



Low-cost hybrid bonding between thermoplastics and PDMS with differential adhesive tape for microfluidic devices

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ABSTRACT

Bonding is the key process in the fabrication of close-channel microfluidic devices. In the general fabrication approach of microfluidic devices, the substrate was processed with various kinds of microfabrication methods for the formation of the microchannel, then a cover plate (the same or different material to the substrate) was bonded to the substrate to enclose the microchannel. Various bonding methods have been previously reported which mainly focused on the bonding between thermoplastics or polydimethylsiloxane (PDMS)–glass bonding. In the past few years, the hybrid bonding between thermoplastics and PDMS was found to be useful to lower the cost and increase the flexibility of PDMS-based microfluidics, and the current approaches for thermoplastic–PDMS bonding are usually involved a series of chemical treatment processes (e.g., salinization). To simplify the bonding process between thermoplastic and PDMS, in this study, a low-cost, low-residue, easy-to-process bonding method was proposed with the help of silicone/acrylic differential double-sided adhesive tape. The differential tape consists of a silicone adhesive layer on one side and an acrylic adhesive layer on the other side, and during the hybrid bonding process, the silicone adhesive layer was bonded with PDMS substrate after a corona treatment process, while the acrylic adhesive layer bonded directly with the thermoplastic plate (polymethyl methacrylate and cyclic olefin copolymer) under the room temperature through a roller laminator. The whole hybrid bonding process is simple and without a chemical surface treatment process, and the bonding strength is also comparable to conventional bonding approaches. More importantly, the enclosed channel on PDMS substrate has consistent properties (e.g., water contact angle) on all four side walls, which may have significant advantages in sophisticated microfluidic applications like droplet

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generation. The bonding strength tests and biocompatibility tests were also conducted in this study.

1 Introduction

Polymer microfluidics has been widely used since the last decade as an alternative to silicon/glass microfluidics [1]. Compared with silicon/glass materials, polymer material has the advantages of low-cost, easy-to-process, disposable, and with broad range of selections of different polymers with different optical, mechanical, and chemical properties. Currently, polymer-based microfluidics has been widely used in biological [2], analytical chemistry [3], and medical fields [4].

Generally, two classes of polymer materials were used in microfluidics: thermoplastics and elastomers. Thermoplastics are easy to process when heated around/above the glass transition temperature, microstructures can be fabricated with hot embossing [5], injection molding [6], laser ablation [7], roller imprinting methods [8], etc. Various types of thermoplastics have been used in microfluidics, including PMMA (polymethyl methacrylate) [9], PS (polystyrene) [10], and COC (cyclic olefin copolymer) [11]. Besides thermoplastics, elastomers have also been widely used in microfluidics, the most commonly used elastomer is PDMS (polydimethylsiloxane) [12, 13]. PDMS-based microfluidic devices were usually processed with the casting (soft lithography) method [14], and microstructures were formed on the surface of PDMS with the help of a SU-8 mold.

In the conventional approaches to the fabrication of polymer microfluidic devices, the microstructures (e.g., microchannels) were fabricated on the surface of the substrate, then a cover plate (usually with inlet and outlet ports) was bonded with the substrate to enclose the microchannels. It is worth mentioning that the substrate and cover plate could be the same or different materials.

The bonding between PDMS and glass is usually conducted with the oxygen plasma treatment process [15]. For thermoplastics, the most commonly used method is thermal fusion bonding [16], the substrate and cover plate were sandwiched between two heated metal platens, and the compression force was applied with the temperature on metal platens raised

around the glass transition temperature of the substrate/cover plate material, the molecular entanglement achieved the bonding. However, the fabricated microstructures on the substrate can be easily damaged (collapsed) during the thermal fusion bonding process, thus, various methods e.g., UV radiation [16], oxygen plasma treatment [17], and chemical treatment [18] were usually used to lower the bonding temperature to avoid the damage of the microstructures.

However, in the application of thermoplastic-based microfluidics, thermal fusion bonding has several drawbacks: firstly, the applied bonding force under high temperature may cause the microchannel collapse; secondly, several applications scenarios (chip contains freeze-dried reagent) are highly sensitive to the exposure of high temperature; thirdly, in hybrid bonding, the glass transition temperature of the substrate and cover plate is different, which makes the thermal fusion difficult to achieve.

