Microfluidic Channel Fabrication by PDMS-Interface Bonding

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Abstract

A novel technique to bond polymer substrates using PDMS-interface bonding is presented in this paper. This novel bonding technique is promising to achieve precise, well-controlled, low temperature bonding of microfluidic channels. A thin (10-25µm) Poly(dimethylsiloxane) (PDMS) intermediate layer was used to bond two poly(methyl methacrylate) (PMMA) substrates without distorting them. Microchannel patterns were compressed on a PMMA substrate by hot embossing technique first. Then, PDMS was spin-coated on another PMMA bare substrate and cured in two stages. In the first stage, it was pre-cured at room temperature for 20 hours to increase the viscosity. Subsequently, it was bonded to the hot embossed PMMA substrate. In the second stage, PDMS was completely cured at 90°C for 3 hours and the bonding was successfully achieved at this relatively low temperature. Tensile bonding tests showed that the bonding strength was about 0.015MPa. Microfluidic channels with dimensions of 300µm×1.6cm×100µm were successfully fabricated using this novel bonding method.

Keywords: PMMA, PDMS, Hot Embossing

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1. Introduction

In recent years, polymer-based microfluidic devices have become increasingly important in biological applications (e.g., see [1]). However, polymer substrates must be bonded to make functional microchannels and the adhesion between the substrates is a problem of great practical concern. Existing polymer-to-polymer substrate bonding methods include thermal-compression, ultrasonic, and gluing by application of either epoxy or methanol. Unfortunately, these techniques are not precise when compared to standard IC/MEMS bonding processes, i.e., they may induce global and localized geometric deformation of the substrates or leave an interfacial layer with significant thickness variation. For channels in the range from millimeter to a few hundred microns, these drawbacks are tolerable. However, it is implausible to construct micron and nanometer sized channels using these techniques since significant global and local material deformations may distort the micro/nano channel geometries. We have recently presented our work in using localized microwave heating to bond polymer (e.g., PMMA) substrates with a uniform interface layer about 1μm without causing any global deformations [2]. The operation of microwave bonding is convenient and well-controlled. However, microwave bonding can only be applied to a relatively small surface area between two bonding substrates, e.g., 1cm×1cm.

Polymer is the most common adhesive bonding material for microfluidic devices because the bonding temperature is relatively low. Benzocyclobutene (BCB) [3] and Teflon-like amorphous fluorocarbon polymer [4] are used as the adhesive layers to bond different materials such as silicon and glass. However, their required bonding temperature is still over 100°C. SU-8 is another polymer that requires bonding temperature of ~95°C [5]. For glass and silicon substrates bonding, bonding temperature over 100°C is still acceptable; but for polymer substrates, this would greatly affect the bonding performance. For example, the glass transition temperature of PMMA is only 105°C, hence, bonding temperature that is over 100°C cannot be applied to PMMA-to-PMMA bonding as it would melt the channel patterns on PMMA substrates.

PDMS, an elastomeric polymer, is becoming more popular among the microfluidic device community. Compared with other polymers, PDMS has a low glass transition temperature ($T_g \approx$ -125ºC [6]), a unique flexibility (the shear modulus G may vary between 100kPa and 3MPa [6]), high dielectric strength (~21kV/mm [7]), high gas permeability, high compressibility, usability over a wide temperature range (at least from -50ºC up to +200ºC [7]), low chemical reactivity (except at extremes of pH), and a non-toxic nature. It is also biocompatible. Devices such as cell sorter [8] and tissue scaffolds [9] were successfully made. Replica molding technique is commonly used to fabricate PDMS microfluidic devices [10]. The preparation process of PDMS is also simple. In addition, its low curing temperature (<100°C) makes it an excellent material for bonding polymer substrates since many polymer substrates cannot withstand a high bonding temperature (>200°C). Currently, PDMS is widely used as the structural material for microfluidic

devices because of its biocompatibility, low cost, and rapid prototyping properties. 3-D microchannels can be made easily and rapidly by replica molding method. An idea can be turned into a device in a few days. Typically, 3-D channels are formed by exposing both PDMS layers to oxygen plasma and then bond them immediately after the plasma treatment [10]. PDMS can be irreversibly adhered to a number of materials such as glass, silicon and quartz [11]. However, PDMS cannot be adhered to PMMA by this method. Instead of using the oxygen plasma treatment, we have developed a novel bonding method, which used spin-coated PDMS as the interface to bond two PMMA substrates during the curing of PDMS. This method is effective, low cost, fast, and simple to fabricate microchannels.

