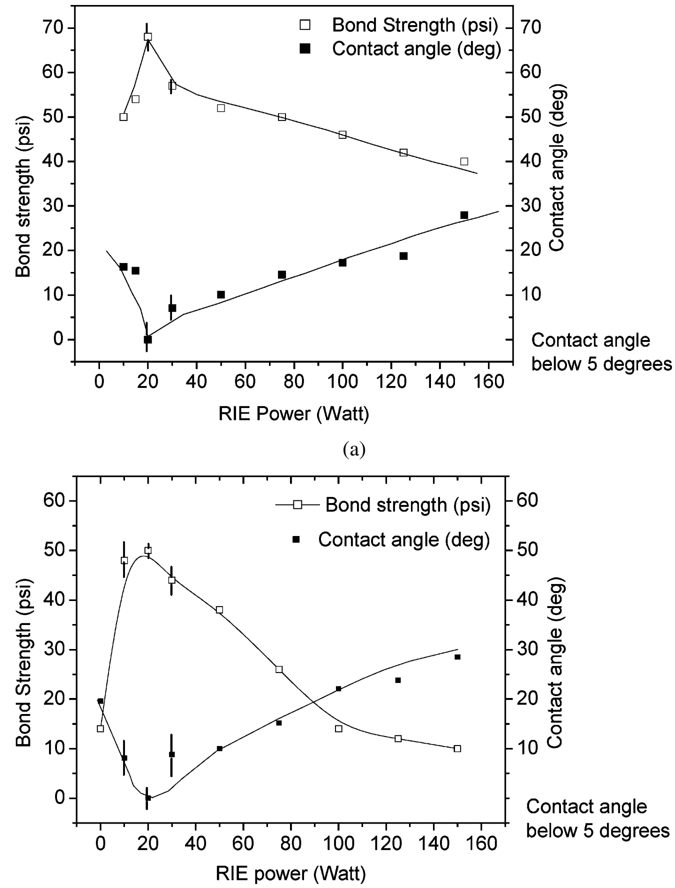
surface chemistry; however, the momentum transfer is not sufficiently strong to affect the sturdy surface structure of glass. Thus, the contact angle in the case of glass does not increase so much at lower pressures as in case of PDMS. As we will show later, in case of PDMS-Glass bonding, the contact angle of PDMS surface exhibits a better correlation with the bond



(a)

(b)

Fig. 3. (a) Plot of contact angle and bond strength with chamber pressure for Glass-PDMS bonding. (b) Plot of contact angle and bond strength with chamber pressure for PDMS-PDMS bonding.



(a)

(b)

Fig. 4. (a) Plot of contact angle and bond strength with RIE power for Glass-PDMS bonding. (b) Plot of contact angle and bond strength with RIE power for PDMS-PDMS bonding.

strength, compared to the contact angle of glass surface. The behavior of the bond strength is by and large reverse to that of contact angle, which fits our theory very well. However, the point corresponding to the pressure value of 50 mtorr [see Fig. 3(a)] shows a sudden reduction in the bond strength value which may indicate an extraordinary damaging of the PDMS structure causing a substantial loss of surface silanol bond density [8]. Thus, in this case, although the alteration of glass surface is relatively less, the damage to PDMS surface causes a huge decrease in bond strength.

B. *Effect of RIE Power Variation*

Fig. 4(a) and (b) shows plots of contact angle and bond strength for variation of RIE power. The data shows an interesting trend wherein the bond strength peaks at 20-W RIE power for glass to PDMS and PDMS to PDMS bonds. The peak values of bond strengths are 68 psi for PDMS-Glass and 54 psi for PDMS-PDMS, respectively. The contact angle trend follows an inverse behavior to bond strength. The contact angle curve has a minimum value of 5° at 20 W RIE power level and then goes up with increase or decrease in RIE power. The bond strength shows a reverse trend to the contact angle.

