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Thermal assisted ultrasonic bonding method for poly(methyl methacrylate) (PMMA) microfluidic devices

Zongbo Zhang [a], Xiaodong Wang [b], Yi Luo [b,*], Shengqiang He [a], Liding Wang [b]

[a] Key Laboratory for Precision and Non-traditional Machining Technology of Ministry of Education, Dalian University of Technology, Dalian, China
[b] Key Laboratory for Micro/Nano Technology and System of Liaoning Province, Dalian University of Technology, Dalian, China

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ABSTRACT

A thermal assisted ultrasonic bonding method for poly(methyl methacrylate) (PMMA) microfluidic devices has been presented. The substrates were preheated to 20–30 °C lower than glass transition temperature (T_g) of the polymer. Then low amplitude ultrasonic vibration was employed to generate facial heat at the interface of PMMA substrates. PMMA microfluidic chips were successfully bonded with bulk temperature well below T_g of the material and with pressure two orders lower than conventional thermal bonding, which was of great benefit to reduce the deformation of microstructures. The bonding process was optimized by Taguchi method. This bonding technique showed numerous superiorities including high bonding strength (0.95 MPa), low dimension loss (0.3–0.8%) and short bonding time. Finally, a micromixer was successfully bonded by this method and its performance was demonstrated.
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1. Introduction

Microfluidic devices have gained considerable interest due to their advantages such as high-speed, high-throughput, small sample and reagent requirements and potential portability. These devices were initially fabricated using glass or silicon. However, in the last decade, polymers were increasingly employed as substrates of microfluidic devices due to their superiorities of low cost, disposability, ease of fabrication and wide choices of materials [1]. As a result of low rigidity for most polymer materials, a challenging step in the fabrication of polymer-based microfluidic device is bonding and sealing the micro-channels or chambers without deformation of the microstructures. Typical requirements for bonding microfluidic devices are liquid tightness at the joint interface, absence of foreign substances and small deformation of microstructures. Many techniques for bonding polymer microfluidic devices were studied in the past decade including thermal bonding [2], adhesive bonding [3], solvent bonding [4] and so on. But the bonding demands could hardly be satisfied simultaneously.

Among the bonding methods for polymer microfluidic devices, thermal bonding was the most common method since it was straightforward and introduced no foreign substances, resulting in homogeneous surfaces of microstructures [5]. However, in conventional direct thermal bonding, the bonding temperature was kept around or above T_g of the polymer. And relatively high pressure was applied to achieve satisfactory bonding strength. Although strong bonding could form under high temperature and pressure, microstructures readily deformed or even collapsed in this case. There was a tradeoff between bonding strength and structure holding in conventional thermal bonding method. To guarantee the integrity of microstructures, thermal bonding with lower bonding temperature or pressure is desired. Plasma or X-ray was used to modify the surface of the substrate before bonding [6,7]. Bonding with satisfied strength was achieved with temperature well below T_g of the substrate. But in this method, bonding step had to be carried out immediately after irradiation as the surface properties of the polymer changed quickly with time and was very sensitive to the humidity of environment. Moreover, it usually took tens of minutes to get satisfactory bonding strength.

Ultrasonic plastic welding is a common non-adhesive and non-mechanical method for bonding macroscale thermoplastic devices. The main advantages of this method are fast bonding speed and well located heating at specific regions. Ultrasonic was firstly used for bonding of polymer microfluidic devices in 2006 [8]. Microchannels with dimensions of 500 μm × 500 μm were successfully sealed. Recently, researchers used ultrasonic method for bonding of polymer microfluidic connectors with internal diameter of 1 mm [9,10]. These applications were all derived from ultrasonic plastic welding technique in which the specimens were connected by the molten polymer. However, it was difficult to control the flow profile of the molten polymer in several microns. With the minimization of microstructures, they were apt to be damaged by the molten polymer. Additionally, convex structures named energy director were usually required in conventional ultrasonic bonding

* Corresponding author. Tel.: +86 411 84707713x2191; fax: +86 411 84707940.
E-mail address: luoy@dlut.edu.cn (Y. Luo).

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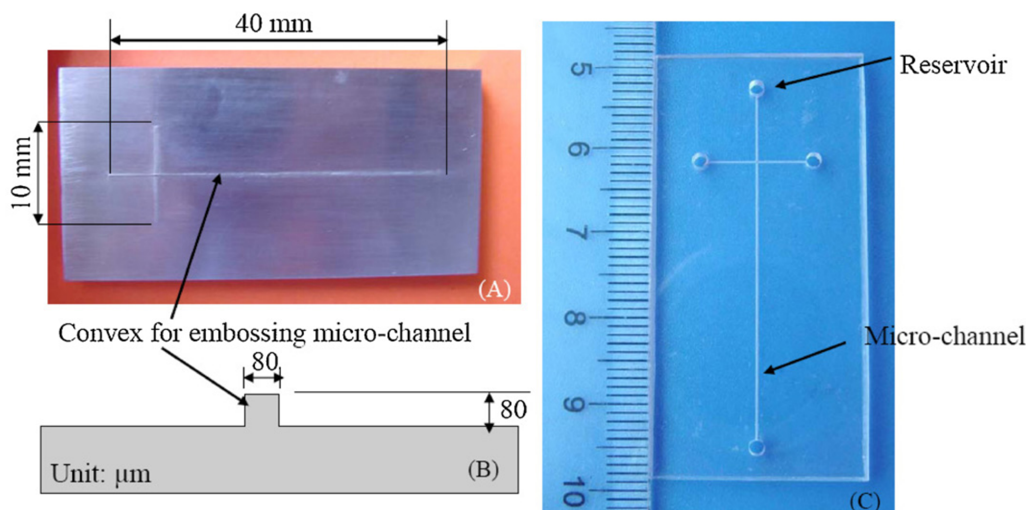

