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Centrifugal Casting of Microfluidic Components With PDMS¹

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This work describes the centrifugal casting and fast curing of double-sided, polydimethylsiloxane (PDMS)-based components with microfeatures. Centrifugal casting permits simultaneous patterning of multiple sides of a component and allows control of the thickness of the part in an enclosed mold without entrapment of bubbles. Spinning molds filled with PDMS at thousands of revolutions per minute for several minutes causes entrapped bubbles within the PDMS to migrate toward the axis of rotation or dissolve into solution. To cure the parts quickly (<10 min), active elements heat and cool cavities filled with PDMS after the completion of spinning. Microfluidic channels produced from the process have a low coefficient of variation (<2% for the height and width of channels measured in 20 parts). This process is also capable of molding functional channels in opposite sides of a part as demonstrated through a device with a system of valves typical to multilayer soft lithography. [DOI: 10.1115/1.4023754]

Introduction

The thermosetting polymer, PDMS, is the most commonly used material in microfluidics [1]. Even though the use of PDMS is ubiquitous in academia, there is a technological gap between building a few PDMS-based devices in the lab and large quantities for widespread use [2]. To decrease this gap between prototyping and manufacturing, this work focuses on processing techniques to move PDMS-based prototyping toward industrialization. The process includes centrifugal casting and fast curing of PDMS [3,4] to reduce the amount of time required for degassing and curing parts. Centrifugal casting is also capable of molding features on multiple sides of parts with fixed thicknesses. The objective of this work is to produce PDMS-based parts suitable for microfluidic applications without visible air bubbles using a method that might scale as a manufacturing process.

PDMS, as a material, has many useful attributes for the fabrication of microfluidic devices [5,6]: transparency to light down to 230 nm, lack of toxicity for cells, controlled adhesion to other surfaces, and fidelity in the replication of very small features (features less than 2 nm in the vertical direction [7] and on the order of 30 nm in lateral directions [8]). PDMS-based microfluidic devices have performed a variety of complex biochemical processes, including polymerase chain reaction (PCR) [9], DNA sequencing [10], and flow cytometry [11]. PDMS has also been used with cell sorters [12], microbioreactors [13], culture of stem cells [14], immunoassays [15], and harvesting of energy in fuel cells [16]. In nonmicrofluidic applications, PDMS is a material for microcontact printing [17], soft lithography [18], molding scaffolds in tissue engineering [19], and transfer of carbon nanotubes in the construction of solar cells [20].

Silicone-based parts (not just microfluidic devices) with micro- or nanofeatures are typically prototyped using the following process. First, the PDMS base and curing agent are mixed together, which is often done with a Thinky™ mixer (planetary centrifugal mixer made by Thinky Corporation, Tokyo, Japan), by hand, or some type of stirring device. Next, the mixed material is placed in a vacuum chamber to remove bubbles introduced during the mixing. PDMS is then poured into a mold, and the mold and PDMS are placed in an oven. In some cases, the mold and the PDMS might be placed in a vacuum chamber before curing to remove

bubbles entrapped in the PDMS during pouring. After the PDMS has cured in the oven, it is manually peeled off the mold. Reported times for degassing PDMS range from 15 min at ~15 Torr [21] to an hour between 0.02 and 0.05 Torr [22,23]. Reported times for curing with prototyping of PDMS-based devices range from 10 min at 150 °C [24] to overnight at room temperature [25,26].

In prior work, PDMS was molded with a specified thickness between fixed boundaries. Kendale and Trumper produced stamps with a thickness of 100 μm between fixed boundaries and attempted to remove as many bubbles as possible with a vacuum while allowing the material to cure at room temperature for approximately 24 h [27,28]. Jo, et al. patterned and produced PDMS parts with a thickness on the order of 100 μm to create three-dimensional microfluidic structures [23]. More recently, Lucas et al. demonstrated double-sided molding of PDMS using a similar technique [29]. In both of these prior works, a flexible top piece was carefully placed/rolled over liquid PDMS in such a way that entrapment of bubbles was avoided.

Devices such as those produced by Unger et al. [30] and Thorsen et al. [31] have intricate microfluidic architecture and circuitry, which use multilayer stacks of PDMS parts with channels on one side of each layer. One layer has control channels that are pressurized or swelled for blocking channels in an adjacent layer. The flow channels in the adjacent layer are then blocked with the pressurization of the control channels. With this architecture, Unger et al. [30] and Thorsen et al. [31] developed intricate structures in multiple layers of PDMS. However, the bonding and alignment procedures used to stack the required PDMS layers are tedious because each PDMS layer is very flexible/floppy. By patterning multiple sides of a PDMS part, it is possible to simplify the bonding and alignment steps involved with multilayer soft lithography (MSL) and to fabricate functional devices more quickly.

Using the centrifugal casting process as outlined in this paper, it is possible to produce microfluidic channels in PDMS with repeatable dimensions (standard deviation less than 2% for a run of 20 parts). Centrifugal casting facilitates control of the overall thickness and simultaneous molding of features into top and bottom surfaces of molded parts. This process is also amenable to replication from micromilled molds and molds produced from hot embossing of bulk metallic glass (BMG) [32].