This behavior can be explained by considering the plasma behavior for various bias levels dictated by RIE power. At low power levels, the kinetic energy of ions incident on the substrate reduces. This coupled with the ambient high chamber pressure leads to a large reduction in the number of reactive ions on the substrate. This is so because a lower power level reduces the electron acceleration within the plasma environment leading to a reduction in the radical density. Thus, less number of active sites form on the substrate surface after etching of CH_3 groups in such a plasma environment leading to a reduction in Si-OH available for surface bondage. Thus, the ions tend to graze on the surface of the substrate without producing much chemical or physical change of the surface. The reverse behavior at higher power levels suggests an increase in the ion bombardment. Thus, the Si-O-Si, whose dissociation energy (445 KJ/mol) is much higher than the Si-C bond dissociation energy (306 KJ/mol.), is affected resulting in damage of the overall uniquely flexible Siloxane backbone [19]. Contrary to the chamber pressure variation case, one important observation in this case is a similar trend in variation of the contact angle and bond strength in both Glass-PDMS and PDMS-PDMS bonds. This can be attributed to the constancy in the chamber pressure due to which directionality never arises in the etching. This helps in preventing the differential nature of trends in both

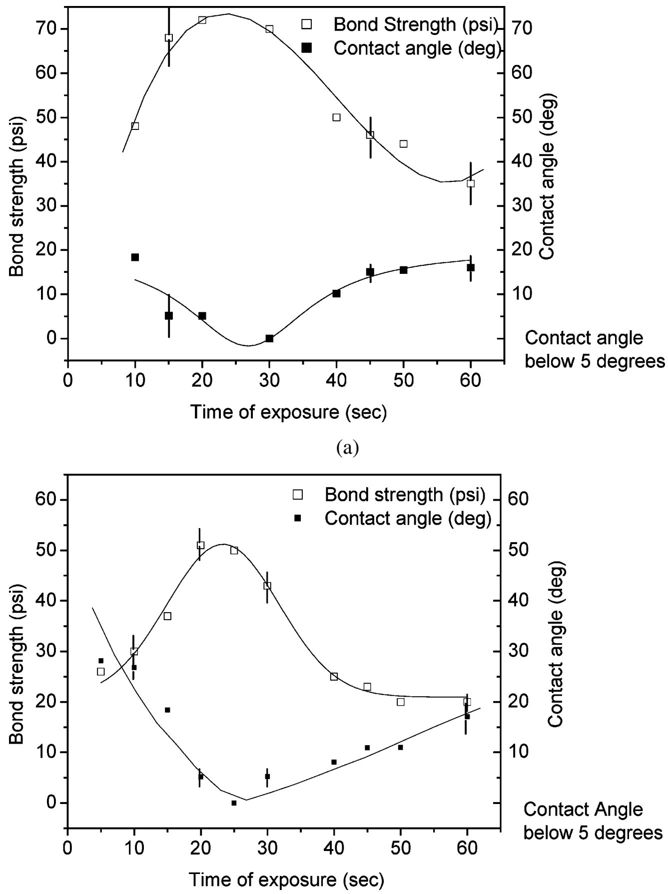


Fig. 5. (a) Plot of contact angle and bond strength with time of exposure for Glass-PDMS bonding. (b) Plot of contact angle and bond strength with time of exposure for PDMS-PDMS bonding.

cases by eliminating the anisotropicity levels, as in the low chamber pressure case.

C. Effect of Time of Exposure

The time of exposure has a similar trend as RIE power [see Fig. 5(a) and (b)]. The bond strength peaks in this case for an exposure time of 20 s. The values of bond strengths are similar to that obtained in the earlier cases, with a rise in contact angle and subsequent fall in bond strength at a longer or shorter exposure time. The least contact angle value at highest bond strength is again less than 5°.

One possible explanation can be obtained from Owen and Smith's investigation on the PDMS surface after exposure to high RF power and longer times. The progressive oxidation of the surface leads to the formation of an extremely brittle silica layer on the surface. Owen and Smith have clearly observed cracking on the surface under scanning electron microscope (SEM). They mentioned that less harsh, lower RF power, and shorter treatment times produced uncracked surfaces with a layer of Silica [SiO_x], which retards the migration of low molar mass molecules from the bulk of the structure. As this layer is exposed longer in plasma environment, it cracks and promotes transport of low molar mass molecules to the surface, which covers the oxidized layer [20]. This is indicated by a change in contact angle from less than 5° to 18°. In anticipation of a similar behavior, we expose samples to optimum (30 s) and long times (70 s) of exposure in the PECVD system. We characterize both surfaces with SEM. The surface with high time of exposure develops cracks.

