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Solvent-assisted low-temperature and low-pressure poly(methylmethacrylate) bonding coupled with selective microchannel hydrophobic coating for reliable sealing

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ABSTRACT

We introduce a facile and robust strategy for bonding a poly(methylmethacrylate) (PMMA) thermoplastic microdevice via ethanol treatment followed by thermal bonding at relatively low temperature and low pressure. Organic solvents can speed up bonding without sacrificing the optical properties of the PMMA substrate, but microchannels are often clogged by the solvent during the bonding process. To prevent channel clogging while achieving robust sealing, a microchannel was selectively treated hydrophobically, and then the entire surface of the PMMA was treated with ethanol at 80 °C for 30 min. Two pieces of PMMA were thermally pressed together at 60 °C for 20 min to obtain a permanent seal. Tensile strength measurement, high-throughput leakage test, and burst test were performed. The highest bonding strength was approximately 12.4 MPa, and the bonding was robust enough to endure intense liquid flow that was almost 450 times higher than the total internal volume of the microchannel.

1. Introduction

For the last 20 years, microfluidic devices have been increasingly used as a platform for chemical and biological experiments and biomedical diagnosis [1,2]. Microfluidic devices have a very wide range of applications [3], but proper combinations of structure design, material selection, fabrication, and substrate bonding are important to meet the particular requirements. The most commonly used material is silicon, but it is expensive and opaque, which limits its applications where optical detection is necessary. Glass, polymers, and paper have also been used [4–6]. Thermoplastic materials have great commercial potential for mass production, which is a critical factor for commercialization [7,8]. Thermoplastics are affordable, easy to fabricate, flexible, optically transparent, chemically inert, and biocompatible [9,10]. Their efficiency has been proven in growing numbers of studies [7]. Thermoplastics have been applied in various experiments, such as cell culture [11,12], DNA analysis [13,14], and pathogen detection [15].

Major types of thermoplastics include polyethylene (PE), polystyrene (PS), polymethyl methacrylate (PMMA), poly vinyl chloride (PVC), polyethylene terephthalate (PET), polycarbonate (PC), and polypropylene (PP). All of these types can be melted down many times and reshaped into different forms. Although they share the basic properties of thermoplastics, each one has subtly different characteristics of rigidity, transparency, and resistivity to solvents [16]. For example, PMMA has excellent light permeability, making it suitable for experiments that include optical observations [17]. The surface polarity of thermoplastics differs with the specific molecular groups. For example, oxygen-containing functional groups in ester-based thermoplastics make them polar [16].

Because of these subtle differences in thermoplastics, there are many options for fabricating and bonding to produce microfluidic devices. Two homogeneous plastic materials can be bonded using adhesive bonding [18,19], thermal fusion bonding [20,21], solvent bonding [22], and localized welding [23]. Among these methods, thermal bonding and solvent-assisted bonding are the most frequently used methods due to process simplicity [24]. Thermal bonding involves heating the substrate to near its glass transition temperature and making it soft or melting it to make a bond. However, thermal bonding requires high energy because the process includes heating to near the glass transition temperature (over 100 °C in many cases) and is accompanied by pressing, which can result in channel deformation.

Solvent bonding is a sealing process that uses solvents that temporarily soften and dissolve the surfaces of thermoplastics and make them bond after applying proper pressure. The solvent can be used alone to bond two different or homogeneous substrates,

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or it can assist in moderate thermal bonding. Various solvents can be selected based on their Hildebrand solubility parameter, which is a good indicator of interactions between solvents and nonpolar polymers [25]. One major challenge of solvent-assisted thermal bonding, however, is optimizing the applied temperature and pressure [7,26]. High temperature and pressure result in channel deformation or collapse. Similarly, solvents that are too strong would induce channel clogging if they remain in the channel after treatment. Therefore, optimizing process factors such as solubility, temperature, pressure, and time is critical to achieve high bond strength while minimizing the deformation of embedded microchannels.

Tuning the surface properties of the inner wall of a fabricated channel is another consideration in conserving the geometry of microchannels. One method is applying specific chemicals to alter the polarity of the substrate surface. Selective hydrophobic treatment of a microchannel prior to applying a solvent can help prevent clogging problems because it limits contact with the hydrophilic solvent. Our previous study examined the effectiveness of surface modification for various plastic surfaces with poly[dimethylsiloxane-co-(3-aminopropyl)methylsiloxane] (amine-PDMS linker) [27]. The amine-PDMS linker significantly increased the hydrophobicity of the surface of various thermoplastics such as PC, PET, PVC, and PI with excellent chemical and thermal stability. This method works by endowing PDMS functionality on the surface of the coated plastic surfaces [27]. The amine-PDMS linker is composed of a PDMS backbone that includes an amine side group, which can also react with the carbonyl group of PMMA [28,29] and makes the treated surface hydrophobic.

