Short communication

Surface modification-assisted bonding of polymer-based microfluidic devices

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ABSTRACT

This paper reports on the development and application of a surface modification technique as an improved method for bonding polymer microfluidic substrates. This technique readily produced complete microfluidic chips via plasma oxidation followed by silane reagent treatment on the polymer surface. Characterization of the bonded chips was performed using scanning electron microscopy (SEM), water contact angle measurement, and tensile strength measurement. SEM images showed that the integrity of the channel features was successfully preserved. A bond strength approaching that of solvent welding was demonstrated. This technique has been successfully applied to bond dissimilar polymer substrates (polymethylmethacrylate (PMMA), amorphous polyethylene terephthalate (APET), polycarbonate (PC)), and is also applicable to bonding a hard polymer substrate to polydimethylsiloxane (PDMS) or glass.

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1. Introduction

The field of microfluidics has rapidly expanded since its introduction in the early 1990s [1]. Microfluidic devices are becoming increasingly attractive as alternatives to their larger-scale conventional counterparts. Microfluidic solutions can offer smaller sample and reagent volumes, high separation efficiencies due to their high surface area-to-volume ratio, rapid analysis time, improved sensitivity, and the possibility of integrating multiple chemical processes all in one chip, which allows for the development of micro total analysis systems (Micro-TAS).

Glass was the first substrate used for fabrication of microfluidic devices due to its well established fabrication process and many benefits, such as its chemical resistance, high voltage tolerance, optical transparency, high thermal stability and biocompatibility. It has been employed successfully as the material of choice for many applications [2,3]. Despite their advantages, glass chips also have some limitations, namely their high unit cost and specialized fabrication procedure.

Recently, polymers [4–7] have become viable materials for microchips because they are easy and cost-effective to fabricate, yet retain many of the desirable characteristics of glass. They are disposable, biocompatible, and possess excellent optical properties, such as low intrinsic autofluorescence and high transparency. Fabrication of polymeric devices has been performed using several mass replication technologies such as hot embossing, injection molding, laser ablation, soft lithography, and casting [8–12]. A wide variety of polymer materials are available; each has unique physical and chemical characteristics, which make them suitable for different microfluidic applications. Of the available polymer materials, thermoplastic polymers (such as polymethylmethacrylate (PMMA) [13–16], polyethylene terephthalate (PETE) [17,18], polycarbonate [19]), and polydimethylsiloxane (PDMS) [11,20], have been intensively studied. PDMS has been extensively used for making microchips due to its ease of fabrication. PMMA and PC are the most widely used materials for chip production due to their low cost, good mechanical strength, optical transparency, and biocompability. PETE is also an attractive material that is gaining popularity due to its physiochemical properties, mainly its resistance to aggressive solvents (acetonitrile, chloroform, tetrahydrofuran, etc.) [18].

Various bonding techniques have been reported for fabrication of polymer substrates, such as thermal bonding [21], solvent bonding [22], plasma oxidation [17,18], the use of adhesives [2], UV/ozone surface treatment [16], and laser welding [23]. A successful bonding process must preserve channel integrity, geometry, and structure, and offer high bonding strength. Thermal bonding is the most popular method for sealing plastic chips. However, the bond strength ($1000\,kN/m^2$) created by this method is usually much lower than that of solvent-bonded chips [15]. Special care must be taken in thermal bonding and solvent welding since the substrates are subjected to high temperatures and strong solvents that lead to channel deformation. Sacrificial layers, such as paraffin wax, low melting temperature alloys, and ice [13,24], have been utilized to protect the channels from deforming; however, it can be difficult

