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Microfluidic channel fabrication by PDMS-interface bonding

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Abstract

A novel technique for bonding polymer substrates using PDMS-interface bonding is presented in this paper. This novel bonding technique holds promise for achieving precise, well-controlled, low temperature bonding of microfluidic channels. A thin (10–25 μm) 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 a hot embossing technique first. Then, PDMS was spin-coated onto another PMMA bare substrate and cured in two stages. In the first stage, it was pre-cured at room temperature for 20 h 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 h and the bonding was successfully achieved at this relatively low temperature. Tensile bonding tests showed that the bonding strength was about 0.015 MPa. Microfluidic channels with dimensions of 300 μm \times 1.6 cm \times 100 μm were successfully fabricated using this novel bonding method.

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

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, ultrasonics, 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 millimetres to a few hundred

microns, these drawbacks are tolerable. However, constructing micron and nanometre sized channels using these techniques is implausible since significant global and local material deformations may distort the micro/nanochannel geometries. We have recently presented our work on using localized microwave heating to bond polymer (e.g., PMMA) substrates with a uniform interface layer of about 1 μm 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., 1 cm \times 1 cm.

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 a bonding temperature of ~95 °C [5]. For glass and silicon substrate bonding, bonding temperatures over 100 °C are 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, a bonding temperature that is over 100 °C cannot be applied in 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 ($T_g \approx -125$ °C [6]), a unique flexural strength (the shear modulus G may vary between 100 kPa and 3 MPa [6]), high dielectric strength (~21 kV mm⁻¹ [7]), high gas permeability, high compressibility, usability over a wide temperature range (at least from -50 °C up to +200 °C [7]), low chemical reactivity (except at extremes of pH levels), and a non-toxic nature. It is also biocompatible. Devices such as cell sorters [8] and tissue scaffolds [9] were successfully made using PDMS. A replica moulding technique is commonly used to fabricate PDMS microfluidic devices [10]. The process of preparation 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 replicability properties. 3D microchannels can be made easily and rapidly by the replica moulding method; i.e., a microchannel design can be turned into a real device in a few days. Typically, 3D channels are formed by exposing both PDMS layers to oxygen plasma and then bonding 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 use to fabricate microchannels.

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

2. Previous attempts

2.1. Treatment of PDMS with oxygen plasma

Besides the bonding method we described above, we have tried another method for bonding PMMA substrates together.

Oxygen plasma treatment is commonly used to bond PDMS. It is reported that the bond between PDMS and glass obtained using oxygen plasma can withstand a pressure of 0.2–0.3 MPa [12]. We tried spinning PDMS onto 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 an RF power of 70 W, pressure at 40 Pa, and a treatment time of 30 s. The two substrates were then bonded immediately under a pressure of 50 kPa. After a few minutes, the sample was examined. We found that the two substrates did not adhere well. The bonded area was only 50%. This 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 be adhered to PMMA irreversibly using an oxygen plasma treatment. The systems can only make van der Waals contact with each other. However, we have found that by decreasing the mixing ratio of the prepolymer and the curing agent of PDMS and increasing the curing temperature, PDMS can be made to adhere to the PMMA substrate irreversibly. The mixing ratio of PDMS was reduced to 3:1 to decrease the thickness of the PDMS layer. Then, the PDMS was on top of the PMMA and was cured at 170 °C for 3 h. PMMA changed to a solid-rubber transition state due to the high temperature and this made it adhere well to the PDMS. However, this method cannot be applied in 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 in PMMA bonding.

3. Fabrication of a microfluidic channel

3.1. Micropatterning of PMMA by a hot embossing technique

Microchannels on PMMA were created by using a 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 a silicon substrate. SU-8 was then patterned on the silver by lithography. Next, nickel was deposited on the SU-8 mould by electroplating to make a metal mould. Nickel was used as the material of the metal mould because it is much harder than PMMA (Young's modulus = 200 GPa). The metal mould was then released and inserted into the hot embossing machine. The hot embossing machine used in our laboratory 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 6.89 MPa was applied by a hydraulic press to compress the mould towards the PMMA substrate, as shown in figure 1(c), which allowed the channel pattern on the metal mould to be transferred to the PMMA substrate. The substrate and the mould were then cooled and separated.