Experimental Design of Centrifugal Casting and Fast Curing

The centrifugal casting described in this work requires a set of molds and a modified centrifuge (Spectrafuge 24D, Labnet

¹This manuscript contains data from author Mazzeo's Ph.D. thesis [3].

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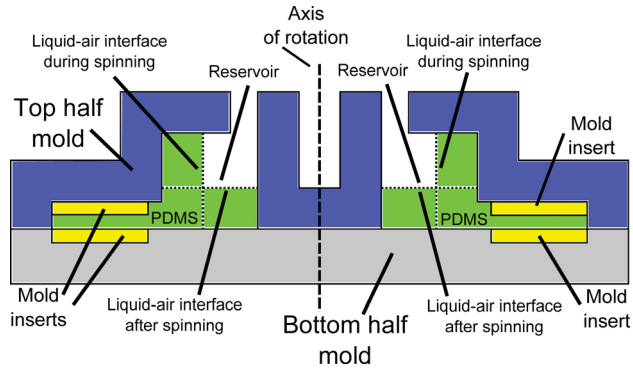


Fig. 1 Layout for centrifugal molding of two separate parts. When stationary, gravity dominates the hydrostatic state of the PDMS. When spinning at high speeds, the PDMS has a vertical liquid-air interface near the center axis of rotation.

International, Woodbridge, NJ) capable of spinning the molds about an axis of rotation. The modified centrifuge has a custom-built cylindrical rotor with a cutout to allow mounting of a molding block that assembles/disassembles into two halves with two reservoirs. Figure 1 depicts the setup with two reservoirs used for centrifugal casting. PDMS (Sylgard 184, Dow Corning, Midland, MI, in the standard 10:1 ratio) is dispensed into the reservoirs through static mixers. During dispensing of the PDMS, the fluid entraps air bubbles. To remove the entrapped bubbles, the molds are spun in the modified centrifuge. During spinning, buoyant forces drive the bubbles toward the center of the mold and out of solution, while increases in the pressure of the fluid cause the dissolution of other bubbles into solution before they have sufficient time to reach the liquid-air interface near the axis of rotation. With sufficient speed and time, it is possible to remove or dissolve the entrapped bubbles. Because buoyancy and diffusion compete with each other to remove bubbles, bubbles of a critical size require the longest time for removal [4].

A subtle consideration for the design of the spinning reservoirs is that the horizontal liquid-air interface within the reservoirs becomes vertical as shown in Figs. 1 and 2. Using a taller, stepped region in the reservoir toward the center axis of rotation allows excess fluid to collect and contribute to an increase in the hydrostatic pressure between the mold inserts. The stepped design also facilitates spinning of the “open” mold with only minor spilling of the PDMS during centrifugation.

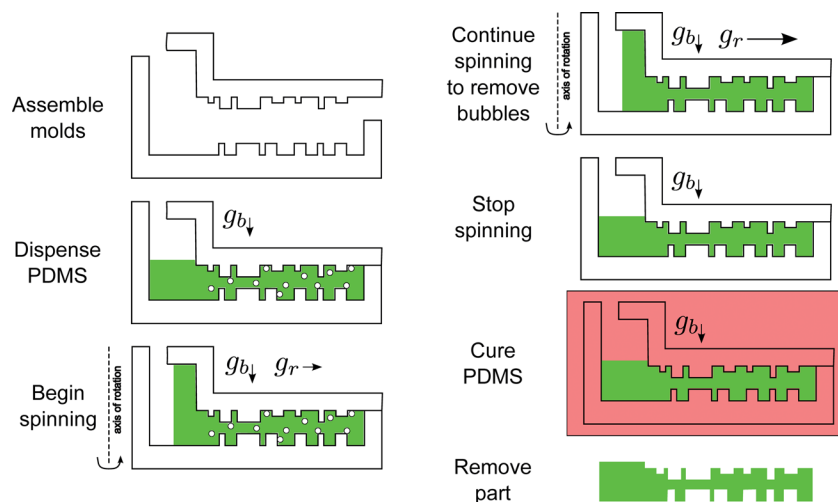


Fig. 2 Centrifugal casting and fast curing process for producing multisided devices. The parameter g_b is acceleration due to the gravity of the earth and g_r is acceleration from spinning.

Curing PDMS quickly in less than 10 min [24] is conducive to manufacturing. While the kinetics of curing PDMS are beyond the scope of this paper, Wong’s thesis provides a rigorous study of the curing kinetics of PDMS and their relationship to manufacturing [33]. As a proof-of-concept experiment, PDMS was poured directly onto a thin piece of sheet metal ($\sim 200 \mu\text{m}$ thick) resting on a hot plate at a temperature greater than 200°C . Within 30 s of pouring the PDMS onto the hot plate, the PDMS cured or hardened enough so that it could be peeled off the hot plate.