Currently, the demand for hybrid bonding keeps rising, especially for the bonding between thermoplastics and PDMS [19, 20]. The microstructures were cast on the surface of PDMS and bonded with a layer of the thermoplastic plate to seal the microchannel. The current approach for hybrid bonding between thermoplastics and PDMS usually depends on the complicated chemical treatment process (e.g., APTES treatment) followed by oxygen plasma treatment [21]. The residues from chemical treatment are inevitably left inside the microchannel during the bonding process. The adhesive tapes were also previously reported for the bonding between thermoplastic and PDMS [22], the major drawback of the previous approaches using double-sided tapes is the low bonding strength. In addition, for both of the hybrid bonding methods described above, only three of the four side walls of the enclosed microchannel are PDMS, while one side wall is either thermoplastic with coated chemicals or adhesive layers of double-sided tape. The non-uniformity of the side walls may cause serious issues with sophisticated fluid manipulation approaches, e.g., high-speed droplet generation.

In this study, a silicone/acrylic differential double-sided adhesive tape was used for the bonding between thermoplastics and PDMS, and the double-sided adhesive tape used in this study consisted of a silicone adhesive layer on one side and acrylic adhesive on the other side. With this unique structure of the adhesive tapes, the silicone adhesive was bonded to the PDMS substrate with corona treatment, while the acrylic layer was directly bonded to the thermoplastics cover plate. Compared with the previous study, the enclosed channel has a uniform water contact angle on all four side walls with a relatively higher bonding strength. In addition, the proposed method is a residue-free bonding process without chemical surface treatment.

2 Materials and methods

2.1 Materials and instruments

A silicon/acrylic differential double-sided adhesive (Tesa 61532, Tesa, Inc., Germany) was used in this study for the hybrid bonding between PDMS and PMMA. Tesa 61532 is a transparent double-sided tape with PET backing; silicone adhesive was coated on one side of the PET backing, while the acrylic adhesive was coated on the other side, and the total thickness of the double-sided adhesives (without liner layers) is around 50 μm (illustrated in Fig. 1). The silicone side will later be bonded to PDMS, while the acrylic side will be bonded to thermoplastics (PMMA or COC plate). Casted PMMA sheet with a thickness of 2 mm and 5 mm is sourced from Bakway, Inc., China. The custom-made COC sheet with a

thickness of 1.5 mm is fabricated by injection molding with COC pellets from TOPAS GmbH, Germany.

The *Bacillus subtilis* CMCC(B) 63501 bacteria used in the biocompatibility test are sourced from China General Microbiological Culture Collection Center (CGMCC). The bacteria nutrition tryptone soy broth (TSB) is sourced from Aoboxing Bio-Tech Co. Ltd., China.

The scanning electron microscope, GeminiSEM 300 (Carl Zeiss AG, Germany), was used for the observation of the cross-section of the bonded chips. The UV-Vis spectrums were obtained from UV-3600i Plus, Shimadzu Corporation, Japan. The hot-roller laminator for the bonding of the chips is GD-320, Golden Corporation, China. A CO₂ laser ablation instrument (HTE-1206-W80) was used for the cutting of thermoplastic sheets and differential double-sided tapes.

2.2 Fabrication and bonding process

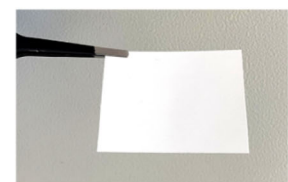
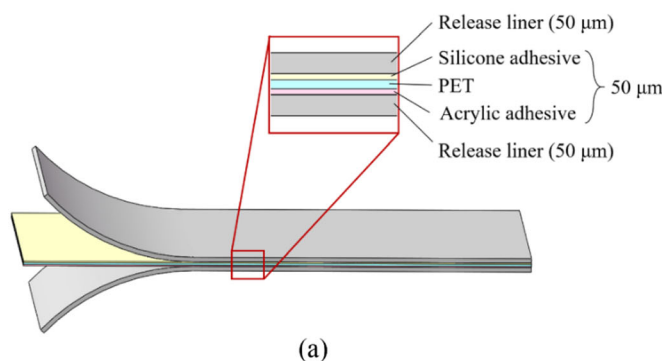
The microfluidic chip bonding process is shown in Fig. 2. The PDMS substrate with microchannels was fabricated with a standard soft lithography approach: the SU-8 photoresist was spin-coated on the surface of a 4-in. silicon wafer, after UV exposure and development, the formed SU-8 patterns were used as mold for the replica molding of PDMS. The PDMS (SLYGARD 184) base and the crosslinking agent were mixed with a ratio of 10:1 and poured on the patterned SU-8 mold, after baking at 65 °C for 4 h. The PDMS layer was peeled off from the SU-8 mold.