In this paper, we will present our recent progress in bonding PMMA substrates with large surface area (4cm×4cm) at low temperature using a thin spin-coated PDMS layer (10-25µm) as the intermediate layer. We found that PDMS could be made to adhere well to PMMA during the curing process of PDMS and no global deformation was generated in the substrates. We have fabricated a closed microchannel using this method. In our experimental results, the bonding strength was 0.015MPa and no leakage occurred inside the channel.

2. Previous attempts

2.1 Treatment of PDMS by oxygen plasma
Besides the bonding method we described above, we have tried another method to bond PMMA substrates together. Oxygen plasma treatment is commonly used to bond PDMS. It is reported that the bond between PDMS and glass using oxygen plasma can withstand a pressure of 0.2-0.3MPa [12]. We tried spinning on PDMS on both bare and embossed PMMA and cured them separately. For the embossed substrate, PDMS was spun on first before compressing the channel on it. PDMS was cured during the heating of the hot embossing process. The two substrates were treated with oxygen plasma with the conditions of 70W, 40Pa, and 30s. The two substrates were then bonded immediately under a pressure of 50kPa. After a few minutes, the sample was examined. We found that the two substrates did not adhere well. The bonded area was only 50%. It was caused by the non-uniformity of the PMMA surface and there was a gap between the two substrates. The contact surface area of the two PDMS layers was small. Therefore, only a weak adhesion force was produced.

2.2 Irreversible adhesion of PDMS and PMMA
Due to their chemical compositions, PDMS cannot adhere to PMMA irreversibly using oxygen plasma treatment. They can only make van der Waals contact with each other. However, we have found that by decreasing the mixing ratio between the prepolymer and the curing agent of PDMS and increasing its curing temperature, PDMS can adhere to PMMA substrate irreversibly. The mixing ratio of PDMS was reduced to 3:1 to decrease the thickness of PDMS layer. The spin-coated PDMS was cured at 170°C for 3 hours. PMMA changed to solid-rubber transition state due to the high temperature and made it adhere well to PDMS. However, this

method cannot apply to PMMA-PDMS-PMMA bonding because the required bonding temperature is above the glass transition temperature of PMMA (T_g=105°C), i.e., the channel patterns will be melted at this high bonding temperature. However, this method can be applied to other materials such as glass and silicon to PMMA bond.

3. Fabrication of microfluidic channel

3.1 Micro patterning of PMMA by hot embossing technique

Microchannels on PMMA were created by using the hot embossing technique similar to the one reported in [12]. The fabrication process used in our group is illustrated in Figure 1. A layer of silver was deposited on silicon substrate. SU-8 was then patterned on silver by lithography. Next, nickel was deposited on the SU-8 mold by electroplating to make a metal mold. Nickel was used as the material of the metal mold because it is much harder than PMMA (Young's modulus=200GPa). The metal mold was then released and inserted into the hot embossing machine. The hot embossing machine used in our lab and its components are shown in Figure 2. The PMMA substrate was first heated to 120°C, which was slightly above the glass transition temperature of PMMA (T_g=105°C). Then a pressure of 6.89MPa was applied by a hydraulic press to compress the mold towards the PMMA substrate, as shown in Figure 1c, which allowed the channel pattern on the metal mold to be transferred to the PMMA substrate. The substrate and the mold were then cooled and separated.

3.2 Formation of microchannel by PDMS bonding process

The fabrication process of microchannel on PMMA is illustrated in Figure 3. PDMS prepolymer (SYLGARD 184 Silicone Elastomer Kit, Dow Corning) was mixed with its curing agent in the volume ratio of 10:1. Then the prepolymer mixture was degassed in a desiccator with a vacuum pump at 50 torr for half an hour to remove any bubbles created during mixing. A 10-50µm PDMS prepolymer mixture was spun on the bare PMMA surface. The size of the PMMA substrates was 4cm wide, 4cm long and 0.2cm thick. After spinning on the PDMS, the substrate was pre-cured at room temperature first for about 20 hours to increase the viscosity of PDMS (originally: evaporate most of the solvents). The pre-curing time could be reduced by increasing the temperature. The thickness of PDMS was controlled by the spinning rate as shown in Figure 4. The spinning time was 40s. The two substrates were not bonded immediately because air could be trapped and bubbles could appear in PDMS layer. However, The PDMS layer was only partially cured after 20 hours. 24 hours is needed to fully cure PDMS at room temperature. This partially cured PDMS was very viscous and sticky, and was suitable for bonding. An example of two PMMA substrates successfully bonded is shown in Figure 5. The bonded substrates were heated at 90°C for 3 hours under a pressure of 50kPa. PDMS was thus completely cured and the channel was sealed.

