

NOTE

Determining the optimal PDMS–PDMS bonding technique for microfluidic devices

Mark A Eddings¹, Michael A Johnson² and Bruce K Gale²

¹ Department of Bioengineering, University of Utah, Salt Lake City, UT, USA

² Department of Mechanical Engineering, University of Utah, Salt Lake City, UT, USA

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Abstract

A number of polydimethylsiloxane (PDMS) bonding techniques have been reported in the literature over the last several years as the focus on multilayer PDMS microfluidic devices has increased. Oxygen plasma bonding, despite cost, additional fabrication time and inconsistent bonding results, has remained a widely used method for bonding PDMS layers. A comparative study of four rapid, inexpensive alternative PDMS–PDMS bonding approaches was undertaken to determine relative bond strength. These include corona discharge, partial curing, cross-linker variation and uncured PDMS adhesive. Partial curing and uncured PDMS adhesive demonstrated a considerable improvement in bond strength and consistency by retaining average bond strengths of over 600 kPa, which was more than double the average bond strength of oxygen plasma. A description of each technique and their performance relative to oxygen plasma bonding is included.

(Some figures in this article are in colour only in the electronic version)

Introduction

A critical design consideration in the development of PDMS-based microfluidic devices fabricated through multi-layer soft lithography is the method of bonding used to combine the molded layers. A number of factors influence the quality of the bond, including the type of technique used, the cleanliness of the bonding surfaces, the ratio of PDMS base to cross-linking agent and the curing/baking process. In most cases, the application and complexity of the design will dictate which bonding technique is required to fulfil the design criteria. Some techniques may not be suitable for all applications. Several bonding techniques alter the surface chemistry within the microchannels, which in the case of biomedical applications may be helpful or problematic. High-pressure gas applications may require high bond strength to maintain the integrity of the device, while other applications may integrate microvalves that depend on low bond strength to help seal microchannels. An expansion of PDMS is desirable for flexing PDMS layers in micropumps, but could cause uncertainty in metering precise volumes of sample for a reaction. Another practical concern, often overlooked in

the research stages of a microfluidic device, is the production volume required, which ultimately will narrow the range of bonding techniques available when fabricating commercial devices.

There are a number of PDMS bonding strategies that have been reported. One of the first reported techniques relied upon the hydrophobic, conformal contact of two fully cured layers of PDMS that are brought together to form an easily reversible seal [1]. Duffy *et al* introduced surface oxidation to increase the bond strength by activating layers of cross-linked PDMS in oxygen plasma [2, 3]. Surface oxidation is believed to expose silanol groups (OH) at the surface of the PDMS layers that when brought together form covalent siloxane bonds (Si–O–Si). This approach makes the channels more hydrophilic, allowing for easier fluid filling for a period of time after the oxygen plasma treatment. However, the surfaces can quickly revert to their hydrophobic tendency if exposed to atmosphere. Oxygen plasma treatments are also advantageous because they enable the bonding of PDMS to other materials such as glass. Drawbacks associated with oxygen plasma include: placing the exposed bonding surfaces in a low-vacuum environment in the presence of oxygen gas and the operation of the

equipment within a cleanroom facility. The oxygen plasma adds a significant cost to the fabrication process while limiting flexibility with the substrates due to cleanliness requirements and the size restriction of the chamber. Unger *et al* reported a bonding technique that relied on using a sticky layer with less cross-linking agent that would be combined with a hard cross-linked layer [4]. The method was implemented in multi-layer microfluidic devices that contained pneumatically actuated peristaltic pumps. This approach is advantageous in that a multilayer device can be assembled right after removal from the molds eliminating, the need for additional surface modification, which can also reduce the risk of dust and other contaminants compromising the bond. The use of different cross-linking ratios may not be suitable for many applications, as the surface characteristics will vary throughout the device from sticky to hard. Satyanarayana *et al* reported the 'stamp-and-stick' approach using intermediate layers of uncured PDMS or UV-curable glue to bond layers together [5]. Recently, Samel *et al* demonstrated a variation of this technique by using curing agent as the adhesive [6]. One of the layers would receive the adhesive layer by being brought in contact with a substrate coated with a thin layer of the curing medium. The layer would then be removed and brought in contact with another PDMS layer. The resulting device could be exposed to UV light or placed in a curing oven depending on the adhesive used. This technique mimics more traditional manufacturing techniques and provides the opportunity of bonding other materials outside of PDMS. It does require significant care when bringing the surfaces together as the PDMS or UV glue can spread into the channels of the microfluidic device. Partial curing of PDMS has also shown to be an effective, inexpensive method for creating multi-layer devices [7, 8]. PDMS mixed at a 10:1 curing ratio is placed into molds and cured at either a short period of time (30–60 min) or at a lower temperature (less than 60 °C). The PDMS layers become hardened enough to handle and remove from the molds, but remain uncross-linked enough at the interfaces between layers to form an effective bond. Corona discharge, reported by the Beebe group [9], is a surface activation technique that can be implemented on fully cured PDMS to bond several layers together. A hand-held corona device generates a high voltage potential across the electrodes at the tip of the unit, ionizing the air to create the localized corona discharge. The process can be performed at room temperature and standard pressure without the need of a vacuum system. The distance between the electrodes can be adjusted to expand the width of the activation area for devices that require a larger bonding surface.