To seal the fabricated microchannels on the surface of PDMS substrate, thermoplastic (PMMA or COC) sheets were laser cut into the same dimension of PDMS substrate, through holes were also cut with

Fig. 1 Illustration and image of the double-sided adhesive used in this study.

a Illustration for the structure of Tesa 61532 differential double-sided adhesive.

b Image of the double-sided adhesives with protective liner layers. **c** Image of the double-sided adhesives without liner layers on both sides



(b)



(c)

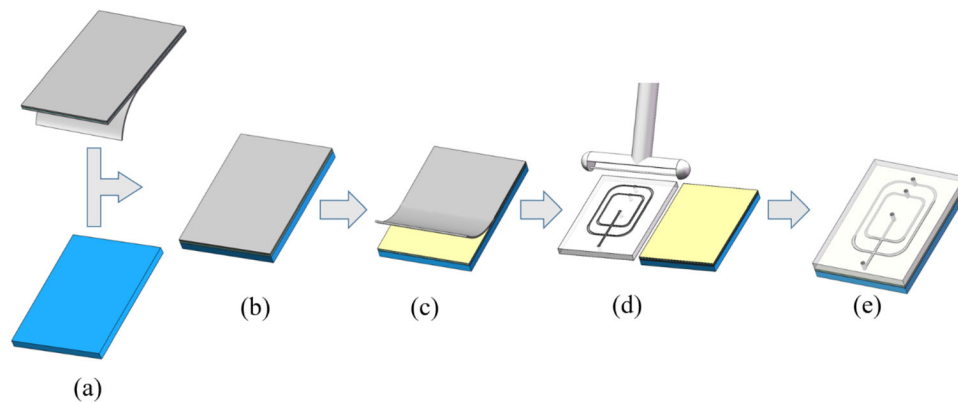


Fig. 2 Chip bonding process. **a** Removal of release liner on acrylic adhesive. **b** Acrylic adhesive was bonded with PMMA/COC cover plate with a roller laminator. **c** Removal of release liner on silicone adhesive. **d** Surface corona treatment on the surface of

silicone adhesive and the surface of PDMS substrate. **e** Bonding between silicone adhesive and PDMS substrate with a gentle hand press

CO₂ laser for the inlet and outlet ports of the fluids, and the power of the laser was set at 50 W with a scan speed of 20 mm/s in the cutting processes. It is worth mentioning that the CO₂ laser is also used to cut the 61532 adhesives into the same dimension as the substrate and cover plate. The formation of microchannels on 61532 adhesive can also be achieved with CO₂ laser ablation.

The bonding process is illustrated in Fig. 2; firstly, the release liner layer on the acrylic side of the 61532 adhesive was removed, then the acrylic adhesive was bonded with PMMA/COC cover plate with the help of an office hot-roller laminator (without turning on the heat), and the PMMA/COC cover plate and 61532 adhesives were bonded under the room temperature. After bonding of thermoplastic cover plate, the liner on the silicone adhesive side was removed and then treated with a handheld corona device (BD-20AC laboratory corona treater, Electro-Technic Products, USA) for 1 min; the surface of PDMS substrate was also treated with corona for 1 min. Finally, the PDMS substrate and silicone adhesive layer were manually aligned, attached, and then gently pressed by hand to finish the bonding process.

For the demonstration of the proposed bonding process with 61532 adhesive, two types of microfluidic devices were fabricated (as shown in Fig. 3). Both of the devices were fabricated with the standard soft lithography procedure: PDMS cast with SU-8 mold and then bonded with PMMA substrate with the help of 6152 adhesive with the procedure shown in Fig. 2 to enclose the microchannel. The inlet/outlet ports on PDMS were punched with a biopsy punch.

The diffusion mixer shown in Fig. 3a has an identical channel width of 400 μm and depth of 200 μm for all the microchannels, and the flowing rates on both phases (DI water with blue food dye) were adjusted from 5 μL/min to 200 μL/min, and no leakage or burst of the bonding was observed in the experimental process with the fabricated diffusion mixer. In addition, no clogging or swelling was observed in the experimental process.