4. Experimental results

4.1 SEM examination

The bonded sample was cut by a diamond saw and the bonding quality of the microchannel was examined. SEM pictures of the cross section of the PMMA-PDMS-PMMA interface are shown in Figure 6. In this figure, we found that the interfacial layer was precise and well-controlled. The thickness was approximately 50μm. PDMS at plain areas, as shown in Figure 6a, adheres well to both PMMA substrates. However, in Figure 6b, we found that PDMS was squeezed to the round edges of the channel. The round edges of the channel were created by the hot embossing process. When the PMMA substrate was heated to a temperature above its glass transition point and then subjected to a tensile stress, the molecules would tend to align themselves in the general direction of the stress. When the substrate was then cooled below its transition temperature while the molecule was still under stress, the molecules would become frozen whilst in an oriented state. As shown in Figure 7, the channel walls were curved due to the contact-stress between the mold and PMMA as we have shown in [13]. Nevertheless, no PDMS was squeezed into the channel to block it.

4.2 Tensile bonding test

The bonding strength test was performed by using the QTest ^{TM} tensile strength tester from MTS Systems Corporation. The test set up is shown in Figure 8a. In order to fit the sample to the gripper of the machine, a piece of PMMA attachment substrate was adhered to both the top and bottom surfaces of the sample as shown in Figure 8b. Chloroform was used to attach this attachment substrate to the samples. The evaluation results with various parameters are listed in Table 1. The bonding strength was about 0.015MPa. The results show that fewer bubbles formed with a thinner PDMS layer. Besides the thickness of PDMS layer, the pre-curing time of PDMS at room temperature also has a significant influence on the bonding quality. Sufficient pre-curing time (~20 hours) is needed to reduce bubble formation and achieves a larger bonded area. A larger bonded area leads to a stronger bonding strength.

4.3 Leakage test

The most common concern about microfluidic system is the leakage problem. Many existing polymer-to-polymer substrate bonding methods such as gluing by epoxy or methanol suffered from uneven bonding and leakage near the edge of the device. Therefore, our fabricated channels were tested for leakage. Since both PMMA and PDMS are transparent, it is difficult to examine the bonding quality by human eyes. Color dye was pumped into the channels at a flow rate 0.8ml/min, and no leakage occurred in the channels as shown in Figure 9. The channel dimensions in Figure 9a and b are w=1mm, h=1mm, l=3.4cm (true length) and w=300μm, h=100μm, l=1.6cm, respectively.

5. Conclusions

A low temperature bonding technique for polymer-based substrates to achieve a precise and well-controlled bonding interfacial layer has been presented. The bonding technique, using spin-coated PDMS, shows a low bonding temperature (~90°C) and bonding strength of 0.015MPa in

PMMA-PDMS-PMMA interface. The PMMA substrates were bonded without any global geometric deformation. The bonded substrates were tested with tensile bonding test and leakage test. Results of tensile bonding test showed that thickness of the interfacial layer and pre-curing time of PDMS at room temperature were critical for realizing good bonding quality. Color dyes were injected into a closed channel to show that no leakage occurred. We have demonstrated an effective, low cost, fast and simple way to fabricate polymer microchannels at relatively low temperatures.

Acknowledgements

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Table 1. Evaluation results of the bonding tests.