We have tried to explain the behavior of both Glass-PDMS and PDMS-PDMS data trends from the available literature about PDMS surface transition and dynamics. Here, we will like to hypothesize that, owing to the prolonged duration of exposure, the treated PDMS forms excessive surface silanol concentration causing a reorientation of surface chemical bonds [20]. This process seems to be accelerated further by the ambient UV radiations in the plasma. Hillborg and Gedde [8] have proposed a set of chemical reactions involved in PDMS surface transition in oxygen plasma with UV radiation. Their work strongly suggests formation of excessive silanol bonds under such an atmosphere. As the silanol bond density increases it results in chemical transformation on the surface known as surface chain scission reactions. In such a situation a marked reduction in the number of surface silanol bonds occur by back biting scission reactions, a physical surface cracking and a gradual migration of the mobile, low molar mass PDMS oligomers to the surface as explained in many earlier PDMS surface transition studies [8], [20], [21]. The dissimilar behavior of bond strength on both sides of the optimum time of exposure is currently not well understood; but, preliminary studies with surface roughness indicate that cracks formed on the surface at higher times of exposure may contribute to the increase of surface roughness and may make bond strength depend not only on the surface recovery but also, on the average surface roughness due to crack formation. Detailed study of the distribution of cracks based on depth and their contribution to bond strength is a promising topic for our future studies. On the other hand, shorter exposure time may be insufficient for a removal of adequate number of methyl groups—which is essential to form an optimum surface silanol bond density.

D. Universal Curves

A general trend was plotted using all values of contact angle and bond strength for all different plasma parameters [see Fig. 6(a) and (b)]. The plot gives a universal increase in bond strength with decrease in contact angle for both Glass-PDMS and PDMS-PDMS cases for variation of RIE power and pressure. The time of exposure does not follow this universal trend for exposure times more than 30 s for reasons discussed earlier. This is true for both Glass-PDMS and PDMS-PDMS bonding. Identical behavior is observed on the PECVD tool and the new data points indicated by the open squares and triangles fit the universal trend perfectly. A curve fitting exercise is performed on the obtained trends, which shows that a linear fit is the best fit for the PDMS-PDMS bonding. The fit indicates a zero bond strength corresponding to a contact angle of 30 deg and a 58-psi bond strength corresponding to zero contact angle. For PDMS-Glass universal curve, one set of data reflects the contact angle of glass versus the bond strength [see Fig. 6(a)]; the other set shows about the contact angle of PDMS versus the bond strength [see Fig. 7]. The data for contact angle of glass shows a large scatter. This is because of the different nature of recovery