For the droplet generator (flow-focusing droplet generator) shown in Fig. 3b, all the fabricated microchannels have the same width and depth of 50 μm; the continuous phase is paraffin oil with 2 wt% Span 80, and the dispersed phase is DI water with blue food dye. The continuous phase was controlled with a syringe pump with the flowing rate ranging from 1 to 20 μL/min, and the dispersed phase was propagating with flow rates of ranges from 0.5 to 5 μL/min, and the droplet formation from squeezing mode to dripping mode was observed in the experimental process. The droplet formation is stable without the formation of the droplet tail [23] or satellite droplet [24], indicating a good consistency of the channel walls.

SEM image for the cross-section of the bonded microfluidic devices is shown in Fig. 4. PDMS substrate (with fabricated microchannel) was bonded with PMMA cover plate with the 61532 double-sided adhesive sandwiched between PDMS and PMMA.

A conventional hybrid bonding approach (silanization) between PDMS and COC was also conducted for comparison with the proposed method. The COC cover plate was treated with oxygen plasma

Fig. 3 Fabricated microfluidic chips for the demonstration of the proposed bonding method. **a** Diffusion mixer. **b** Droplet generator

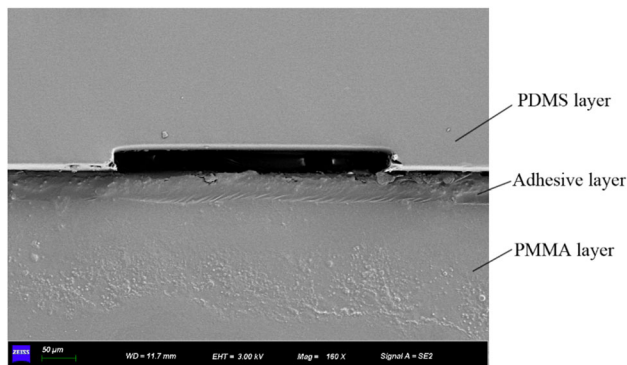
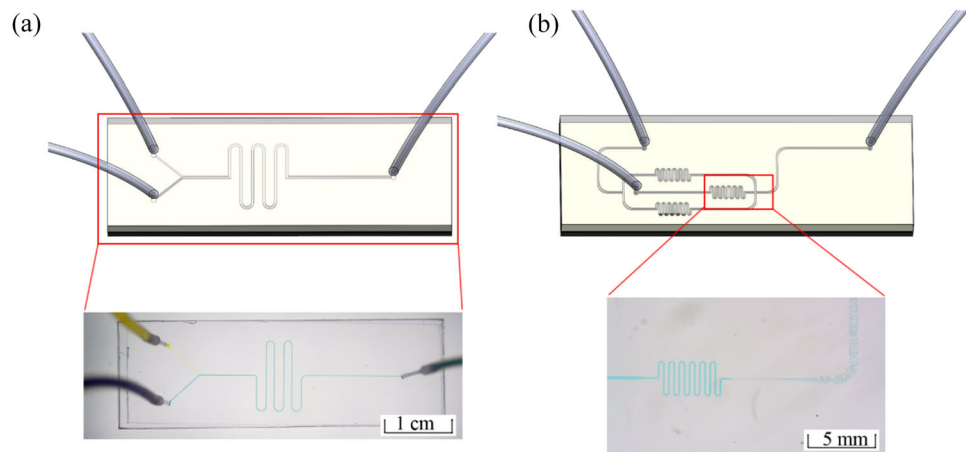


Fig. 4 Cross-section of PDMS–PMMA bonded chips, 61532 adhesive was sandwiched between PDMS and PMMA layer

(225 W for 18 s), and then immersed in APTES (aminopropyltriethoxysilane) solution (1% w/w) for 20 min. The COC plate was then cleaned with DI water and dried with nitrogen, after that, both PDMS and COC plates were treated with oxygen plasma again (225 W for 18 s) and the treated surfaces were aligned and attached under room temperature with a gentle hand press to finish the hybrid bonding process.

3 Results and discussion

3.1 Bonding strength test

The bonding strength between PDMS and PMMA/COC was measured with two approaches: the tensile bonding strength method and the burst opening method. The tensile bonding strength measurements were conducted with a universal tensile testing machine, four groups of tests were conducted, and all

bonded specimens have a 10 mm by 10 mm bonded area.