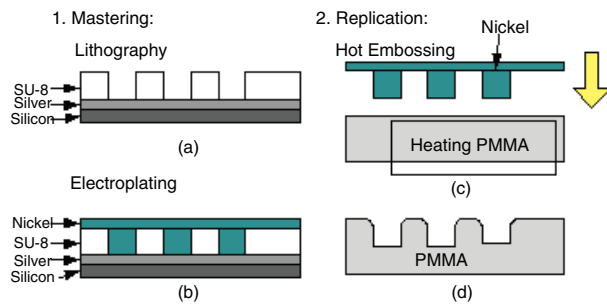


Figure 1. Illustration of the hot embossing process. Two main procedures: mastering and replication. (a) SU-8 was patterned on silver by lithography. (b) Nickel was electroplated on the SU-8 mould to make the metal mould. (c) The nickel mould was released and compressed on the heated PMMA substrate. (d) The channel pattern on the nickel mould was transferred to the PMMA substrate.

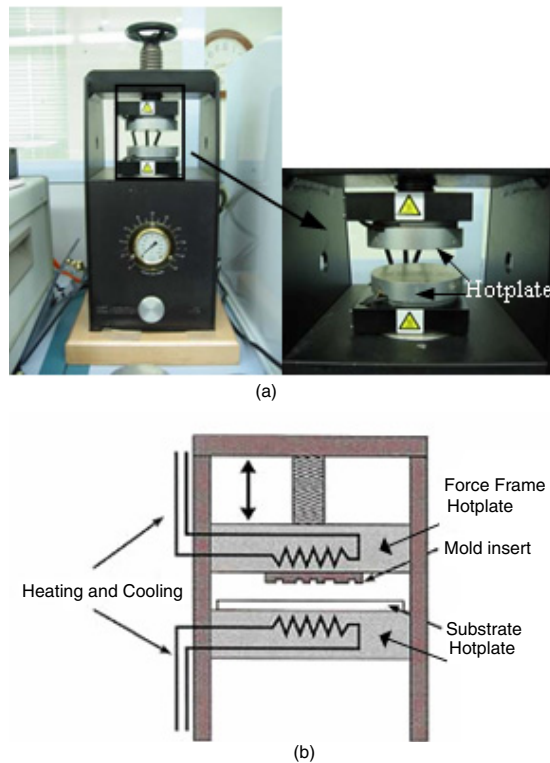


Figure 2. Hot embossing machine for compressing channel patterns on PMMA. (a) Photograph of the machine. (b) Schematic diagram of the machine.

3.2. Formation of microchannel by the PDMS bonding process

The process of fabrication of a 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 10–50 μm PDMS prepolymer mixture was spun onto the bare PMMA surface. The PMMA substrates were 4 cm wide, 4 cm long, and 0.2 cm thick. After spinning on

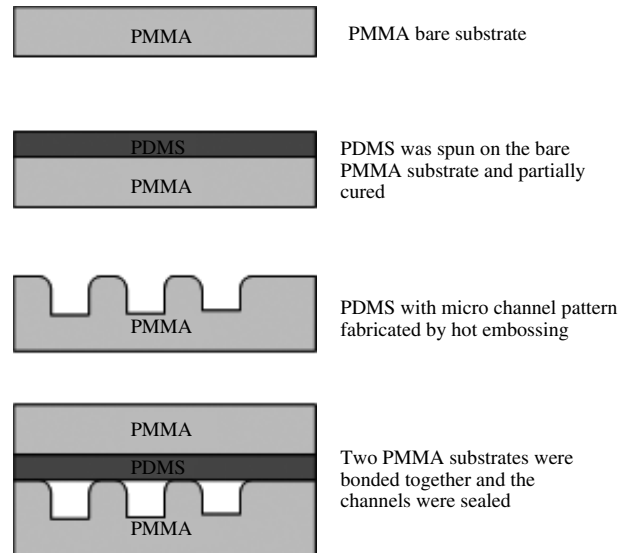


Figure 3. Process of fabrication of closed microstructures.