Sample No.	PDMS thickness (µm)	Curing time at room temperature (hr)	Bonding temperature (°C)	Bonding time (hr)	Bonding strength (MPa)	Bonded area (%)	Bubbles formed
1	10	20	90	3	0.015689	100	No
2	25	20	90	3	0.015389	95	Yes
3	35	20	90	3	0.014711	95	Yes
4	10	6	90	1.5	0.011922	90	Yes
5	25	6	90	1.5	0.009900	85	Yes
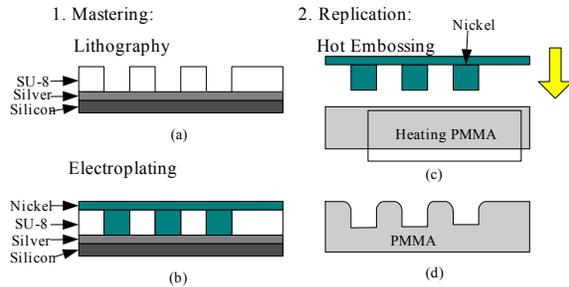
Figure 1. Illustration of the hot-embossing process. Two main procedures: Mastering and replication. (a) SU-8 was patterned on silver by lithography. (b) Nickel was electroplated on the SU-8 mold to make the metal mold. (c) Nickel mold was released and compressed on the heated PMMA substrate. (d) Channel pattern on the nickel mold was transferred to the PMMA substrate.
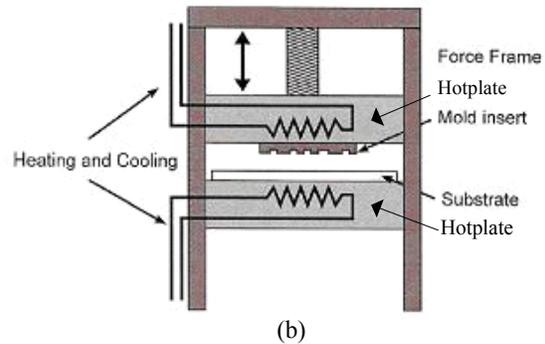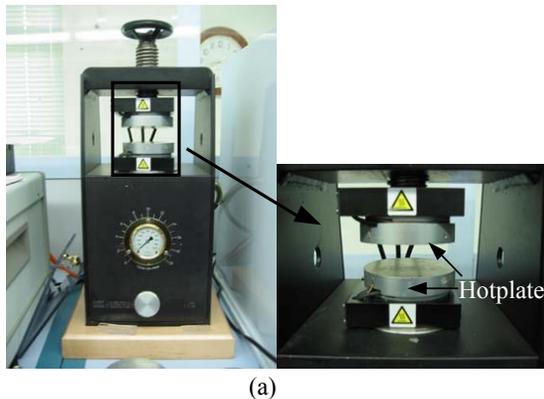
Figure 2. Hot embossing machine for compressing channel patterns on PMMA. (a) Photo of the machine. (b) Schematic diagram of the machine.
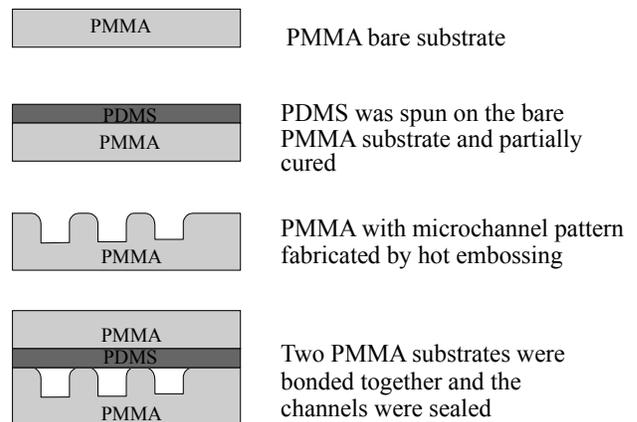
Figure 3. Fabrication process of closed micro structures.
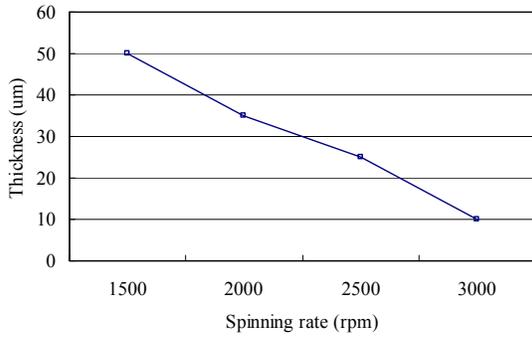
**Figure 4.** Thickness of spin-coated PDMS versus spinning rate.
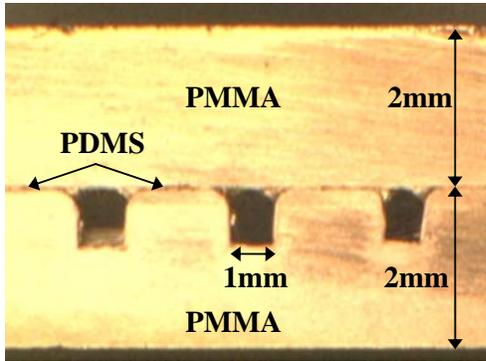
Figure 5. Microscopic cross-sectional view of microchannels formed between 2 PMMA substrates.
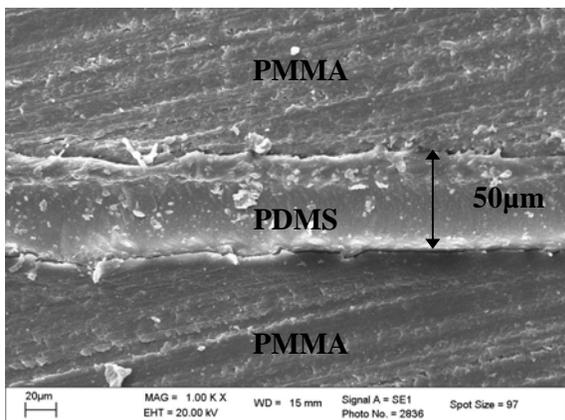
(a)
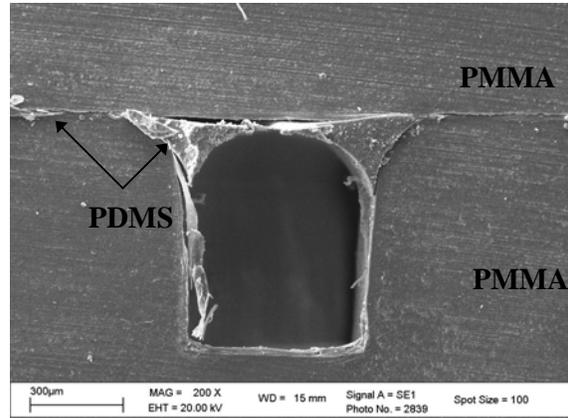
(b)