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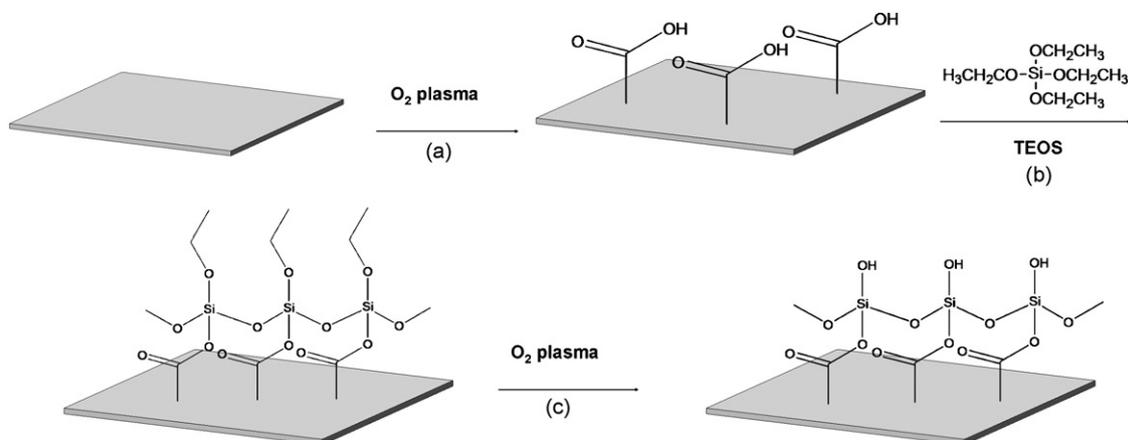 *E-mail address:* Vincent.Remcho@oregonstate.edu (V.T. Remcho).

Fig. 1. Schematic of the PMMA surface modification using oxygen plasma and TEOS.

to completely remove the sacrificial materials from the channels. Due to these challenges with the bonding process, a need remains for effective bonding methods for microchip fabrication.

Low bond strengths between two polymer substrates are largely due to the low specific energy of the polymer surface [16], typical of hydrophobic or weakly hydrophilic polymers. Several methods have been developed to increase the surface energy, including surface grafting and atmospheric plasma activation [25,26]. Plasma oxidation has commonly been used in bonding elastomeric materials, such as PDMS. The energy comes from ions, electrons, and UV photons in the plasma, which break chemical bonds on the polymer surface to produce highly reactive free radicals and generate polar functional groups on the surface. Plasma treatment creates the desired charged groups and increases the overall surface energy, which in turn also increases hydrophilicity and wettability [15]. Through the generation of polar functional groups on the polymer surface, hydrogen or covalent bonds may be formed across the interface; strengthening the bonds between the two contacting surfaces.

Low temperature bonding of PMMA and cyclic olefin copolymer (COC) has been reported using O_2 plasma activation or UV/ozone surface treatment [16], however, the reported bond strength values limited these devices to low pressure applications. In a recent publication by Vlachopoulou et al. [14], a method for bonding PMMA to PDMS via a surface modification technique using 3-aminopropyltriethoxysilane (APTES) to create covalent bonds was presented. The drawback of this approach is the necessity of using a thin PDMS layer as an intermediate to bond two pieces of polymer substrates, such as PMMA to PMMA. Thus, channels do not have homogeneous wall materials, which limits the range of application of devices prepared in this manner.

In this work, an improved bonding method via surface modification of plastic and glass materials is presented. The bonding was performed through surface modification by plasma oxidation followed by application of silane reagents (tetraethyl orthosilicate or TEOS) to incorporate Si functionalities on the polymer surface, facilitating siloxane bonds between the two polymers (Fig. 1). This bonding process is simple, robust, and cost-effective, making it applicable to a wide range of polymer substrates (PMMA, PC, APET, PDMS and glass) and native Si-containing materials, such as glass or PDMS.

