Sensors and Actuators B 235 (2016) 302–308

Contents lists available at ScienceDirect

Sensors and Actuators B: Chemical

journal homepage: www.elsevier.com/locate/snb

Rapid, low-cost fabrication of circular microchannels by air expansion into partially cured polymer

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ARTICLE INFO

Article history:
Received 9 December 2015
Received in revised form 18 April 2016
Accepted 2 May 2016
Available online 12 May 2016

ABSTRACT

This paper reports a simple fabrication process for microfluidic channels with circular cross-sectional shapes using a polydimethylsiloxane (PDMS) master and thermal air expansion. This technique can be easily used to generate circular microchannels with a wide range of diameters from 25 to 150 μm through a simple bench-top fabrication process. By controlling the gelation time of the PDMS, we can obtain circular microchannels in a variety of diameters. This technique does not require plasma-activated bonding or any alignment processes. We can apply this technique to fabricate networks of circular microchannels to simulate the vascular system, micro-concave platforms for culturing microspheroids, micronozzles for droplet-generation devices, and micro-sized patch clamps for cell immobilization.

1. Introduction

Microfluidic systems have grown rapidly since the introduction of rapid prototyping methods, especially the widely accepted standard photolithography and soft lithography techniques. However, microchannel fabrication based on standard photolithography can only produce microchannels with rectangular or trapezoidal cross-sections [1], which are commonly used in microfluidic research. Semi-circular channels are favorable for various applications, such as microvalves based on membrane push-up and micropumps used to close microchannels completely. In addition, many tissue-engineering systems, such as those used to mimic human blood vessels, require circular cross-sections to ensure uniform wall shear stress under culture conditions. Therefore, there has been a strong interest in developing technologies to fabricate microchannels with semi-circular or circular cross-sections.

Several fabrication methods were introduced to make circular microchannels. They involve replication of semi-circular cross-sections from a master. The final devices could then be produced by aligning and bonding two halves of the semi-circular PDMS microchannels [2–5]. The semi-circular channel molds were fabricated using reflowed positive photoresist [2], laser writing or micro milling [3,4], and isotropically etched silicon wafers [5]. The technique of bonding two pieces of PDMS together to generate a complete channel has a substantial disadvantage: misalignment during bonding because of manual manipulation and the elasticity of PDMS. This misalignment causes irregularities in the final channel profile, especially for channels as small as tens of microns in diameter. Therefore, other methods to directly fabricate circular microchannels have been developed, including coating solvent-diluted PDMS to modify rectangular channels [6], using sucrose as a sacrificial template material [7], absorbing polymethylmethacrylate (PMMA) solution into PDMS molds [8], expanding degradable poly(lactic acid) [9], and liquid template inkjet printing [10]. However, these methods require expensive master molds, have low reproducibility, are time consuming, and involve complex fabrication steps. Furthermore, most fabricated circular channels do not have a perfectly circular cross-section despite the relatively complicated processes.

Several groups used pre-polymer to fabricate 3-D cured microstructures by constrained gas expansion into photopolymer [11–13]. By controlling the preheating times, uniform microlens arrays as small as 2 μm were successfully produced [11]. Other groups have also applied the PDMS pre-polymer and gas-expansion technique in closed cavities to fabricate microwells [12,13]. However, the deep wells achievable with a master were required to prevent the PDMS pre-polymer from remaining wet in the microwells. These techniques were successfully used for the rapid fabrication of 3-D cured structures for embryo body formation.