Fig. 1. Mold and PMMA substrate in hot embossing (A) the mold used in hot embossing, (B) schematic cross-section view of the mold and (C) the substrate with micro-channels and reservoirs on it.

for large area bonding. These newly introduced energy directors greatly complicated the fabricating process of the devices. The concept of ultrasonic bonding for polymer devices without melting of polymer has never been reported yet. It is an attractive idea for bonding of polymer MEMS devices. In ultrasonic bonding, interfacial heating mechanisms make temperature on the interface rise faster and higher than the inner parts of the specimen [11]. Combined with the interfacial heating mechanisms during ultrasonic bonding and the poor heat conductivity of polymer, it is possible to make the temperature at the interface rise while keep the bulk temperature of the polymer relatively low.

In this study, a thermal assisted ultrasonic bonding method without melting of polymer was presented. A hot plate was used to preheat the polymer substrates to 20–30 °C below T_g of the material. Then low amplitude ultrasonic was employed to generate facial heat at the interface of the substrates. Temperature of the interface rose to around T_g of the material while the bulk temperature of the substrates was still well below T_g. Combined with ultrasonically oscillating pressure, bonding formed at the interface with little deformation of microstructures. Bonding experiments of PMMA microfluidic devices were carried out. The bonding parameters were studied and optimized by Taguchi method. In order to show further utility of this method, a PMMA micromixer was bonded and its performance was demonstrated.

2. Experiments

2.1. Substrates preparation and bonding process

In this study, PMMA plate (Optix Acrylic Sheet, Asahi Kasei Corporation, Shanghai, China) with thickness of 2 mm was cut into 50 mm × 25 mm. T_g of the material was 105 °C. In order to show the dimension loss of microstructures, micro-channels were fabricated on the substrates using a hot embossing machine (RYJ-II, Dalian University of Technology, China). A steel mold fabricated by micro-electrical discharge machining (micro-EDM) method was used for embossing micro-channels on the substrates. The cross-section of the micro-channel was square-shaped with design dimensions of 80 μm × 80 μm, as shown in Fig. 1A and B. After embossing, reservoirs with diameter of 1 mm were drilled at the ends of micro-channels as shown in Fig. 1C.

The bonding process was performed on a standard ultrasonic welding system (2000× f/aef, Branson Company, USA), with generator frequency of 30 kHz. A temperature control module was fixed on the leveling anvil of the welding machine. Fig. 2 shows the schematic diagram of temperature control module. It mainly comprises of a copper hot plate for preheating the polymer substrates and a temperature controller (XMT, Shanghai Dezhao Instrumentation Company, China) for controlling of the temperature. A steel

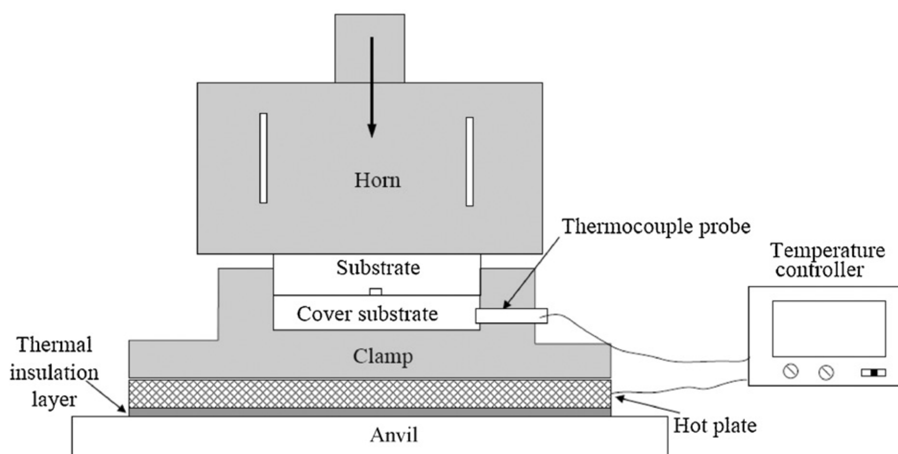

Fig. 2. Schematic of temperature control module.