Based on this simple experimental result, a heating and cooling station (see Fig. 3) was designed to cure PDMS in molds after being spun in a centrifuge. In this heating and cooling station, it was possible to cure and cool the bubble-free PDMS in 8 min. The customized curing station heated the PDMS parts up to a temperature between 90°C and 100°C in 4 min. After 4 min of heating, the curing station then cooled the PDMS down to less than 30°C in an additional 4 min. For electrical heating, aluminum nitride (ceramic) Ultramic heaters (Watlow, Chicago, IL) were sandwiched between two thin pieces of aluminum as shown in Fig. 3(b). The heaters were raised to make contact with the mold by a pneumatic cylinder at the bottom of the heating and curing station. When the heating portion of the cycle was completed, the pressurized cylinder lowered the heaters away from the mold. To begin cooling, a pressurized cylinder at the top of the curing station pushed the cold blocks shown in Fig. 3(c) down to make contact with the top of the mold. The cold blocks were mounted to a cold plate (Lytron, Woburn, MA) with a circulating mixture of ethylene glycol and water chilled by an 1175 MD Signature Refrigerated Recirculating Chiller (VWR, Radnor, PA). Additional details concerning the heating and curing station, along with profiles of measured temperature are available in Ref. [3].

Results and Discussion

Removal of Bubbles and Molding of Features. Removal and dissolution of bubbles of PDMS during centrifugation has been modeled as described in Mazzeo [3] and Mazzeo, et al. [4]. To demonstrate the speed at which centrifugation can remove bubbles from solution, Fig. 4 shows two images from a high speed camera taken 3 s apart. Within just a few seconds, many of the bubbles shown in Fig. 4(a) are no longer present in Fig. 4(b). Although we only show two images, high speed video demonstrates that many of the bubbles shown in Fig. 4 were removed by buoyant forces pushing them toward the center axis of rotation. While not all bubbles are removed this rapidly, the theory developed in Ref. [3] shows that there is a well-defined time when all bubbles are either

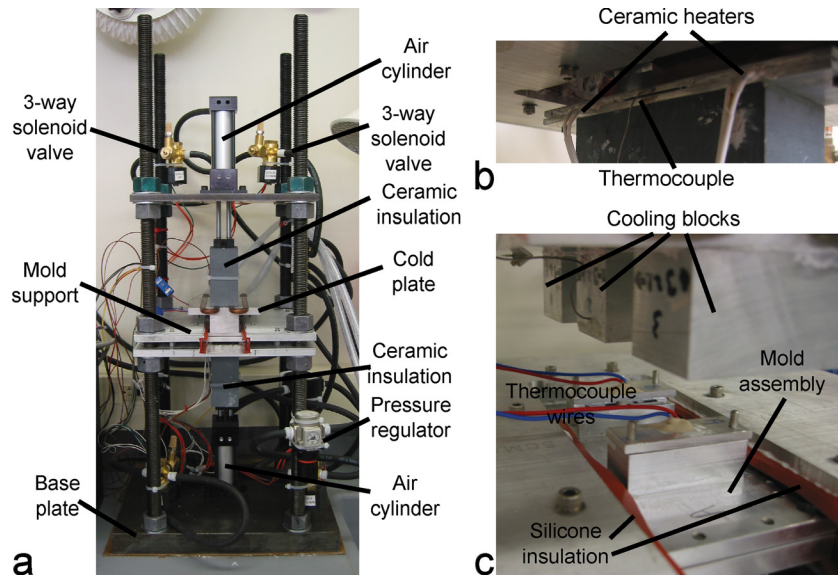


Fig. 3 (a) Heating and cooling station for curing PDMS in a mold assembly for centrifugal casting. The total height of the system is 91 cm. (b) Ceramic heaters on a pneumatic cylinder rise to make contact with the bottom of the mold and heat the mold assembly for 4 min. (c) After heating the PDMS is complete, a set of chilled blocks come down from the top to cool the mold for 4 min.

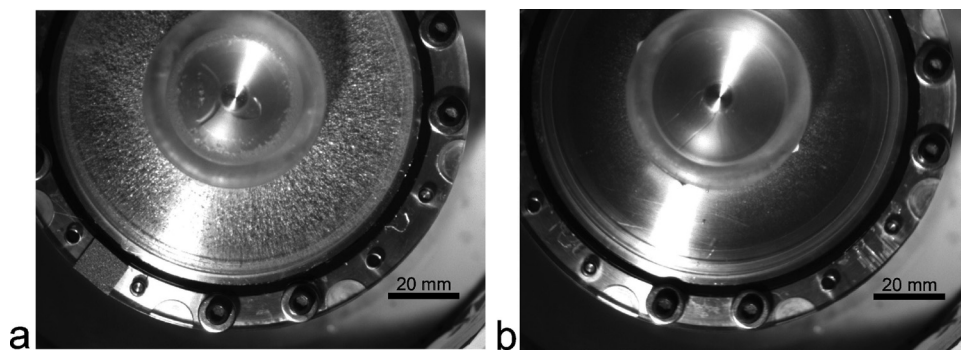


Fig. 4 (a) High speed video image taken while a spinning mold with a transparent top is accelerating up to 5000 rpm. (b) High speed video image taken 3.4 s later, while the centrifuge is still spinning. The PDMS solution within the disk contains fewer bubbles.