In the first group, PDMS and 2-mm-thick PMMA were bonded with sandwiched 61532 adhesive (a detailed bonding process has been introduced in the previous session), and the measured tensile bonding strength (average of 8 measurements) is 0.51 MPa. In the second group, PDMS and 5-mm-thick PMMA were also bonded with 61532 adhesive, and the measured tensile bonding strength (average of 8 measurements) is 0.52 MPa. In the third group, PDMS and 1.5-mm-thick COC plates were bonded with 61532 adhesive, and the measured tensile bonding strength (average of 8 measurements) is 0.44 MPa. In the fourth group, PDMS and COC were bonded with the conventional hybrid bonding approach with the help of APTES (fabrication detailed introduced in the previous session), and the measured bonding strength is 0.46 MPa. The load–elongation curves of PDMS–PMMA and PDMS–COC in the tensile bonding test are shown in Fig. 5a, b. The system setup for the tensile bonding strength test is shown in Fig. 5c. The tensile bonding strength test indicates the proposed method is comparable with the conventional silanization-based hybrid bonding method.

The system setup for the burst opening bonding strength test is shown in Fig. 6. The compressed air from a compressor was pumped into the bonded chip through a pressure regulator and a pressure sensor (SDE1, Festo Corporation, Germany) for the accurate measurement of the air pressure. As shown in Fig. 6, the air pressure was adjusted (rise slowly) manually with the pressure regulator until the chip was broken. The bonding specimen used in this study has a

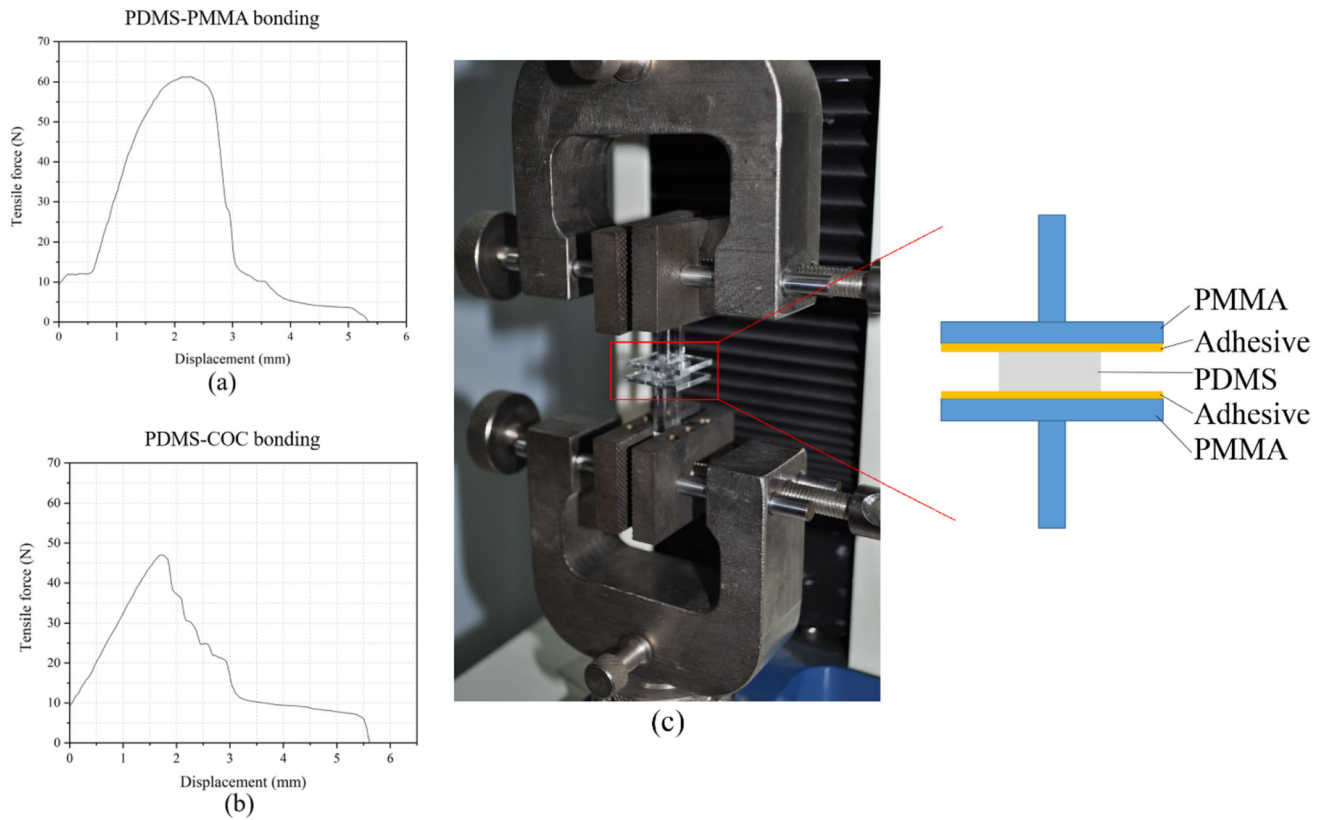
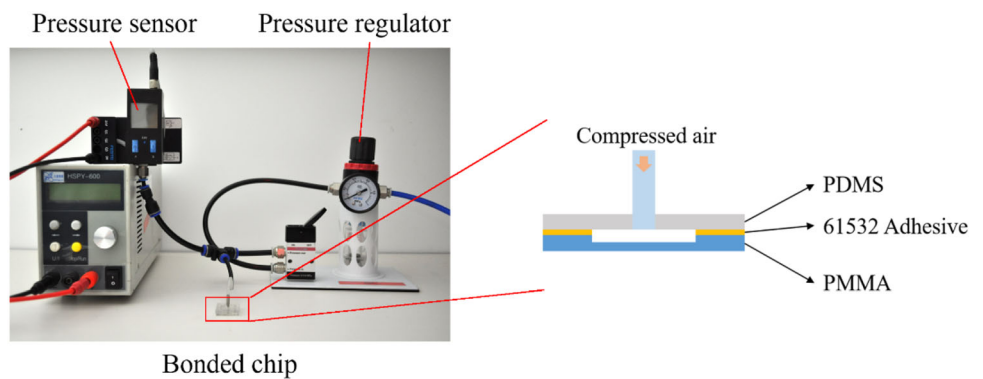