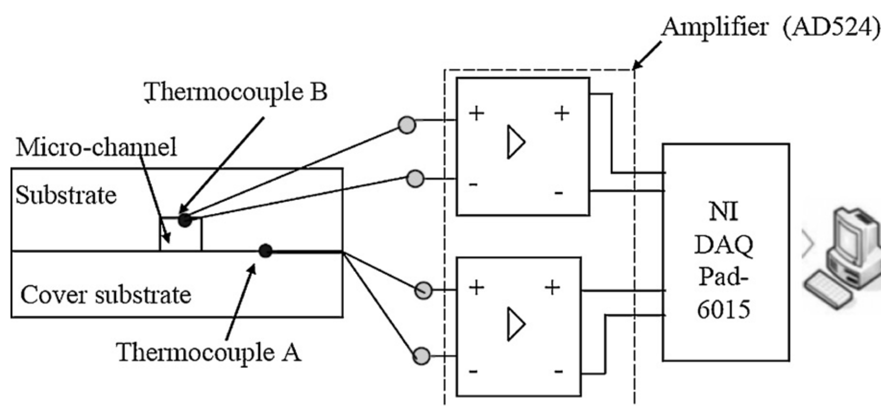

Fig. 3. Schematic of temperature test system.

clamp was fixed on the hot plate for clamping the substrates. Temperature curves were measured by a thermocouple probe in the clamp.

Before bonding, a blank PMMA cover substrate and a patterned substrate were fixed on the clamp. After leveling the horn of the welding machine went down with a speed of 50 mm/s. An increasing pressure was applied on the substrate until it reached the previously setting pressure. Then the substrates were preheated to a certain temperature which was 20–30 °C below T_g of the polymer by the temperature control module. Subsequently, ultrasonic vibration with previously setting amplitude was applied by the horn. After a period of ultrasonic bonding, the vibration closed and the pressure changed to holding pressure. Meanwhile, the chip was cooled down till its temperature was below 50 °C. Then the horn lifted and the bonding process was finished.

Previous researchers revealed that the temperature during ultrasonic bonding increased with the increase of ultrasonic amplitude [12]. And the temperature increase rate was relatively low when the temperature was below T_g of the polymer, which can make the temperature controlling easier [13]. So in order to avoid melting of the polymer, low amplitude ultrasonic was employed in this method. In ultrasonic bonding, interfacial heating mechanisms make temperature on the interface rise faster and higher than the bulk of the specimen [11]. Therefore, the ultrasonic vibration can make the temperature at the interface rise relatively high while keep the bulk temperature of the polymer low. Preliminary experiments indicated that the using substrates would not melt when the amplitude was below 10 μm. So amplitudes below 10 μm were used in the following experiments.

2.2. Temperature test

In order to study the heating characteristics during low amplitude ultrasonic bonding, temperature test was carried out at the interface (point A) and in the micro-channel (point B). The test was carried out at room temperature. Fig. 3 shows the temperature measuring system.

Rapid response thermocouples (Chal-0005, Omega Company, USA) with diameter of 12.7 μm were used. Responding time of the thermocouple was less than 10 μs. A thermocouple was embossed on the surface of substrate when micro-channels were fabricated by hot embossing method. Another thermocouple was fixed on the bottom of micro-channel using heat-conducting glue (Buddle 3401, Shenzhen Buddle Electronic Inc., China). Amplifier (AD524, Analog Devices Inc., USA) with magnifying ratio of 1000 was used to amplify the sampled signal. Then the signal was sampled by a multi-channel data acquisition board (NI DAQPad-6015, National Instruments, USA) with sample rate of 50 kHz per channel. Data acquisition program was developed using LabVIEW to realize signal sampling and processing in a computer. This system was calibrated by temperature calibrator (MKST TP 28850, Omega Company, USA) from 20 to 200 °C with a measuring error of ±2 °C. Amplitudes of 6 and 8.4 μm which were not able to make the substrates melt were tested.

2.3. Taguchi experiments

In this study, Taguchi method was employed to evaluate and optimize the bonding process. Four bonding parameters were chosen: preheating temperature *A*, bonding pressure *B*, bonding time *C* and ultrasonic amplitude *D*. Each of them was analyzed at three levels. The $L_9(3^4)$ orthogonal array (OA) was adopted. Table 1 shows the factors and their levels in the experiments.

Small dimension loss and high bonding strength are important for bonding technique of polymer MEMS devices. So dimension loss and tensile strength were selected as evaluation indicators in the experiments. The depths of micro-channel before and after bonding were measured using a microscope (U-7VD, Olympus, Japan). Five locations for each bonded chip were measured and the average value was calculated as the evaluating indicator. Three bonded chips with the same bonding parameters were measured. The tensile strength was measured using tensile machine (MTS 5/G, Sintech Inc., USA). And two bonded chips with the same bonding parameters were measured as the evaluation indicator for bonding strength.

2.4. Application on bonding of micromixer

As a further evidence for utility of this new bonding method in microfluidic devices, a passive micromixer was designed, fabricated and tested. Micro-channels with 300 μm in width and 100 μm in depth were carved on a PMMA substrate by engraving machine (YB7310, Yan Bei Inc., Shanghai, China). The mixer contained three mixing units each of which consisted of five C-shaped mixing cells, as shown in Fig. 4. Two inlets and one outlet holes with diameter of 1 mm were drilled at the ends of the channels. Then it was bonded with a blank PMMA substrate using the optimized parameters achieved by Taguchi experiments.

Table 1
Levels and factors in the Taguchi experiments.