removed by the effects of buoyancy (bubbles above the critical size) or by dissolution (bubbles below the critical size). As shown in Fig. 5, the longest removal times are for those bubbles at or near the critical size. For typical PDMS properties and parameters for centrifugal casting in this work, this critical size is on the order of $100\ \mu\text{m}$. The critical bubble size is dependent on the spinning parameters (spin speed and slew rate), the geometry of the mold (in particular, the distance of the liquid-air interface from the axis of rotation), properties of the resin (density, viscosity, and surface tension), initial concentration of dissolved air in the solution, environmental pressure, temperature, and the starting position of the bubble [4].

Figures 6(a), 6(b), and 6(c) show a few examples of parts free of bubbles in the desired regions of interest, while Fig. 6(d) shows one part full of bubbles. The example part in Fig. 6(d) was not spun long enough at a high enough speed to remove all of the bubbles in the middle of the part. The PDMS parts in Figs. 7(b) and Fig. 7(d) were spun up to 7000 rpm with a slew rate of 300 rpm/s for 7 min of spinning (not including deceleration time). At a spin speed of 7000 rpm with the geometry used in this work, the estimated maximum pressure within the mold was 7 atm. After being centrifugally cast, the PDMS parts shown in Figs. 6(a), 6(c), and

6(d), along with those in Figs. 7(b) and 7(d) were cured in a period of 8 min.

In replicating micro/nanofeatures with molding processes, there is a need to ensure proper demolding and fidelity of the molded features to the master or mold being used. To show that centrifugal casting is amenable to use with different types of molds, parts were replicated from molds created in bulk metallic glass and aluminum. Figure 7 shows images of the Y-mixer portions of molds made from hot embossed BMG [32] and micromilled aluminum. These images were taken with a NewView 5000 (Zygo Corp., Middlefield, CT) optical profilometer. Figures 7(b) and 7(d) show corresponding features centrifugally cast in PDMS off of bulk metallic glass and aluminum. The heights of the channels were nominally $40\ \mu\text{m}$.

Part-to-Part Variation. To evaluate the potential part-to-part variation of a large-scale manufacturing process based on centrifugal casting and fast curing, parts were produced in a mini/manufacturing run using the BMG mold used to mold the features shown in Fig. 7. A channel section 0.5 mm below the Y-junction where the two inlet streams meet and become one stream was

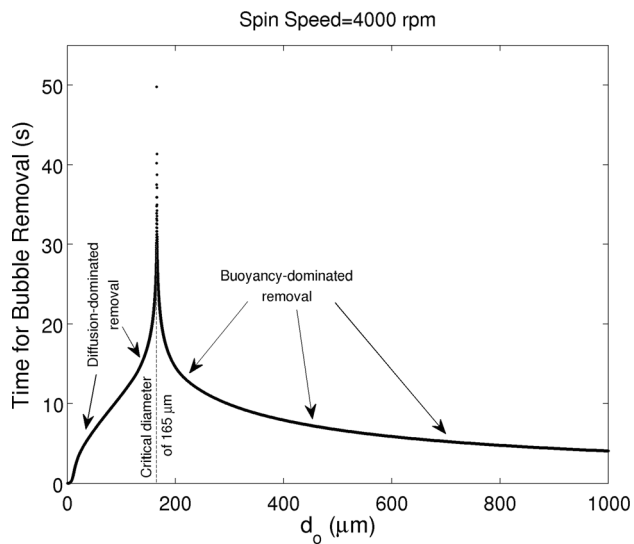


Fig. 5 Simulated time to remove bubbles from spinning PDMS (Sylgard 184) versus their initial diameter before spinning. There is a critical diameter (165 μm for this set of parameters) for which removal will require the longest amount of spinning. To the left of the peak, diffusion dominates bubble removal (i.e., small bubbles dissolve into solution). To the right of the peak, buoyancy dominates removal of the bubbles (i.e., large bubbles travel to the liquid-air interface near the center of the centrifuge). The centrifuge spins up to 4000 rpm with a slew rate of 300 rpm/s, each bubble starts 6.2 cm from the center axis of rotation, and the liquid-air interface is 3.4 cm from the center axis of rotation. See Ref. [4] for more information.

measured on 20 consecutively produced, centrifugally cast parts. The height of the channels was nominally 40 μm and the width was 50 μm . These parts were spun for 1.5 min (1 min of energized spin time followed by 30 s of deceleration). The set spin speed was 7000 rpm, and the accelerating slew rate was 300 rpm/s. The

parts were then cured in 8 min with a maximum temperature of 100 $^{\circ}\text{C}$. The height and width measurements were calculated using the algorithm described in appendix B of Ref. [3]. Within the regions of interest for the 20 consecutive parts, only one bubble with size on the order of a millimeter was observed, and no other bubbles were visible by eye.