2. Experimental

2.1. Chemicals/materials

Tetraethyl orthosilicate (TEOS ≥99.0%), isopropyl alcohol (99.5%), and Corning cover glass no. 1 (24 mm × 60 mm) were obtained from Sigma–Aldrich (Milwaukee, WI, USA) and were used as received. Rhodamine B was obtained from Lambda Physik (Acton, MA, USA). SU-8 3050 and SU-8 developer were obtained from Microchem (Newton, MA, USA). Polyetherimide (PEI), polycarbonate (PC), polymethylmethacrylate (PMMA) were obtained from McMaster-Carr (Santa Fe Springs, CA, USA). Amorphous polyethylene terephthalate (APET) was obtained from ALRO Plastics (Jackson, MI, USA). Polydimethylsiloxane (PDMS) Sylgard 184 was obtained from Dow Corning (Midland, MI, USA), consisting of a base polymer and curing agent. PDMS was mixed in a 10:1 ratio of monomer to the curing agent, poured onto a clean unpatterned glass slide, and thermally cured for 3 h at 60 °C.

2.2. Microfabrication

The SU-8 master was fabricated on a silicon wafer via a standard photolithography technique using a maskless exposure system (SF-100, Intelligent Micro Patterning, LLC, St. Petersburg, FL, USA). SU-8 3050 was spun on the wafer at a thickness of ~100 μm according to a recently published program [22].
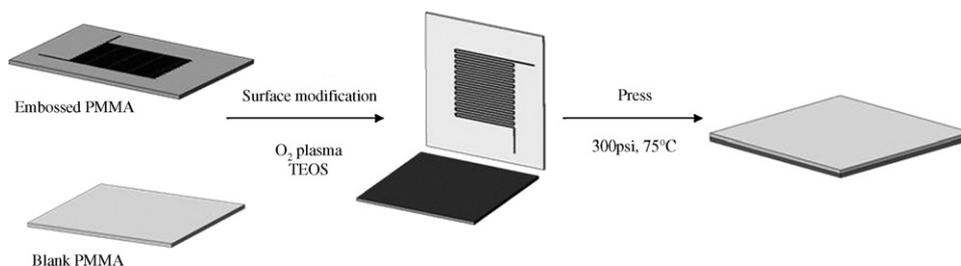

Fig. 2. Schematic of the PMMA surface modification-assisted bonding procedure. Both the embossed and blank PMMA pieces were surface modified with oxygen plasma and TEOS treatment. Following exposure to oxygen plasma, the two pieces were brought into contact and pressed together at 300 psi, 75 °C until bonding occurred.

Table 1
Bonding procedures for a variety of materials using a novel surface modification method.

Bonding materials	Pressure applied (psi)	Temperature (°C)	Time (min)
Bonding of PMMA based materials to PMMA or glass	300	75	20
Bonding of APET based materials to APET or glass	150	60	20
Bonding of PC based materials to PC or glass	300	110	20
Bonding of PMMA/APET/PC based materials to PDMS	Clamp	50 (oven)	60

The process used for two-stage embossing was published previously [8]. In the first step of the embossing process, the positive features of the SU-8 master were hot embossed into a PEI substrate ($T_g \sim 210\,°C$) using a hot press (Fred S. Carver Inc., Summit, NJ, USA). The resulting imprint produced negative features in the PEI substrate. In the second imprinting step, the PEI was used as a secondary master to emboss features into PMMA ($T_g \sim 106\,°C$), APET ($T_g \sim 75\,°C$), and PC ($T_g \sim 150\,°C$) substrates. The two-stage embossing process results in a final product with features that faithfully reproduce the primary master.

2.3. Surface modification-assisted bonding procedure

2.3.1. Bonding of two polymer substrates (PMMA, APET, PC)

A schematic of the PMMA surface modification-assisted bonding procedure is shown in Fig. 2. Prior to bonding, the surfaces of the polymers were treated using a home built oxygen plasma system. The RF source was obtained from XEI Scientific (Redwood City, CA, USA). The substrates were placed inside the plasma chamber, and the plasma was operated at 20 W and 100 mTorr for 90 s. Immediately following the plasma treatment, both surfaces were silanized using 10% TEOS solution in 60:40 (v/v) isopropyl alcohol:water at 50 °C for 30 min. Next, the functionalized surfaces were activated a second time with the oxygen plasma under the same conditions as described above. After plasma exposure, the two surfaces were brought into contact and pressed together. Bonding temperatures and pressures of different type of polymers were optimized and determined experimentally (Table 1). A post-annealing bake was performed for 2 h at 60 °C to increase bond strength.