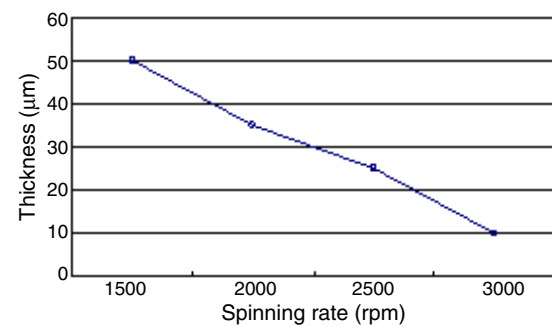


Figure 4. Thickness of spin-coated PDMS versus spinning rate.

the PDMS, the substrate was pre-cured at room temperature first for about 20 h to increase the viscosity of PDMS. The pre-curing time could be reduced by increasing the temperature. The thickness of the PDMS was controlled by the spinning rate as shown in figure 4. The spinning time was 40 s. The two substrates were not bonded immediately because air could be trapped and bubbles could appear in the PDMS layer. However, the PDMS layer was only partially cured after 20 h. 24 h 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 $^{\circ}\text{C}$ for 3 h under a pressure of 50 kPa. 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

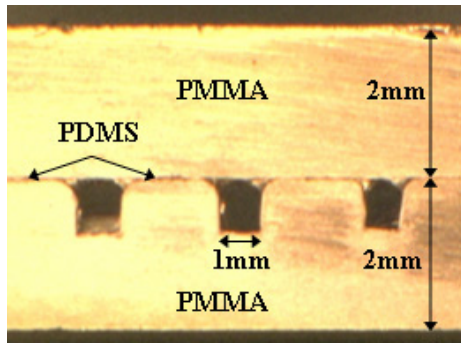


Figure 5. Microscopic cross-sectional view of microchannels formed between two PMMA substrates.

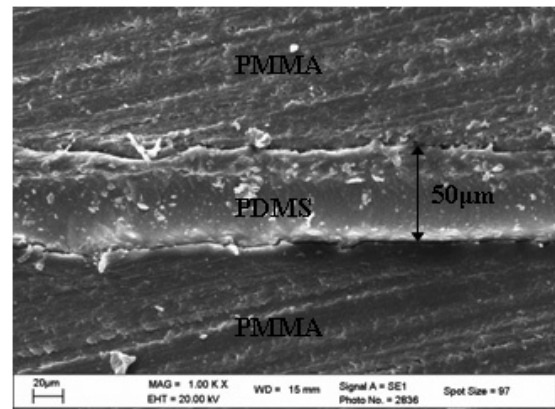
approximately $50\ \mu\text{m}$. PDMS in plain areas, as shown in figure 6(a), adheres well to both PMMA substrates. However, in figure 6(b), we found that some PDMS was squeezed to the round edges of the channel. The round edges of the channel were created by the hot embossing process. This happens because when the PMMA substrate is heated to a temperature above its glass transition point and then subjected to a tensile stress, the molecules will tend to align themselves in the general direction of the stress. When the substrate was then cooled to 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 of the mould and the PMMA as we have shown in [13]. Nevertheless, no PDMS was squeezed into the channel to block it.

4.2. Tensile bonding test

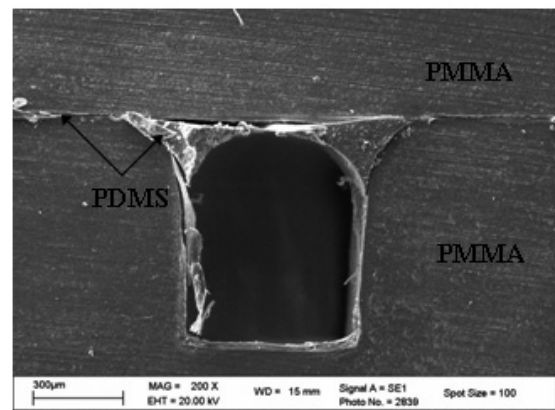
The bonding strength test was performed by using the QTestTM tensile strength tester from MTS Systems Corporation. The test set-up is shown in figure 8(a). In order to fit the sample to the gripper of the machine, a piece of PMMA attachment substrate was adhered to both the top and bottom surfaces of the sample as shown in figure 8(b). 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 0.015 MPa. 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 ($\sim 20\ \text{h}$) is needed to reduce bubble formation and achieve a larger bonded area. A larger bonded area leads to a greater bonding strength.

4.3. Leakage test

The most common concern for microfluidic systems is the leakage problem. Many existing polymer-to-polymer substrate bonding methods such as gluing with 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 with the naked eye. Colour dye was pumped into the channels at a flow rate of $0.8\ \text{ml min}^{-1}$, and no leakage occurred in the



(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 the channel. PDMS was squeezed to the round edges of the channel.