Initial measurements to determine the repeatability of the measuring process itself with the Zygo optical profilometer showed the standard deviation of the height and width measurements to be less than 200 nm (coefficient of variation less than 0.5%) for loading, measuring, and unloading the same part 20 times. The resulting measurements for a run of 20 produced parts are shown in Fig. 8. The standard deviation for the height measurements was submicrometer (less than 0.5 μm), and the standard deviation of the width measurements was also submicrometer (less than 0.8 μm). With respect to the heights and widths of the channels, the coefficient of variation (standard deviation divided by the measured mean) was less than 2%.

Casting Double-Sided Parts. In addition to replicating microfeatures on a single side of a PDMS part and controlling the overall thickness of the part, centrifugal casting is useful for replicating features simultaneously on multiple surfaces of a part. As mentioned previously, Unger et al. [30] and Thorsen et al. [31] produced intricate, multilayered microfluidic architectures. However, since bubble removal without spinning requires a large open surface to permit rapid removal of bubbles, each layer of channels in their work was molded in an individual piece of PDMS, which was then aligned and bonded to another molded piece of PDMS. Typical bonding and alignment procedures used to stack the required PDMS layers can require manual manipulation of the flexible layers of materials with tweezers under a microscope. In addition, if one uses plasma to prepare the surfaces for bonding and aligns the two flexible layers poorly to each other, the parts can stick to each other, making a second attempt impossible because of the irreversible bond [5]. The use of centrifugal casting to remove the bubbles eliminates this restriction and permits

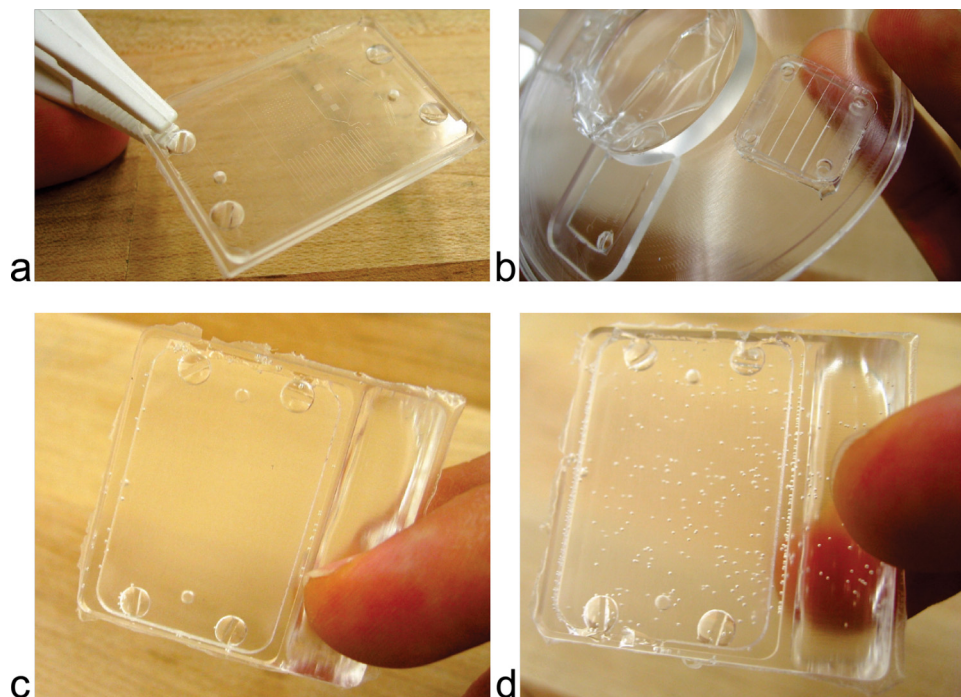


Fig. 6 (a) PDMS part with microfluidic channels. (b) PDMS part with a diameter of 10 cm, that includes some microchannels (bottom left of image), along with some larger ones. (c) Blank PDMS part used for evaluating appropriate spin times and spin speeds. (d) Same as (c) but not spun long enough and fast enough to remove all the bubbles