Factors/levels	*A* (°C)	*B* (MPa)	*C* (s)	*D* (μm)
1	75	0.08	20	6
2	80	0.16	25	7.2
3	85	0.24	30	8.4

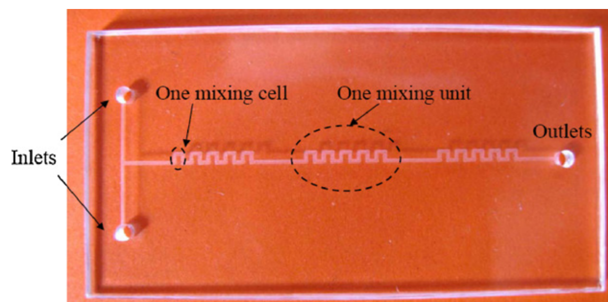

Fig. 4. Patterned substrate for micromixer.

In this paper, the mixing evaluation method employed by Wang [14] was used to evaluate the mixing performance. Blue dye (Water-dye 8836, Feda, Wenzhou, China) and DI water were injected into the two inlets by a syringe pump (WZS-50F6, Zhejiang University medical apparatus Inc., Hangzhou, China), as shown in Fig. 5. A CCD camera and a microscope were employed to characterize the mixing performance in the micro-channel by the color intensity of dyed water. The images were captured and processed in a computer.

The mixing extent of the dye and water in the micro-channel can be obtained by the color distribution. Before mixing experiment, solution of blue dye in ID water with different concentrations was injected into the mixer. The concentrations were from 0 to 100% with increment of 10%. The images of the solution with different concentration in micro-channels were captured. Then the RBG values of the images were obtained. The blue value of RBG was dependent on the concentration of the dye. So the relationship between the blue value of RBG and the concentration of the solution was calibrated. According to the calibration results and the captured images in the mixing experiment, the mixing performance in the micro-channels can be calculated.

Generally, mixing performance in microfluidic systems is typically dominated by diffusion instead of turbulence due to the low Reynolds number in the micro-scale channels. In order to show the changes from diffusion flow to turbulence flow in the mixing process, a wide flow rate range was tested. The performance of micromixer with flow rates of 2, 20, 400 and 1200 μl/min were demonstrated in the experiments.

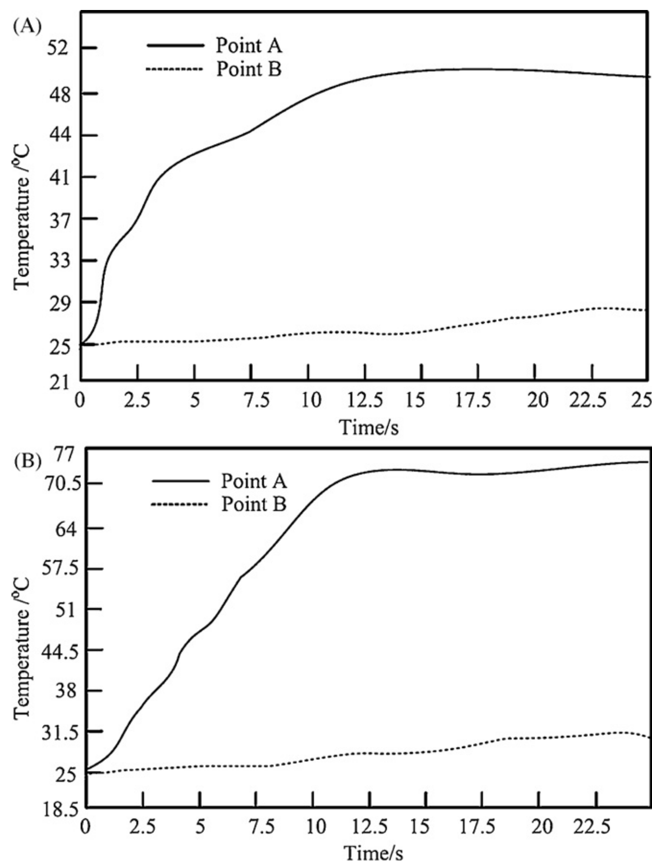

Fig. 6. Temperature profile with different amplitudes (A) 6 μm and (B) 8.4 μm.