In this study, we develop a simple and reliable method of solvent-assisted thermal bonding to construct a PMMA microfluidic device. In specific, several experimental procedures were tested to find the optimum temperature and time for substrate ethanol treatment as well as the pressing. We also perform selective hydrophobic treatment of a PMMA microchannel using an amine-PDMS linker. The robustness and reliability of the introduced bonding method are verified by performing tensile strength measurement, leakage test, and burst test. Contact angle measurement and an ink injection test are also performed.

2. Materials and methods

2.1. Materials

Ethyl alcohol (95.0%) was purchased from Daejung Chemicals & Metals. Poly(dimethylsiloxane) (PDMS) prepolymer (Sylgard 184) and a curing agent were purchased from Dow Corning. Poly[dimethylsiloxane-co-(3-aminopropyl)methylsiloxane] (amine-PDMS linker) was purchased from Sigma-Aldrich.

2.2. Ethanol treatment and bonding conditions

The optimum temperatures for ethanol treatment and thermal pressing were examined. PMMA was treated with ethanol at room temperature, 60 °C, and 80 °C, and treatment times of 5, 15, and 30 min were considered. For sealing, two pieces of PMMA were pressed at either room temperature for 20 min or at 60 °C for 20 min. The PMMA substrates were cut into 20 × 30 mm and 60 × 80 mm pieces, and the corresponding pressures applied for thermal pressing were 0.3 MPa (3.0591 kg/cm^2) and 0.4 MPa (4.0788 kg/cm^2), respectively.

2.3. Bonding procedure

Fig. 1 shows the overall scheme for bonding two PMMA substrates via ethanol treatment followed by thermal pressing. One PMMA substrate was engraved with a microchannel and another was left flat. These substrates were cleaned with distilled water and dried completely prior to bonding. The microchannel-engraved PMMA was also plasma treated (Fig. 1a) and placed in conformal contact with a slab of PDMS (Fig. 1b). The PMMA and PDMS were sealed reversibly, and poly[dimethylsiloxane-co-(3-aminopropyl)methylsiloxane] (the amine-PDMS linker) was introduced into the inlet and guided along the microchannel for selective hydrophobic treatment of the microchannel (Fig. 1c). After 20 min of reaction, the microchannel-engraved PMMA and PDMS were detached, and the two pieces of PMMA were completely immersed in ethanol that was preheated at a predetermined temperature for 30 min (Fig. 1d). Immediately after drying the substrates, the assembly was thermally pressed at a predetermined temperature for 20 min for permanent sealing (Fig. 1e).

2.4. Bond strength analyses

2.4.1. Tensile strength measurement

Tensile strengths of the PMMA assemblies were measured using a texture analyzer (QTS 25, Brookfield, Middleboro, MA, USA) [30]. PMMA substrates with the dimension of 85 × 10 × 2 mm were prepared for the measurement of bond strength. For the insertion of twines, through-holes were punctured on each PMMA sheet of the assembly using a drilling machine. The partially overlapped PMMA substrates were fixed in the strength measurement analyzer, and were pulled apart at a speed of 150 mm min^{-1}. The overlapped length of the two PMMAs was 1 mm. The experiments were repeated four times to examine bonding reproducibility.

2.4.2. Leakage test

To perform leakage test, a serpentine microchannel was fabricated on a 50 × 50 × 10 mm PMMA substrate using a CNC milling machine. Channel width, depth, and total length of the microchannel were fabricated to be 1 mm, 1 mm, and 30 cm, respectively. Inlet and outlet ports were punctured using a drilling machine, and silicone tubes (o.d. 2.0 mm, i.d. 1.0 mm) were inserted. A syringe pump (Legato 200, KD Scientific, New Hope, PA, USA) was connected to the silicone tube to introduce colored ink solution. The experiments were repeated three times.

2.4.3. Burst test

To perform burst test, compressed air was introduced into the microchannel of the sealed PMMA microdevice with the same dimension used for the leakage test. The pressure at which either the sealing was destroyed or silicone tube was detached from the microdevice was measured. The experiments were repeated three times.

2.5. Contact angle measurement

The water contact angles were measured on the surfaces of flat PMMA substrate by the sessile drop technique using a Phoenix 300 contact angle measuring system (Surface Electro Optics, Korea) before and after coating PMMA with the amine-PDMS linker to observe wettability change after the hydrophobic treatment. The measurements were repeated three times and averaged.

3. Results and discussion

3.1. PMMA bonding performance

Fig. 2 shows the results of bonding using various temperatures and times for ethanol treatment and subsequent thermal pressing. All of the PMMA assemblies either did not bond or only partially bonded. Newton's rings occurred under all conditions tested except