2.3.2. Bonding of a polymer substrate to an Si-containing substrate (PDMS or glass)

This method was used to bond polymer substrates and Si-containing substrates to produce hybrid chips, i.e. PMMA–glass or PMMA–PDMS. For substrates that contain Si at the surface, the TEOS treatment can be omitted. The procedure described in Section 2.3.1 was employed on the polymer substrates. Prior to bonding, both the polymer substrate and the Si-containing substrate (such as PDMS) were exposed to an oxygen plasma and brought into contact, followed by the bonding protocol described in Table 1.

2.4. Characterization

Water contact angle measurements were taken before and after surface modification treatment using an FTÅ 135 Contact Angle and Video Analysis System (First Ten Angstroms, Portsmouth, VA, USA). 5.0 μL of water was pipetted onto the polymer surface, contact angles were measured in triplicate and the average contact angle for each substrate was reported.

Bond strength between the two contacting surfaces was determined by measuring tensile strength using an Instron 5500R tensile tester (Instron, Norwood, MA, USA). Two pieces of PMMA (25.4 mm × 25.4 mm × 1.6 mm) were bonded using the procedure described in Section 2.3.1. The bonded samples were clamped into the grips of the tensile tester, and pulled away from one another at a rate of 0.06 mm/s. The force at which the bonded PMMA failed was recorded and divided by the cross-sectional area of the bonded surface to obtain the tensile strength of bonding. The reported values are the average of at least three individual measurements tested for each method of bonding (PMMA samples bonded with and without the surface modification procedure).

The morphology of the bonded microchips was studied using an FEI Quanta 600F scanning electron microscope (SEM) (FEI Company, Hillsboro, Oregon, USA) operated at 5 kV. The microchips were frozen in liquid nitrogen and fractured to obtain the cross-section images.

3. Results and discussion

The use of this surface modification technique for bonding of microfluidic devices provides an inexpensive, straightforward, accessible method to produce polymer or hybrid microchips. The procedure is attractive as it provides a general method for the bonding of polymer substrates to adjacent polymer layers, but also to Si-containing substrates such as glass or PDMS. The method also obviates the need for high bulk temperatures (thermal bonding) and strong solvents (solvent bonding). Permanent bonding of polymers can be achieved at temperatures below T_g. As shown in Table 1, this technique is applicable to the bonding of various types of substrates by careful selection of an appropriate bonding temperature and pressure. These parameters can readily be altered and optimized depending on the type(s) of polymer(s) used and their properties (such as T_g). Therefore, the bonding method is not limited to the substrates chosen for this study.

In this work, the surface of PMMA was modified by oxygen plasma exposure, followed by TEOS treatment. The exact chemistry occurring at the surface of the polymer during oxygen plasma treatment is complex and still poorly understood [15,25]; however, studies have shown that as the plasma bombards the surface with high-energy oxygen gas molecules, active species that strongly interact with the polymer surface are generated, resulting in polar functional groups. These may include functional groups containing oxygen, such as carboxyl groups, oxides, acid anhydrides and aldehydes, which contribute to the polarity of the surface [15].
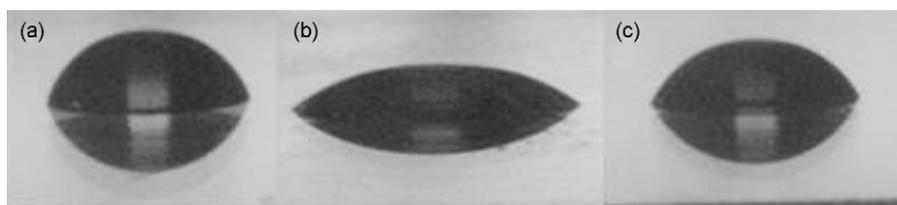
Fig. 3. Water contact angle measurements on (a) native PMMA ($75 \pm 3°$), (b) O_2 plasma-treated PMMA ($36 \pm 3°$), and (c) O_2 plasma and TEOS-treated PMMA ($65 \pm 2°$).