Fig. 5 Tensile bonding test for the PDMS–PMMA and PDMS–COC bonding

Fig. 6 System setup for the burst opening bonding strength test



bonded area of 284 mm², and the air chamber at the center of the chip has a cross-section area of 116 mm². The maximum burst pressure for PDMS–PMMA bonding with 61532 adhesive is 293 kPa (average of 4 measurements), while the PDMS–COC bonding with 61532 adhesive has a maximum burst pressure of 158 kPa (average of 4 measurements).

3.2 Surface contact angle and UV transmission

Compared with the previous hybrid bonding methods, with the help of 61532 adhesive, the enclosed channel has a consistent water contact angle on all four side walls of the microchannel. The measured water contact angle on silicone/acrylic adhesive layer and PMMA/PDMS substrates is shown in Fig. 7. The measured water contact angle on PDMS substrate is 110.87°, while the measured water contact angle on silicone adhesive is 109.7°, thus, the sealed

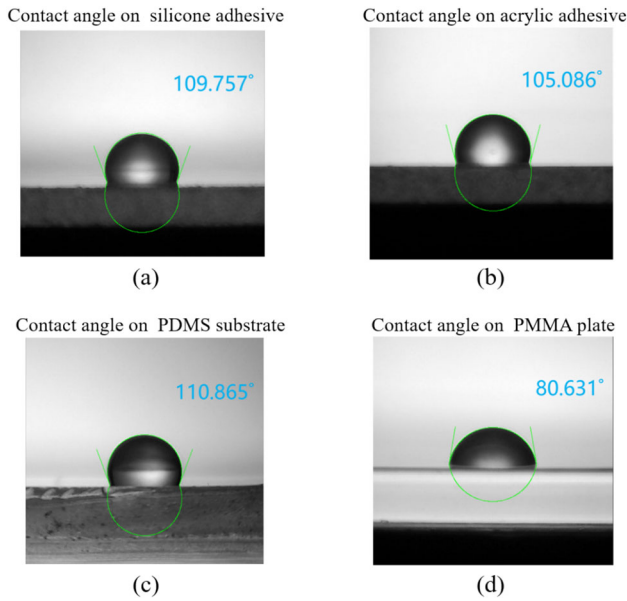


Fig. 7 Water contact angles on silicone and acrylic side of 61532 adhesive compared with the water contact angle on PDMS substrate and PMMA plate. **a** Contact angle on silicone side of 61532 adhesive. **b** Contact angle on acrylic side of 61532 adhesive. **c** Contact angle on PDMS substrate. **d** Contact angle on PMMA plate

microchannels on PDMS substrate (PDMS bonded with silicone side of the 61532 adhesive) are able to maintain the consistent water contact angle on all four side walls. On the other hand, the water contact angle on PMMA cover plate and acrylic side of 61532 adhesive shows slight difference, the measured water contact angle on PMMA plate is 80.63°, while the measured water contact angle on acrylic adhesive is 105.1°.