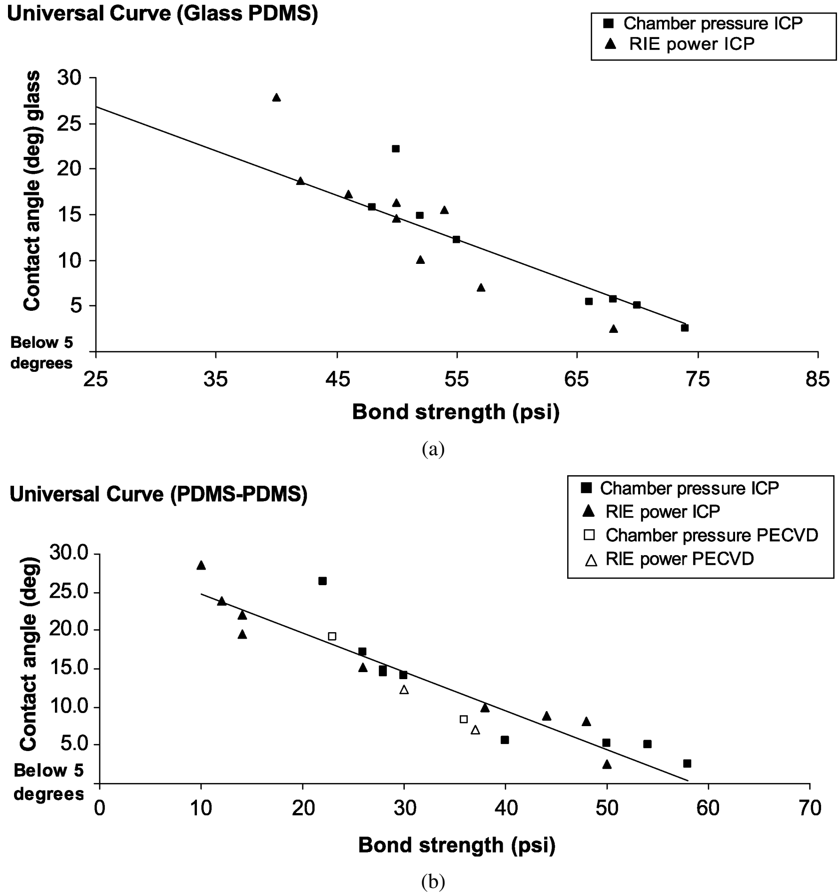


Fig. 6. (a) Plot of contact angle of glass versus bond strength for variation of chamber pressure and RIE power for Glass-PDMS bonding. (b) Plot of contact angle versus bond strength for variation of chamber pressure and RIE power for PDMS-PDMS bonding.

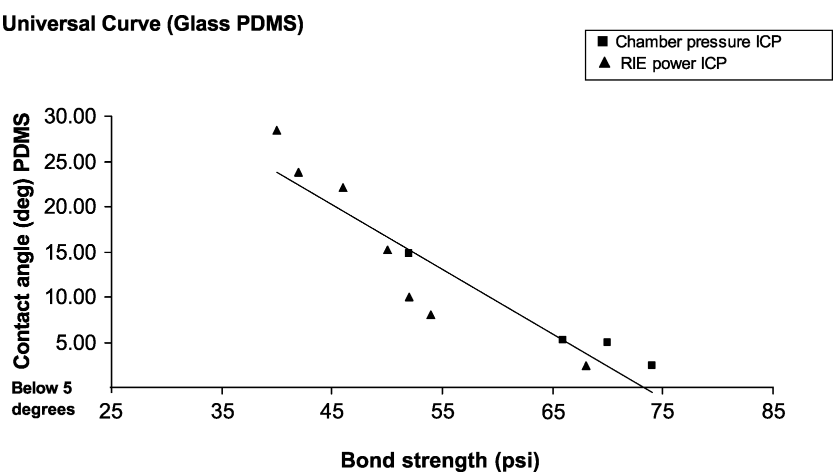


Fig. 7. Plot of contact angle of PDMS versus bond strength for variation of chamber pressure and RIE power in Glass-PDMS bonding.

mechanisms on the glass and PDMS surfaces. As we have explained earlier, the surface modification of PDMS influences the bond strength more than the glass surface. Thus, when the contact angle of PDMS is plotted against bond strength for PDMS-Glass assembly, we obtain a good linear fit [see Fig. 7], thereby supporting our hypothesis. In this case the fit indicates a zero bond strength corresponding to a PDMS contact angle of 55 deg and a 72 psi bond strength corresponding to zero contact angle. Thus, it is clear that the trend shown by the universal curve is valid until the surface is not damaged severely at larger exposure times, causing surface cracks. Depending on the plasma properties, the surface that influences the bond strength could be glass, PDMS or both.

IV. Conclusion

The hydrophobic surface of PDMS becomes hydrophilic upon oxygen plasma treatment under certain process conditions. We believe that oxygen plasma exposure at lower RF power with shorter duration makes a thin layer of undamaged oxide on the surface of PDMS with active silanol groups. This largely facilitates an irreversible sealing between the surfaces. The results follow a universal trend in terms of bond strength and contact angle measured on plasma treated surfaces. In case of change of a plasma exposure setup, the new parameters can be easily established by following the universal trend. It is observed that one gets stronger bonding as contact angle decreases, when PDMS or glass is treated by O_2 plasma. An excellent correlation between different plasma parameters and surface wettability of PDMS or glass surface measured in terms of contact angle is found. All the results indicate that a contact angle below 5 degree is a general requirement for getting very good bond strength and thereby, one can obtain the correct plasma parameters for surface treatment by investigating and monitoring contact angle.

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