Table 2
Water contact angle measurements for a variety of polymers following oxygen plasma and TEOS treatments. Each reported value is the mean of at least three samples for each substrate, and three measurements were performed at different locations on any given substrate. The ± values correspond to standard deviation of the mean.

Materials	Native	O_2 plasma	TEOS
PMMA	$75 \pm 3°$	$36 \pm 3°$	$65 \pm 2°$
APET	$71 \pm 4°$	$33 \pm 3°$	$61 \pm 3°$
PC	$82 \pm 2°$	$41 \pm 3°$	$71 \pm 3°$

According to the reaction scheme shown in Fig. 1a, exposure to the oxygen plasma is postulated to result in conversion of a fraction of the native PMMA backbone into carboxylic acid groups, making the surface more hydrophilic. The acidic groups on the surface may then facilitate bonding of TEOS, as shown in Fig. 1b. The carboxylic acid groups on the PMMA surface may then react with the ethoxy groups on TEOS to introduce Si-containing functionalities on the PMMA surface. Subsequent exposure to the oxygen plasma converts the reactive ethoxy groups at the polymer surface into silanol groups (Fig. 1c). When two modified pieces of PMMA are brought into contact, siloxane (Si–O–Si) bonds are formed across the interface. Covalent bonding between a surface-modified polymer and a Si-containing substrate (i.e. PDMS or glass) can also be achieved using this surface modification process.

The extent of surface modification was assessed via water contact angle measurement. The average contact angle measurement for an unmodified PMMA surface was $75 \pm 3°$ (Fig. 3). This is in agreement with a previously published value of $73 \pm 3°$ for a native PMMA substrate [27]. Following oxygen plasma exposure and TEOS treatment, the surface contact angle was re-measured. A decrease in contact angle after plasma activation was observed: the initial contact angle for the oxygen plasma-treated substrate was $20 \pm 2°$, and increased to $36 \pm 3°$ after drying overnight at room temperature. The angle increased further to $65 \pm 2°$ after TEOS treatment. Next, plasma oxidation was performed on the TEOS-modified surface. The contact angle was measured and decreased to $34 \pm 2°$, which indicates an increase in polarity or hydrophilicity of PMMA

surface after plasma activation, thus confirming our postulation that the polymer surface was converted into silanol groups as a result of the surface modification process. The contact angle results clearly indicate that there is a substantial change in the surface chemistry of the PMMA surface.

The stability of the PMMA surface after plasma oxidation and surface modification was evaluated by re-measuring its contact angle after washing the treated surface with water and isopropanol, followed by oven drying at 60 °C for 1 h. No measurable changes were observed in the results. Furthermore, TEOS-treated polymer samples were stored at room temperature for 7 days and contact angles were re-measured. The results did not show any measurable change for any of the samples, indicating a stable surface modification procedure.

The contact angles measured for untreated amorphous PET (APET) and polycarbonate (PC) were $71 \pm 4°$ and $82 \pm 2°$, respectively, which correlate to reported values of $75 \pm 4°$ and $80 \pm 3°$ [18,27]. After oxygen plasma activation and drying, the contact angle decreased to $33 \pm 3°$ for APET and $41 \pm 3°$ for PC. The increase in hydrophilicity of the plasma-treated samples implies that the plasma had sufficient energy to break the hydrophobic polymer backbone and introduce polar groups (presumably carboxylic groups) onto the surface that were used in the ensuing surface modification step with TEOS. Table 2 gives a summary of contact angle measurements for PMMA, APET, and PC substrates following oxygen plasma and TEOS treatment.