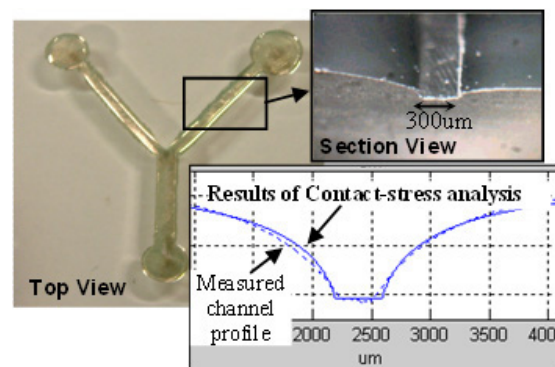
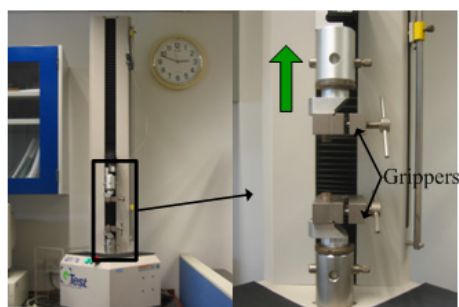


Figure 7. 3D microscope pictures of a PMMA moulded with microchannels using the hot embossing process. As shown in the insets on the right, the channel walls are curved due to contact stress of the mould and the PMMA [13].

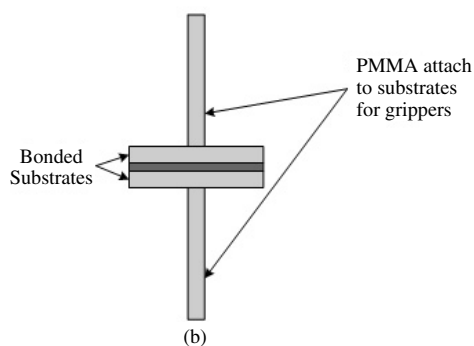
channels, as shown in figure 9. The channel dimensions in figures 9(a) and (b) are $w = 1\ \text{mm}$, $h = 1\ \text{mm}$, $l = 3.4\ \text{cm}$ (total length of curve) and $w = 300\ \mu\text{m}$, $h = 100\ \mu\text{m}$, $l = 1.6\ \text{cm}$, respectively.

Table 1. Evaluation results of the bonding tests.

Sample No	PDMS thickness (μm)	Curing time at room temperature (h)	Bonding temperature ($^{\circ}\text{C}$)	Bonding time (h)	Bonding strength (MPa)	Bonded area (%)	Bubbles formed
1	10	20	90	3	0.015 689	100	No
2	25	20	90	3	0.015 389	95	Yes
3	35	20	90	3	0.014 711	95	Yes
4	10	6	90	1.5	0.011 922	90	Yes
5	25	6	90	1.5	0.009 900	85	Yes



(a)

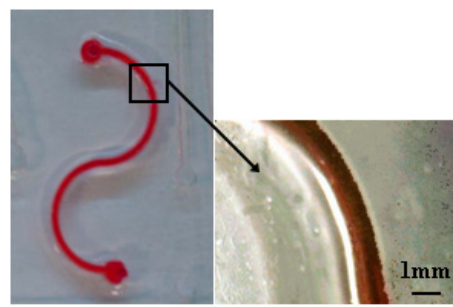


(b)

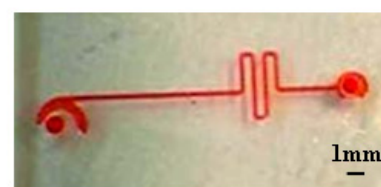
Figure 8. Experimental set-up of the tensile bonding test. (a) Photograph of the QTest™ tensile testing machine. (b) Two PMMAs were mounted on the top and bottom surfaces of the bonded substrates to fit the grippers of the machine.

5. Conclusions

A low temperature technique for bonding 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 ($\sim 90^{\circ}\text{C}$) and bonding strength of 0.015 MPa in the PMMA–PDMS–PMMA interface. The PMMA substrates were bonded without any global geometric deformation. The bonded substrates were tested with a tensile bonding test and a leakage test. The results of the tensile bonding test showed that the thickness of the interfacial layer and the pre-curing time for PDMS at room temperature were critical for realizing good bonding quality. Colour 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.



(a)



(b)

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

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