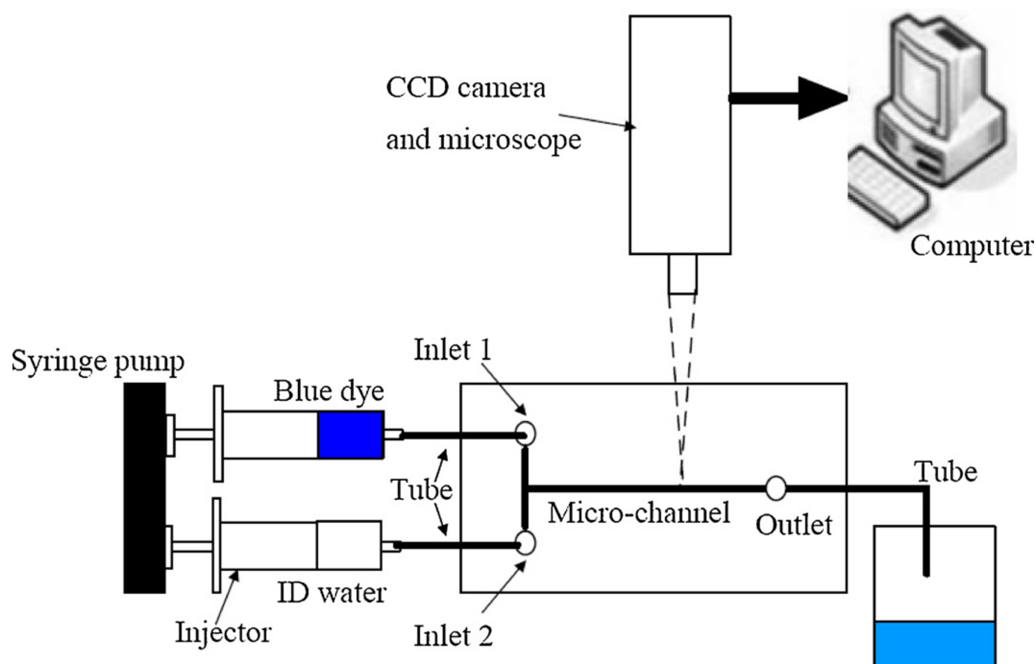

Fig. 5. Experimental setup of mixing test.

Table 2
Orthogonal array experimental data and SN ratio of dimension loss test.

Experiment No.	*A*	*B*	*C*	*D*	Experiment data (μm)			Dimension loss ratio (%)	SN ratio (dB)
					*y*1	*y*2	*y*3		
1	1	1	1	1	0.8	1.4	1.7	1.6	−7.1
2	1	2	2	2	0.4	0.6	1.1	0.9	−2.2
3	1	3	3	3	2.3	2	2	2.6	−10.3
4	2	1	2	3	2.1	2.6	1.9	2.8	−11
5	2	2	3	1	3.3	3	2.1	3.5	−12.8
6	2	3	1	2	1.6	2.2	1.6	2.3	−9.4
7	3	1	3	2	6.3	6.1	3.2	6.5	−18.3
8	3	2	1	3	5.8	6.8	6.6	7.8	−20.3
9	3	3	2	1	12.2	9.4	8.7	12.6	−23.8

Table 3
Summation of SN ratios at different levels and their main effects for dimension loss.

Levels/factors	1	2	3	Main effect
A	−6.5	−11.1	−20.8	14.3
B	−12.1	−11.8	−14.5	2.7
C	−12.3	−12.6	−13.8	1.5
D	−10	−13.6	−14.9	4.9

Table 4
Summation of SN ratios at different levels and their main effects for bonding strength tests.

Levels/factors	1	2	3	Main effect
A	13.2	13.3	16.9	3.7
B	12.8	14.7	15.8	3
C	12.5	18.2	12.6	5.7
D	13.1	14.3	15.9	2.8

3. Results and discussion

3.1. Heating characteristics of low amplitude ultrasonic bonding

Fig. 6 shows the temperature curves of point A and point B under bonding amplitudes of 6 and 8.4 μm, respectively. Temperature of the interface rises earlier and higher than that of the channel with both amplitudes. During the welding process, temperature of point A increases by about 25 °C with amplitude of 6 μm while it increases by around 45 °C with amplitude of 8.4 μm in 10 s. Then the heat generation and thermal conduction effects come to a balance. And the temperature does not rise any more. The tiny increase of temperature at point B was mainly caused by thermal conduction from the interface. And as a result of poor heat conductivity of PMMA, the increases of temperature at point B are below 5 °C for both amplitudes, as shown in Fig. 6.

Based on the facial heating phenomenon, it is reasonable to preheat the substrates to 20–30 °C below T_g and use low amplitude ultrasonic to heat up the interface of the substrates to about T_g while keeping the bulk temperature of substrates still well below T_g. Combined with ultrasonically oscillating pressure, bonding forms at the interface with little deformation of microstructures resulting from low bulk temperature.

3.2. Bonding results

Depth loss is a "the-smaller-the-better" evaluation indicator. So the signal-to-noise ratio (SN) is calculated using Eq. (1). Table 2 presents the SN ratios of depth loss in Taguchi experiments:

$$\text{SN ratio (dB)} = -10\log\left[\frac{1}{2}\sum_{i=1}^{n} y_i^2\right] \quad (1)$$

where y_i is the depth loss of the ith measuring location and n is the total measuring number.

The tensile strength is a "the-larger-the-better" evaluation indicator. So the SN ratio is calculated using Eq. (2):

$$\text{SN ratio (dB)} = -10\log\left[\frac{1}{2}\sum_{i=1}^{n}\frac{1}{y_i^2}\right] \quad (2)$$

The Taguchi experiment results of tensile strength are shown in Table 2.

Table 3 and Fig. 7 show the summation of SN ratios of dimension loss for various factors and different levels. It is clear that parameter *A* is more prominent than the other parameters. And parameters *D*,

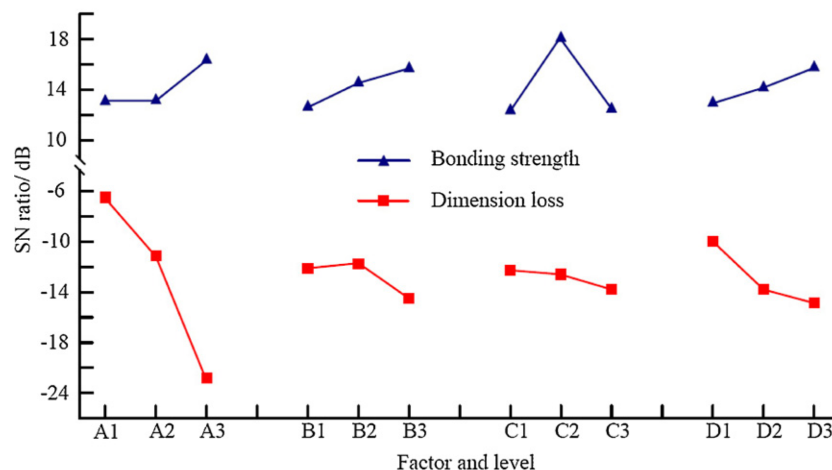

Fig. 7. SN ratios of various parameters at different levels from bonding strength and dimension loss tests.