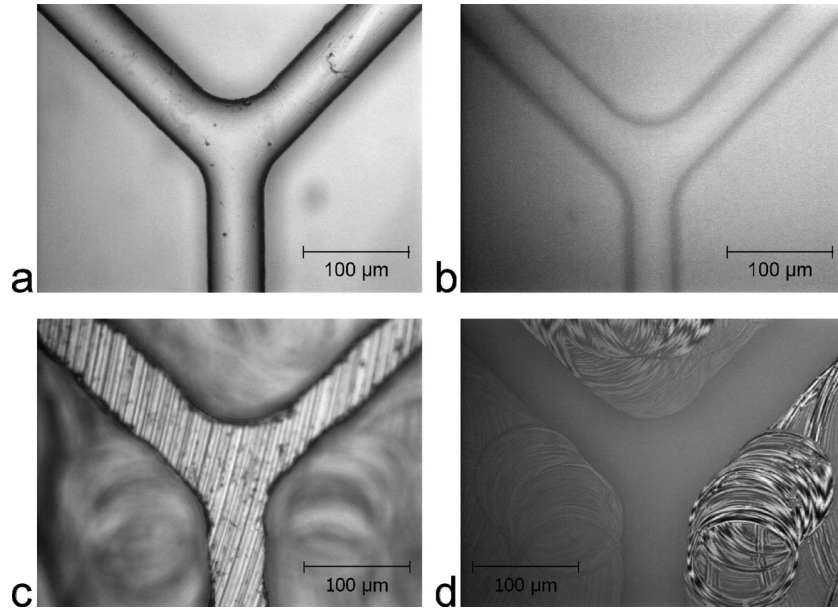


Fig. 7 (a) Protruding channels for a Y-mixer in a mold created in bulk metallic glass. The measured height using the algorithm in appendix B of Ref. [3] was 37.9 μm . (b) Y-mixer channels in a PDMS part produced by centrifugal casting off of bulk metallic glass features shown in (a). The measured height of the channels was 36.8 μm . (c) Y-mixer micromilled in aluminum. (d) Y-mixer channels in a PDMS part produced by centrifugal casting off of the micro-machined features shown in (c). As a result of molding PDMS off of (a) to produce (b), (b) is a mirror image of (a). The images in (c) and (d) are also mirrored.

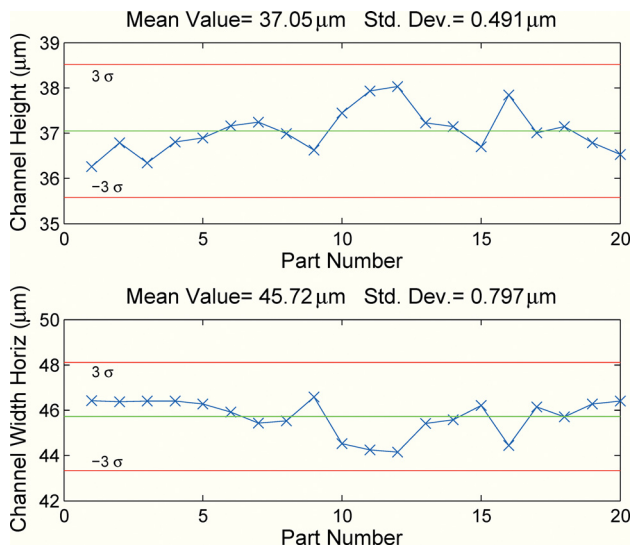


Fig. 8 Run chart of the specified channel section measured on 20 different parts produced with the centrifugal casting and fast curing process

patterning multiple sides (the top and bottom portions) of a single PDMS part with rigid, mated molds. With centrifugal casting, there is the potential to simplify/eliminate the bonding and alignment steps involved with multilayer soft lithography (MSL) and to fabricate functional devices more quickly.

To produce thin, double-sided PDMS parts (i.e., parts with replicated features/channels on their top and bottom surfaces), the setup with the modified centrifuge shown in Fig. 9 was used. Two PDMS parts were cast simultaneously in two separate molding regions. In one of the molding regions, there was a mold insert with micromachined features for each side of the cast PDMS part. One mold insert had bosses (250–500 μm wide and 500 μm tall)

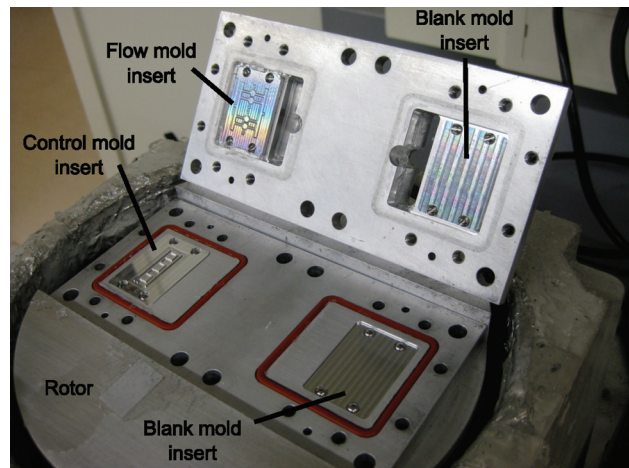


Fig. 9 Open mold assembly sitting on a centrifuge. Two parts are centrifugally cast simultaneously. The mold inserts for the double-sided MSL architecture are shown on the left. Blank mold inserts on the right are used to produce one of the cover pieces used to complete the assembly of functional devices. The two halves of the mold are held together with a set of 12 bolts.

for the control channels, while the other mold insert had bosses for the flow channels with heights of 10 μm and widths of 130 μm . Figure 10 shows images obtained with the Zygo optical profilometer for portions of the flow channels.