Figure 6. SEM images of the cross-section of the PMMA-to-PMMA bonding interface. (a) Interface between two plain areas showing that PDMS bonds well to both PMMA substrates. (b) Interface at channel. PDMS was squeezed to the round edges of the channel.
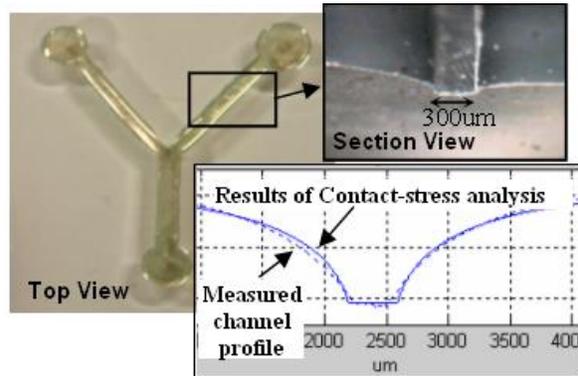

Figure 7. 3D microscope pictures of a PMMA molded with microchannels using the hot embossing process. As shown in the insets on the right, the channel walls are curved due to contact-stress between the mold and PMMA [13].
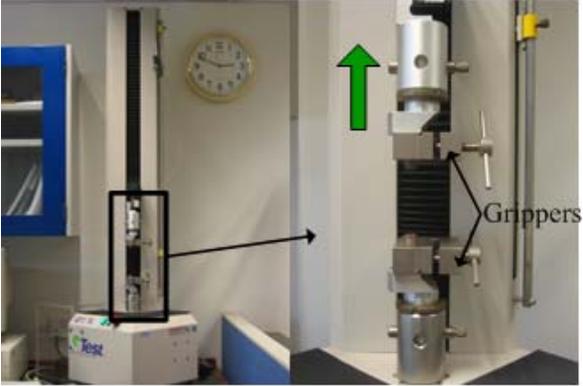
(a)
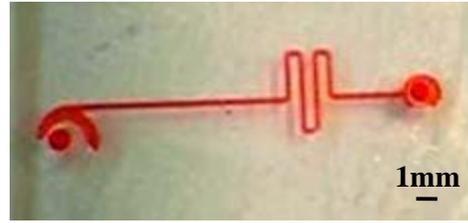
(b)

Figure 9. Channels with color dyes injected showing that no leakage occurred. (a) A curved channel. (b) A winding micro channel.
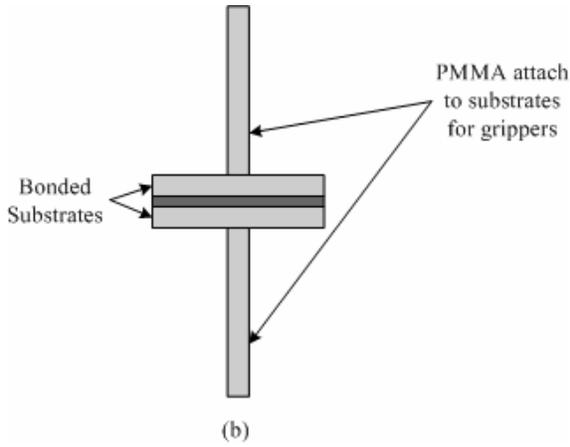
(b)

Figure 8. Experimental setup of the tensile bonding test. (a) Photo of the QTest ^{TM} tensile testing machine. (b) Two PMMAs were mounted to the top and bottom surfaces of the bonded substrates to fit the grippers of the machine.

(a)