The viability of the bonding technique was tested for the fabrication of a microfluidic chip having a serpentine channel feature with dimensions of 400 μm width, 100 μm depth, and 1 m length (Fig. 4). This dimension was chosen for proof-of-concept studies. The bonding approach was repeated to produce channels with features as low as 100 μm width, 20 μm depth, and 3 cm length and as large as 3 mm width, 100 μm depth, and 2 cm length to demonstrate the applicability of this technique to bond channel of varying range of dimensions. A photograph of the fabricated PMMA microchip is shown in Fig. 4c. Ten chips were fabricated using the surface-modification bonding technique and tested for leakage to demonstrate device integrity. A solution of rhodamine B dye was
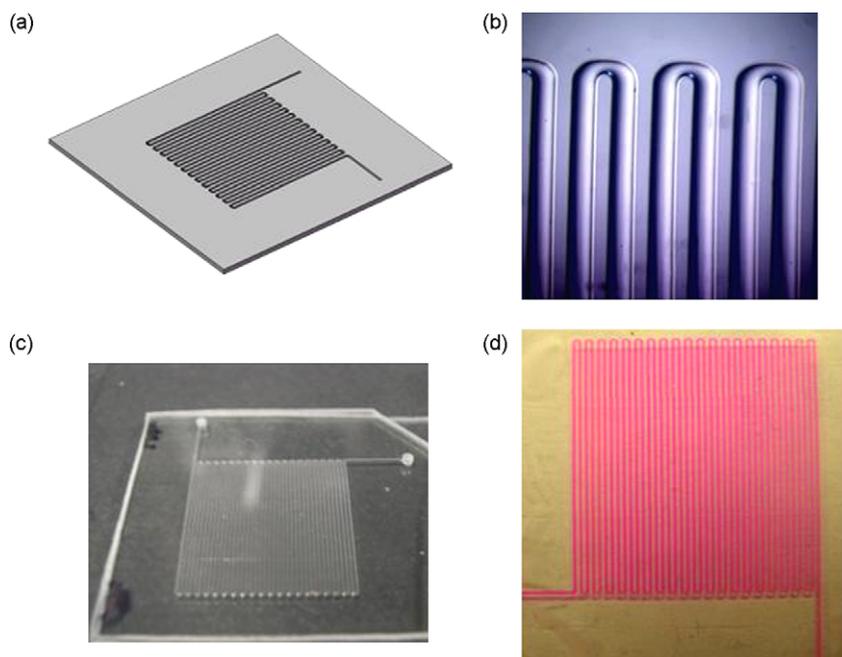
Fig. 4. (a) A representation of the fabricated microchannel created via two-stage embossing, (b) a photograph of the microchannel (400 μm width, 100 μm depth, and 1 m length), (c) PMMA–PMMA microchip fabricated with surface-modification bonding technique, and (d) bonding test with rhodamine B dye to check for leaks. The channel was formed by sealing the PMMA–PMMA chips by surface modification technique.
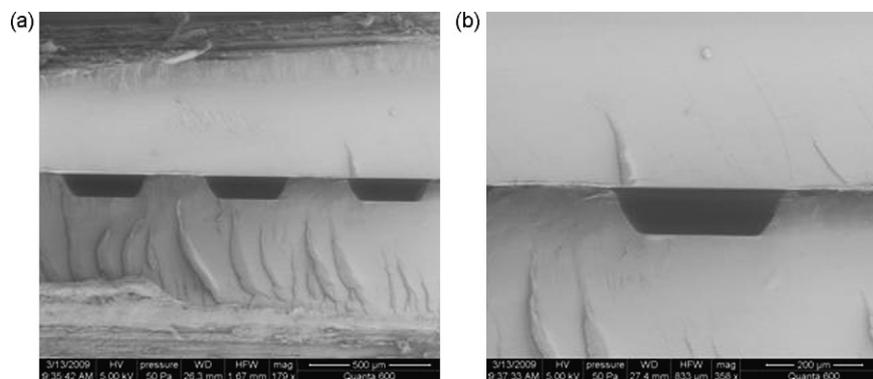
Fig. 5. SEM images of PMMA–PMMA microchip showing the microchannel cross-section: (a) the cross-section of three adjacent channels, (b) a magnified view of one channel shown in (a).

introduced at the inlet of the microchannel. No leaks were observed in or between the channels. Fig. 4d shows a photograph of the dye solution flowing through the PMMA channels.