The flow channels were machined with a steplike pattern in an attempt to approximate the semicircular/curved flow channel designed by Unger, et al. [30]. Semicircular/curved flow channels are easier to block with lower pressures in the control channels. To produce a similar stepped structure with standard semiconductor processing techniques would require multiple masks/etching steps. These machined channels had steps of 2.5 μm in height and

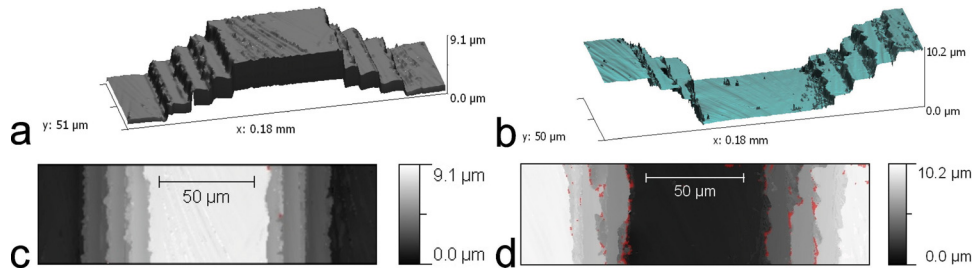


Fig. 10 (a) Rendering of measurements made with an optical profilometer on a portion of a channel micromachined into an aluminum mold insert. (b) Rendering of measurements made with an optical profilometer on a portion of a channel molded into a double-sided PDMS part. (c) Same region shown in (a) with height data depicted in gray scale. (d) Same region shown in (b) with height data depicted in gray scale

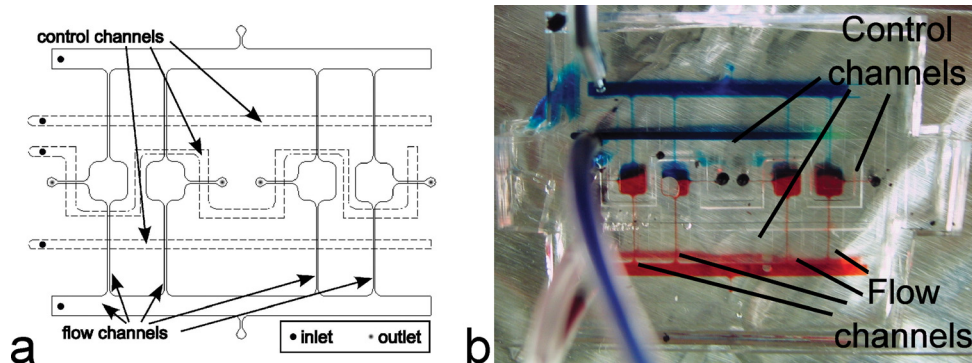


Fig. 11 (a) Schematic diagram of the device. The serpentine control channel (generally unused) was designed to block the flow channels leading to the outlets. (b) Image of food coloring being introduced to a completed microfluidic device. The flow channels on the double-sided component are facing the glass slide at the bottom of the stack, while the control channels on the double-sided component are bonded to a top PDMS blank.

represent an initial attempt to approximate the curvature common to flow channels. The channel profiles shown in Fig. 8 are just portions of the machined and replicated channels produced in PDMS. Further work would be required to improve the uniformity of the cross sections shown along the length of the channels.

Micromachining multiple heights in the mold was also performed to prevent collapsing channels in the molded and bonded parts. The microfluidic design demonstrated has simple squarelike reservoirs with edges of 1.5 mm in length. If the depths/heights of the reservoirs were restricted to 10 μm , they would have most likely collapsed. To avoid collapse in these regions, the height in these areas was increased to 50 μm . In this way, it was still possible to have small, valvelike regions and large reservoirs in the same device patterned off of the same mold.

The overall thickness of the double-sided parts was determined by the open gap between the two mold inserts. The gap between the mold inserts was set to produce parts with a thickness of approximately 650 μm . To fine tune the thickness of the membranes between control and flow channels, shims were placed behind the mold inserts. Increasing the thickness of the shims decreased the thickness of the membranes, while decreasing the thickness or removing the shims resulted in thicker membranes and a slightly thicker part.

The completed PDMS parts with an overall thickness of 650 μm had a membrane thickness of 140 μm at the crossovers of the control and flow layers. In a production environment, it would be critical that these spacings and the uniformity of spacings over the tool be controlled more carefully. Any variation would lead to nonuniform thicknesses of the membranes with the potential for tearing or leaking of the valves.

Figure 11(a) shows the schematic of the microfluidic devices produced for this work. The control channels are represented by

dashed lines, and the flow channels are represented by the solid lines. The control and flow channels are on opposite sides of the molded PDMS. When the control channels are pressurized, the flow channels are blocked. The four chambers in the middle of the device are reservoirs where different fluids from the two sets of flow channels meet. The two straight control channels were designed to block the flow channels leading into the reservoirs. The pressurization of the two straight control channels can be alternated to block the two sets of flow channels periodically. By alternating the pressurization of the straight control channels, the filling behavior of a reservoir can be altered or partially controlled.