While many of these bonding techniques have been optimized and widely studied, it is hard to ascertain if one method has an advantage over another in terms of bond strength. The techniques vary due to cost, convenience and processing time, however, no experimentation has been undertaken to determine their relative bond strengths. This study compares the various bonding techniques on identical test devices, to identify the strengths and weaknesses of each technique.

Methods

Mold fabrication

The PDMS test devices were fabricated from molds created using Xurography [8, 10]. This rapid prototyping technique utilizes a high-resolution knife plotter (FC5100 A-75, Graphtec, Inc.) to cut microstructures in adhesive-backed masking material. The unwanted material is removed leaving the microstructures that will form the mold. The structures are limited to the thickness of the material. An adhesive-backed vinyl masking (Scotchcal 220, 3M) approximately 100 μm thick was used and placed in polystyrene dishes to form the molds. The patterned masking consisted of 5 mm diameter circles. Because the stress concentrations are highest around the bonding interface, a test chamber with a large diameter was selected for the comparative experiments. Geometries typical in microfluidic systems would see a reduction in both the bonding interface and the total area in contact with the regulated air as the dimensions of the channels scale from mm to μm .

Bonding experiments

PDMS (Sylgard 184, Dow Corning) was mixed at 10:1 ratios except for the curing agent ratio variation experiments. Oxygen plasma test devices were created using an Oxford Plasmalab 80 Plus RIE unit. Optimal test conditions of 700 mTorr chamber pressure, 20 W RIE power and a 30 s exposure time were obtained from previously reported oxygen plasma bonding studies [11]. For the corona discharge experiments a hand-held discharge unit from Enercon (Dyne-A-mite 3D Treater) was used, with an output voltage of 15 kV. As there were no previously reported optimal conditions, an exposure time of 30 s was used to be comparable to the oxygen plasma test condition. PDMS was poured into molds consisting of round 5 mm diameter circles and was cured overnight at 60 °C. The molded half was capped with another molded PDMS layer and was bonded using different bonding methods, oxygen plasma, corona discharge, partial curing, varying curing agent ratio and stamp/stick. For partial cure bonding, the PDMS was pre-cured at 60 °C for 35 min prior to bonding and then allowed to fully cure overnight following the bonding. Prior to bonding, a tubing inlet hole was cored into the molded circles to provide the pressure source connection to the test devices. The molded layer was 5 mm thick to improve the pressure fit around the tubing interconnect. New devices consisting of nine replicates were fabricated for selected parameters for each bonding method. Tubing modified with a pipette tip was packaged within the cored inlet creating the tight pressure fit needed for the high-pressure experiments. A diagram showing the test devices is shown in figure 1.

Test conditions

Regulated air pressure from the building compressed air line was supplied to the pressure chamber inlet of the PDMS test devices. The air pressure was varied in increments of 35 kPa at

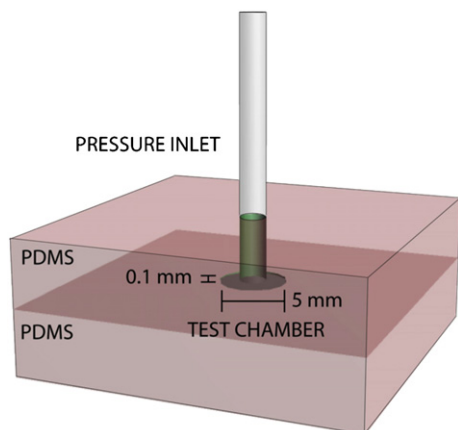


Figure 1. A representation of the two-layer test device. The two layers are brought together to form the pressure chamber. An inlet is cored into the chamber to control the pressure.

time intervals of 5 s until bond integrity was compromised. The ramping of the air pressure was used to maintain similar fatigue conditions among the test devices. Device failure was visually observed by seeing the two layers delaminate. Devices that withstood pressures greater than the regulator’s capacity were recorded as holding that maximum capacity. To verify no air pressure losses due to the packaging interconnect, a soap-water solution was placed around the tubing-device interface on all test runs, and an additional mechanical support was used to fix the tubing in place.