The surface modification and bonding process was evaluated for various polymer substrates (PMMA, APET, and PC). The bonding procedure was repeated ten times for each substrate to demonstrate the reproducibility of the technique. Fig. 5 shows SEM images of a PMMA–PMMA microchip bonded using the surface modification technique. The cross-sections of three adjacent microchannels and the magnified view are shown in Fig. 5a and b, respectively. Despite the low aspect ratio of the channels, no evidence of channel wall deformation was seen to result from the bonding process. These results clearly demonstrate the utility of this bonding method in microchip fabrication.

Bonding of PMMA substrates (T_g is 106 °C) was carried out at a temperature of 75 °C. As expected, no bonding was observed for untreated PMMA chips at this temperature. Therefore, bonding of the surface modified chips at this temperature may be attributable to the relatively strong chemical interactions occurring at the modified polymer surface.

Many μTAS applications still require the use of glass due to the benefits that glass substrates offer. In this study, direct bonding of organic polymers (such as PMMA, APET, or PC) to inorganic substrates (such as Si or glass) was also demonstrated using the O_2 plasma/TEOS surface modification technique. The two substrates, however, have very different thermo-mechanical properties, namely Young's modulus and thermal expansion coefficient. For example, these properties differ by more than a factor of 10 for PMMA and glass substrates [28]. Thus, bonding of these two substrates required strict control of pressure and temperature to prevent fracturing the glass during the bonding process. Based on experimental observations, temperature was one of the most important parameters to control. In order to avoid bond failure at the PMMA/glass interface, cooling rate was decreased and optimized to minimize thermal stress between the two pieces.

Bond strength was evaluated by tensile strength measurement of the bonded PMMA/PMMA microchips. An average bond strength of 4000 kN/m^2 was measured for the O_2 plasma/TEOS-treated samples. The bond strength obtained for the PMMA samples was greater than previously reported values for other bonding procedures. The tensile strength values for thermal bonding and plasma bonding of PMMA chips were 1000 and 600 kN/m^2, respectively [15]; thus a significant improvement in bond strength was achieved for PMMA samples bonded using the O_2 plasma/TEOS technique.

The O_2 plasma/TEOS bonding technique described here offers robust microchips that are amenable to high pressure applications such as chromatography, where a high internal pressure is often required. The higher bonding strength produced using this technique presents an ideal alternative for polymer microchip bonding that obviates the need for high bulk temperatures (thermal bonding) and strong solvents (solvent bonding).

4. Conclusions

An improved method for bonding polymer devices via a surface modification technique (oxygen plasma and TEOS treatment) was presented. This technique readily produced complete microfluidic chips without the need to use strong solvents or high temperatures. The method was straightforward and the integrity of microfluidic features was successfully preserved after bonding. A high bond strength was realized, with a value approaching that of solvent-bonded chips.

This fabrication method provides a cost-effective, simple, and versatile approach to bonding polymer-based microdevices. The technique lends itself readily to many polymers and Si-containing substrates, such as PDMS and glass, facilitating device production for a variety of applications and even enabling hybrid chip fabrication.

Acknowledgements

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David T. Mandrell received his BS in Industrial Engineering from Oregon State University in 2009 and is currently pursuing a MS with the Remcho Group. In addition to studying at OSU, he works collaboratively at the Oregon Nanoscience and Microtechnologies Institute (ONAMI) specializing in nano/micro product and capability development.

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