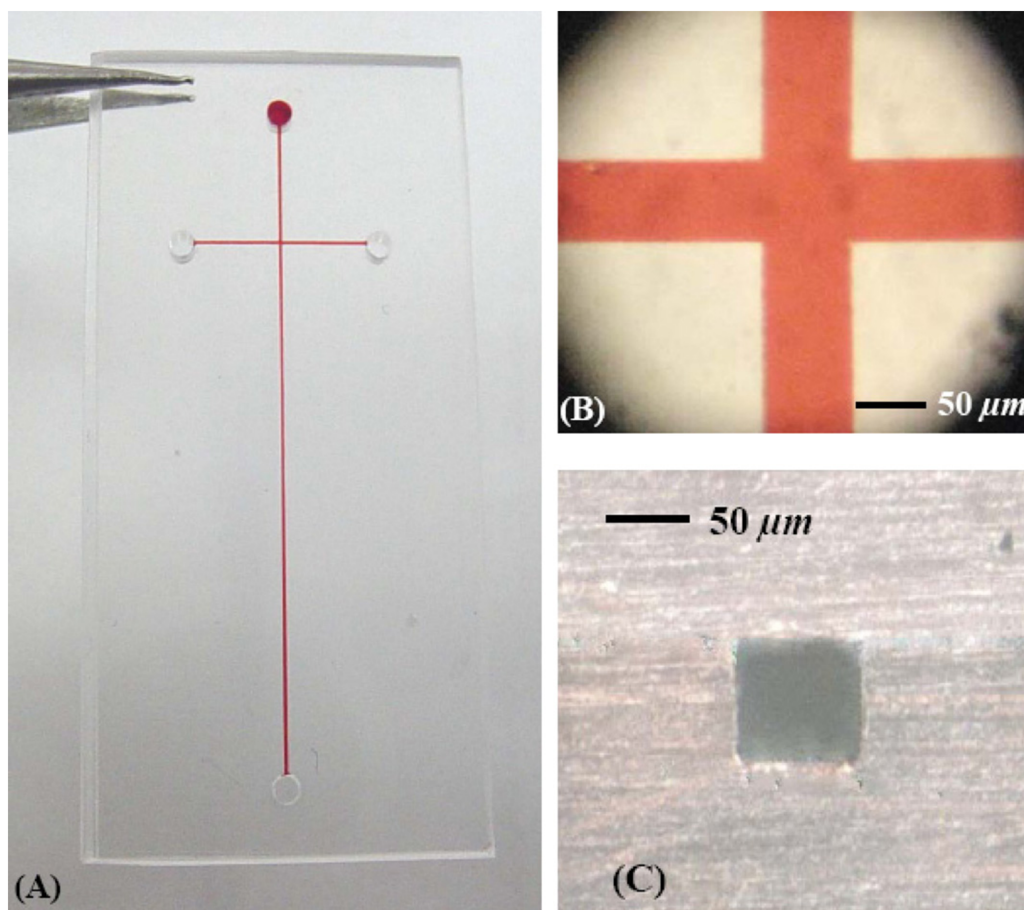

Fig. 8. The bonded chip (A) overall appearance of the bonded chip with red dye in the micro-channel, (B) micrograph of the bonded channel and (C) cross-sectional micrograph of the bonded channel.