Results and discussion

Bond strength comparison

A comparison of the five PDMS bonding method results are shown in figure 2. The oxygen plasma treatment parameters were selected from a comprehensive study mentioned previously [11]. The results matched well with the published reports of bond strengths averaging 300 kPa. There was, however, significant variability in bond strength per test device. The bond strength ranged from 180 kPa to 715 kPa, which reduces confidence in the repeatability of oxygen plasma treatments under the same conditions. In addition to the cost and cleanroom requirements, this variability contributes to the fabrication frustration surrounding the use of this technique. However, the flexibility of bonding non-PDMS materials to PDMS may override the problems associated with this method. Corona discharge performed similarly to oxygen plasma except with a slightly lower standard deviation of 8.9 compared to 13.4 with oxygen plasma. The corona may have performed comparably to oxygen plasma due to the ability to quickly assemble the two treated PDMS substrates before the free sites reorient themselves. The plasma treatment should provide a more uniform and consistent activation of the surface, however, there is a longer delay in the assembly of the oxygen-plasma-treated surfaces as the chamber returns to atmospheric conditions and the substrates are removed from the chamber. The average

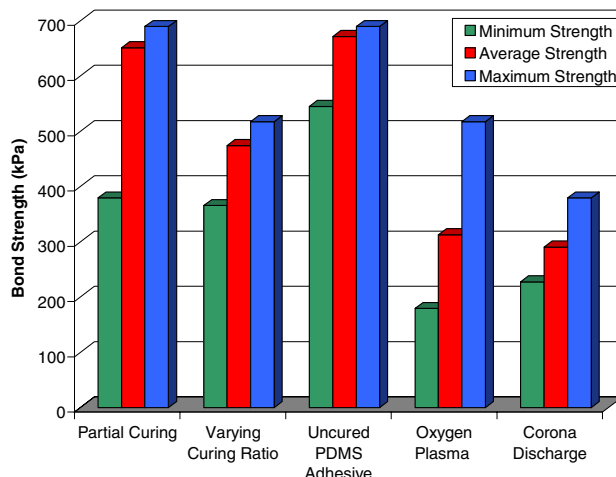


Figure 2. A comparison of five different bonding techniques at their optimal conditions at 60 °C. Partial curing experiment: 35 min precure, varying curing ratio experiments: PDMS base to cross-linking agent (15:1), uncured PDMS adhesive experiments: completely uncured PDMS layer stamped, oxygen plasma treatment: 700 mTorr chamber pressure, 20 W RIE power, 30 s exposure time. Corona discharge: 15 kV output, 30 s exposure time. The minimum and maximum bond strength values are shown with the average bond strength to illustrate the range of values obtained during experimentation.

bond strength was 290 kPa, while the variation from the minimum to the maximum was 227–380 kPa. The ability to use the corona discharge unit in a non-cleanroom setting dramatically reduces the cost and complexity when assembling PDMS layers. Like oxygen plasma it provides the flexibility of bonding other types of material like glass with similar bond strengths [9]. It also simplifies multilayer stacking of device layers.

The alteration of the cross-linker to base ratio method demonstrated an optimal ratio around 15:1. The performance at the 15:1 ratio performed better than oxygen plasma and corona discharge in the overall strength and consistency with an average bond strength of 474 kPa and a performance range from 365 to 517 kPa. It should also be noted that the standard 10:1 ratio demonstrated an average bond strength of 160 kPa, which would probably be suitable for most microfluidic applications where minimal pressure is required. This technique provides an inexpensive, rapid, non-cleanroom approach to bonding multiple layers; however, it does produce a softer, stickier material that may not be suitable for all applications. Partial curing of PDMS layers presents a slight alteration of the previous method but yielded significantly better bond strength capability at the optimal conditions. At the time condition of 35 min, the average bond strength was found to be 651 kPa. This method provides all the advantages of adjusting cross-linking agent without having to work outside the standard PDMS (Sylgard 184) 10:1 ratio. The stamp-and-stick technique demonstrated a substantial bond strength capability with low variability. The average bond strength was 671 kPa with a performance range from 545 to 690 kPa. This method provides the most flexibility in fabrication because time, temperature and cleanliness

considerations have less of an impact on performance. Multilayer devices (3+layers) are also more practical with this technique because they can be bonded after the layers are fully cured. However, multilayer devices requiring thin membranes will be limited by the minimum achievable thickness of the stamped layer. The accidental filling of the microfluidic channels is also possible if care is not taken during the stamp or glue application stage.

Conclusion

The bonding strength experiments demonstrated an ability to withstand pressures exceeding 250 kPa for all the techniques explored in this study, which is suitable for most microfluidic applications. The methods based on curing properties exhibited a significant increase in bond strength as compared to oxygen plasma bonding and corona discharge. Partial curing and uncured PDMS adhesive techniques showed the highest bond strength and are recommended as simplified, cost- and time-saving replacements for oxygen plasma bonding in PDMS–PDMS microfluidic devices.

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