Figure 11(b) shows a completed microfluidic device. The centrifugally cast and fast cured double-sided PDMS component was first bonded to a blank piece of PDMS by exposing both mating surfaces to air plasma in a Plasma Excitor 300-1 (product of the former Technics, Inc.) for 45 s with a plate current of 70 mA and a radio frequency power of 25 W. The inlet and outlet holes with a diameter of 0.5 mm were then punched through both pieces of bonded PDMS using a Harris Uni-Core (coring tool available from Ted Pella, Inc., Redding, CA). To encapsulate the remaining, exposed channels, the bonded PDMS parts with punched holes were then bonded to a glass slide using the same parameters for plasma treatment. The completed device was then left overnight on a Cimarec, aluminum top, hot plate set to 100 $^{\circ}\text{C}$ to strengthen the plasma-activated bonds.

The completed device was then connected to Tygon tubing and four separate syringes. Two syringes were used to pressurize the two straight control channels, and two syringes were used to flow food coloring through the flow channels. When the control channels were not pressurized, the colored fluid in the flow channels passed freely and continuous lines of colored fluid were visible in

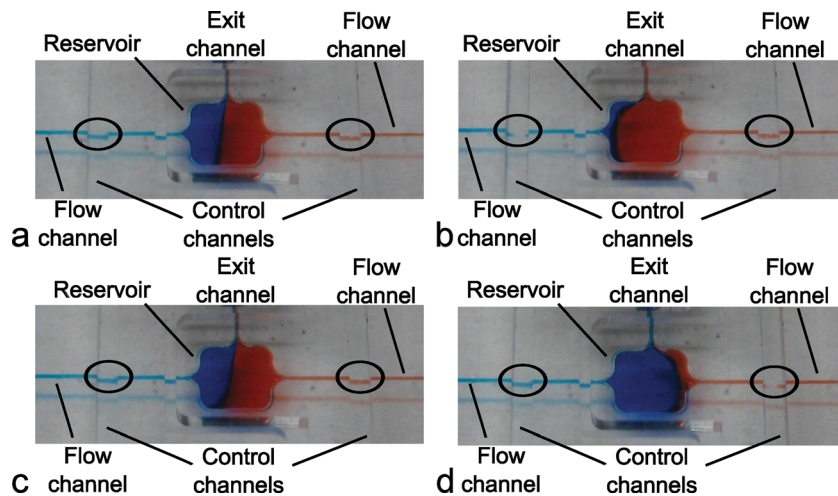


Fig. 12 Images of pneumatically actuated channels controlling microfluidic flow. (a) Flow channels are not blocked; reservoir is evenly filled. (b) Left flow channel is block; reservoir fills with food coloring from the right (interface between fluids moves to the left). (c) Flow channels are not blocked; reservoir is evenly filled. (d) Right flow channel is blocked; reservoir fills with food coloring from the right (interface between fluids moves to the right). The ovals highlight the regions where the flow and control channels cross over each other.

the flow channels at the crossover region. When the control channels were manually pressurized, the flow channels were blocked.

Figure 12 demonstrates the result of alternatively pressurizing the two straight control channels. With the constant laminar flow provided by the syringe pump and unpressurized control channels, the reservoir filled evenly with blue and red food coloring as shown in Figs. 12(a) and 12(c). With the control channels in their unpressurized state, there was a distinct line in the reservoir where the two separate laminar flows met and proceeded to the outlet. When one of the straight control channels was blocked as shown in Figs. 12(b) and 12(d), the reservoir filled unevenly and the meeting line of the two laminar fluids shifted left or right. While a control channel was blocking one or more flow channels, pressure built in the blocked flow channels. When the blocked flow channels were released and both straight control channels were not pressurized, the stored pressure in the flow channel(s) forced the meeting line of the fluids in the reservoir to return to the center. This simple demonstration of blocking flow channels with pressurized control channels shows the potential of using PDMS parts produced by centrifugal casting and fast curing for microfluidic chips with multiple layers.

Conclusions

The process presented in this paper makes use of centrifugal casting and fast curing to produce microfluidic parts without visible air bubbles. This work also addresses the two rate-limiting steps of typical PDMS-based prototyping: degassing (bubble removal) and curing. PDMS components with microfeatures were centrifugally cast to remove bubbles in as little as a minute of spinning at thousands of revolutions per minute. After spinning, the parts were cured with a modest heating and cooling time of 8 min. These spinning and curing times do not represent absolute lower limits for centrifugal casting and fast curing but demonstrate the potential this process might have in a manufacturing context for low to medium volumes of production without the use of large injection molding machines (the entire setup for this process fits on a lab bench). The maximum pressures in the molds were approximately 7 atm (mold spinning at 7000 rpm), and molded regions were as thin as 140 μm . In a run of 20 consecutive parts, the measured coefficient of variation for both the height and width of selected regions on the 20 different parts was less than 2%.

The centrifugal casting process also permits simultaneous patterning of multiple surfaces of a part. The need for a large free

surface (almost half the total surface area of a typically cast PDMS part for microfluidic applications) for bubble escape is eliminated. The thickness of a designed part can be precisely controlled without the need for measuring the volume of dispensed PDMS. Furthermore, alignment between features on opposite sides of a part can be achieved by the use of rigid, mating molds. To demonstrate its utility, the centrifugal casting and fast curing process was used to successfully mold channels simultaneously on opposite sides of a PDMS part, and these channels were shown to function as membrane valves.

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