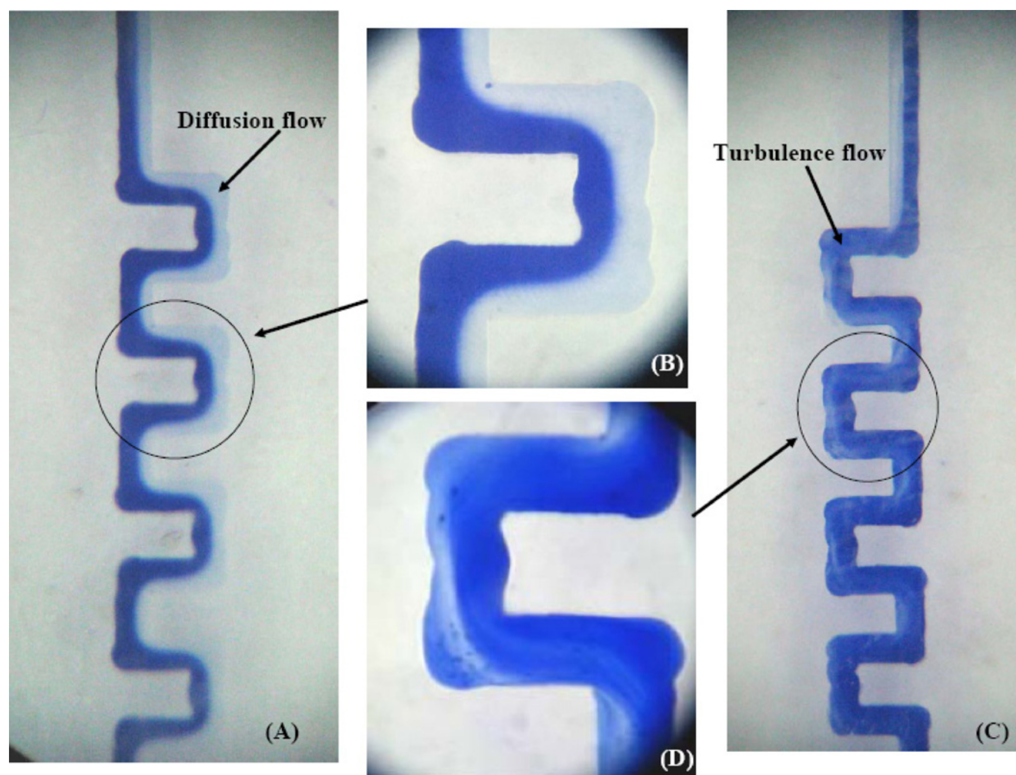

Fig. 9. Results of mixing experiment: (A) captured video frame of the first mixing unit with flow rate of 2 μl/min, (B) micrograph of one mixing cell with flow rate of 2 μl/min, (C) captured video frame of the first mixing unit with flow rate of 1200 μl/min and (D) micrograph of one mixing cell with flow rate of 1200 μl/min.

Table 5
Comparison of bonding strength and several bonding parameters with other thermal bonding methods of PMMA microfluidic devices.

Bonding methods	Bonding time	Preheating temperature (°C)	Bonding pressure (MPa)	Tensile strength (MPa)
Thermal assisted ultrasonic bonding	30 s	75	0.16	0.95
Vacuum thermal bonding [17]	60 min	112	3	0.15
Thermal bonding in water [18]	60 min	100	10	0.13
Thermal bonding with plasma modification [19]	30 min	85	18	0.6

B and *C* are the subsequent significant parameters. The depth loss is minimal at the first level for parameters *A*, *C*, *D* and at the second level for parameter *B*, as shown in Table 3. So the combination of *A*1, *B*2, *C*1 and *D*1 is the optimum combination of parameters for reducing the depth loss. And the minimum depth loss could reduce to 0.9%, which was even lower than that of 2–6% in solvent bonding method [4].

Table 4 and Fig. 7 show the summation of SN ratios of bonding strength for various factors and levels. The results indicate that parameter *C* is more prominent than other parameters. The results in Table 4 indicate that the combination of *A*3, *B*3, *C*2 and *D*3 is the best parameters for getting high bonding strength.

Based on the results above, parameters *A* and *D* significantly influence the dimension loss of microstructures while they are not so prominent for bonding strength. So the optimized values of these two parameters are determined by the results in Table 3. Parameter *C* is the most prominent parameter for bonding strength while it is the least prominent parameter for dimension loss. So it is determined by the results in Table 4. Different from conventional thermal bonding, neither dimension loss nor bonding strength is significantly affected by bonding pressure (parameter *B*). In our opinion, the reason is the difference of pressure values between the two methods. In conventional thermal bonding method the applied loading is usually between 3 and 18 MPa [15,16]. However, it is only 0.08–0.24 MPa in our method, which is 1–2 orders lower than that in conventional thermal bonding. In this method, oscillating pressure from the ultrasonic vibration instead of static pressure is applied. Combined with relatively high temperature at the interface, polymer chains mutually penetrate and entangle more easily over the boundary of neighboring substrate than in conventional thermal bonding. Taking both low dimension loss and high bonding strength into consideration, the optimum bonding parameters are determined as *A*1, *B*2, *C*2 and *D*1.

Microfluidic chips bonded with optimized parameters were tested. The tensile strength of six chips was measured using tensile machine (MTS 5/G, Sintech Inc., USA) with pulling speed of 0.1 mm/s. The average tensile strength is 0.95 MPa, which is much higher than the values reported using other thermal boding methods [17–19]. Some bonding parameters and the bonding strength of different thermal bonding methods are compared in Table 5. It indicates that the bonding temperature and pressure in this method are both much lower than other methods, which are of great benefit to preserve the integrity of microstructures. However, the bonding strength is 1.5–6 times higher than that of other thermal bonding methods. In our opinion, the high bonding strength is mainly attributed to relatively high surface temperature generated from ultrasonic vibration at the interface and the ultrasonically oscillating bonding pressure instead of static pressure. Moreover, the bonding time is less than 30 s, which is much shorter than that of other methods. During the bonding process, most time is consumed by preheating the substrates before bonding and cooling them after bonding. This problem can be improved by using more dedicated heating and cooling equipment [18]. Nevertheless, the whole bonding process takes less than 5 min using the current temperature control system.