In order to evaluate the influence of the optical transparency from 61532 adhesive sandwiched

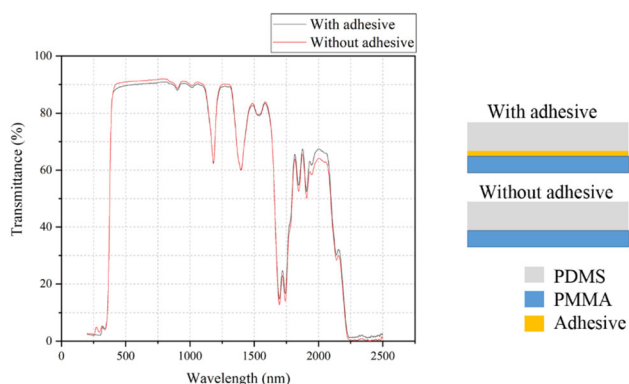


Fig. 8 UV-Vis spectrum of the PDMS-PMMA bonded chips with and without 61532 adhesive

between the PDMS and PMMA plates, the UV-Vis spectrums were measured with stacked (unbond) PDMS/PMMA plates and bonded PDMS/PMMA plates (with 61532 adhesive in between the plates). As shown in Fig. 8, the 61532 adhesive has no significant influence on the optical transparency.

3.3 Biocompatibility test

The biocompatibility of the 61532 adhesives was evaluated by culturing *B. subtilis* CMCC(B) 63501 bacteria in direct contact with 61532 adhesive. As shown in Fig. 9, the PDMS and PMMA plates were bonded with 61532 adhesive to form a culture chamber, and the bottom of the chamber is covered with the silicone side of the 61532 adhesive. The *B. subtilis* bacteria were cultured in TSA medium at 37 °C for 10 h. The viability of the bacteria after 10 h was verified with the Gram staining method (as shown in Fig. 9c). It is worth mentioning that the conducted biocompatibility test solely depends on the *B. subtilis* bacteria used in this study, and further biocompatibility or chemical inertness test is required with different applications.

4 Conclusion

This study proposed a new hybrid bonding method between thermoplastics and PDMS, a double-sided adhesive tape was used to achieve the bonding process. Compared with the conventional silanization-based hybrid bonding method, the proposed method has the advantages of a simple bonding procedure,

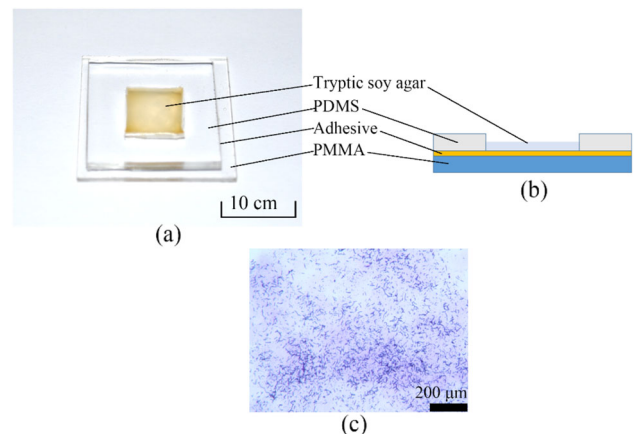


Fig. 9 Biocompatibility test (bacteria culture) on the surface of the 61532 adhesive

low residue inside the microchannel, suitable for various thermoplastics, and comparable bonding strength with the conventional method. In addition, the proposed bonding method can maintain the consistency of the water contact angle on microchannels fabricated on PDMS, which is highly important for applications like droplet generation.

Author contributions

YL performed the fabrication and testing of microfluidic chips. XW collected the data and performed the analysis. YW conceived and designed the experimental process. YF supervised this study and drafted the manuscript. All authors read and approved the final manuscript.

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Data availability

The datasets generated during and/or analyzed during the current study are available from the corresponding author upon reasonable request.

Declarations

Conflict of interest The authors have no conflicts of interest to declare that are relevant to the content of this article.

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