Fig. 8 shows the overall appearance and details of the bonded chip with red dye (Water-dye 8836, Feda, Wenzhou, China) in the micro-channels. The straight borderline of the dye liquid in the micro-channels indicates that there is no clogging problem. And no evident deformation of the channel is observed from the cross-section image of the channel, as shown in Fig. 8C. In order to quantify the geometry of the channel, the chip was cut at six different locations. Depths and width of the cross-section were measured. The average dimension loss ratios are 0.8% in depth and 0.3% in width with relative standard deviation of less than 3%. It is much lower than that in conventional thermal bonding method [17–19]. The results demonstrate very low dimension loss and high size uniformity of micro-channels after bonding.

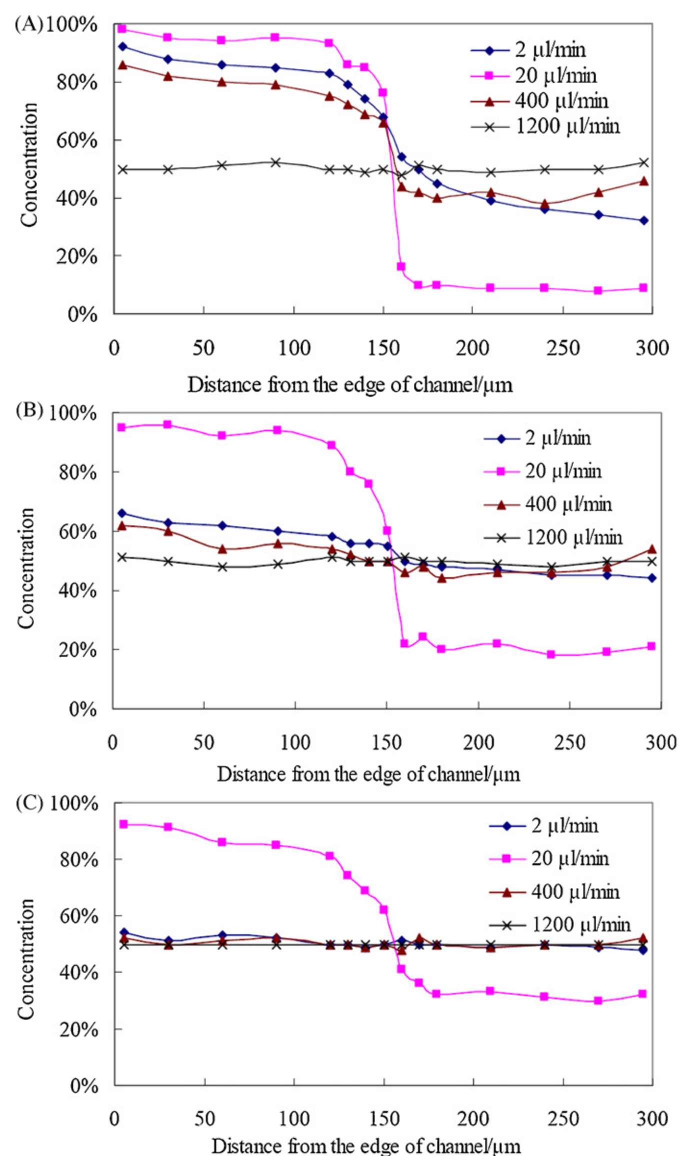

Fig. 10. Concentration distribution of the dye with different flow rates in the micro-channel (A) after the first mixing unit, (B) after the second mixing unit and (C) after the third mixing unit.

3.3. Performance of micromixer

The designed micromixer was bonded by the proposed bonding method. The channels were successfully sealed without any leakage occurred throughout the whole mixing experiment with various flow rates, as shown in Fig. 9.

The concentration distribution in the micro-channel with different flow rates after each mixing unit is shown in Fig. 10. The results indicate that the mixing performance becomes better along with the increase of mixing units at the same flow rate. And the mixing efficiency decreased with the increase of flow rate when the flow rate is low (below 20 μl/min) in the experiment. Due to the low Reynolds number resulting from low flow rate and small channel dimensions, mixing performance is dominated by diffusion in this case, as shown in Fig. 9A and B. So the mixing efficiency is proportional to the number of mixing units that the liquid flows through. However, when the flow rate is relatively high such as 400 and 1200 μl/min in the experiment, turbulence flow forms at the winding positions of the mixing cells, as shown in Fig. 9C and D. The turbulence phenomenon is more obvious with the increase of the flow rate. So the mixing efficiency increases with the increase of flow rate in this case. The dye and water are perfectly mixed only after one mixing unit when the flow rate is 1200 μl/min, as shown in Fig. 10A. It not only showed the validity of this new bonding method for polymer microfluidic devices but also illustrated the mixing performance of simple C-shaped micromixer with different flow rates.

4. Conclusions

A thermal assisted ultrasonic bonding method for polymer microfluidic devices was proposed in this paper. It was based on the characteristics of temperature distribution of polymer substrates under low amplitude ultrasonic. Temperature of the interface rose to around T_g as a result of facial heat effect of ultrasonic, while the bulk temperature of the substrates was still well below T_g. Combined with ultrasonically oscillating pressure, bonding formed at the interface with little deformation of microstructures. The bonding parameters were optimized using Taguchi's method. PMMA microfluidic chips were successfully bonded with high bonding strength and low dimension loss using this method. In order to give further evidence of the utility of this newly proposed method, a micromixer was successfully bonded and its diffusion and turbulence mixing performance with different flow rates was demonstrated.

Acknowledgements

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