Hoffman et al. used 'micro-tunable' molds to create many replicas with different curvature microstructures from a single master

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http://dx.doi.org/10.1016/j.snb.2016.05.008
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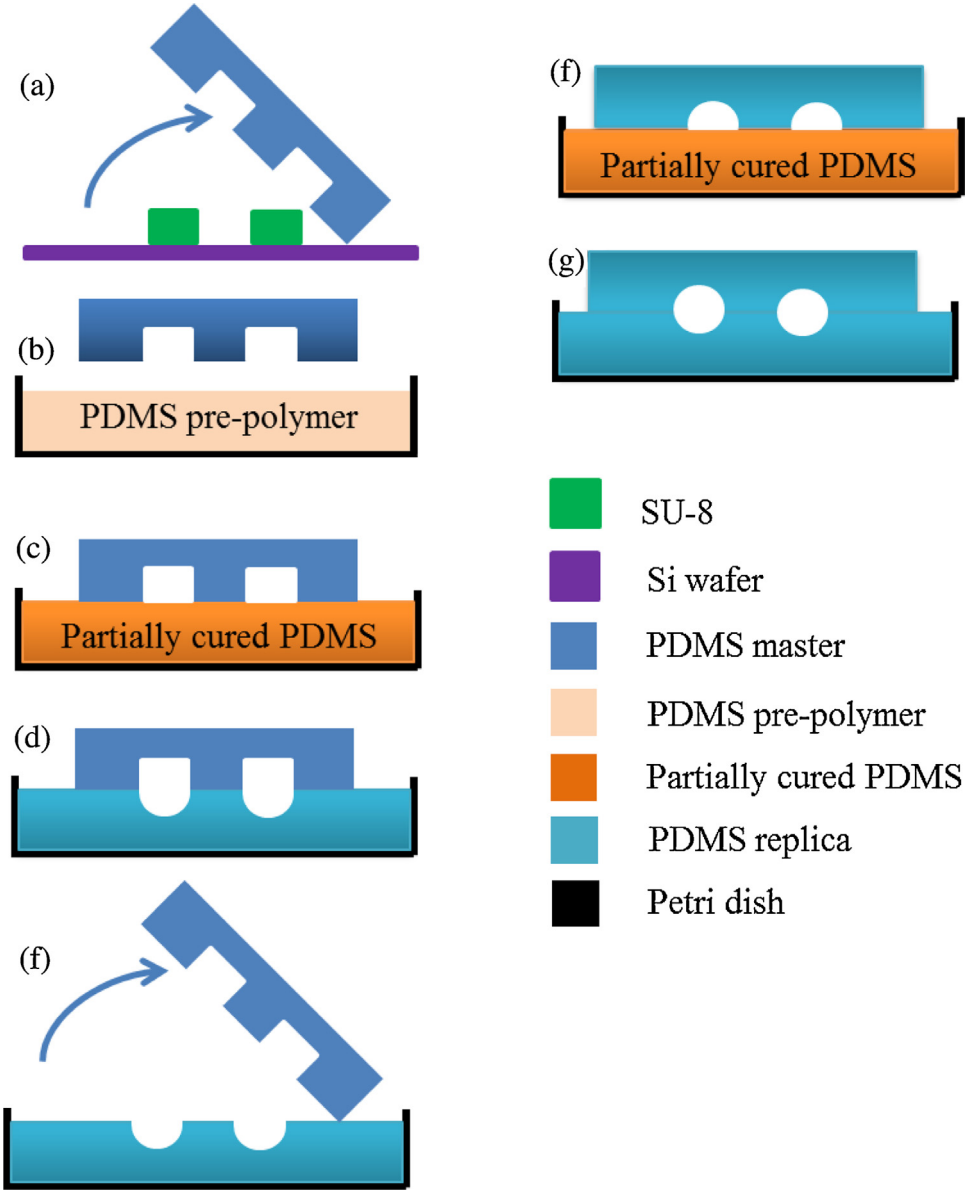

Fig 1. Schematic of the fabrication procedure for a channel with a circular cross-section. (a) A PDMS master is replicated from a SU-8 mold. (b) The PDMS master is treated with anti-adhesion solution, and PDMS pre-polymer (c) The partially cured PDMS (d) The stack is heated at 90 °C for 60 min to achieve complete solidification. (e) The master is detached. (f) The PDMS replica with the semi-circular microchannel is used as the master for circular microchannel fabrication. (g) Circular microchannel product.

[14]. In this method, a master was initially bonded to a thin PDMS membrane, and then, the closed cavity was pressurized to deform the membrane and generate the final replicated profile. One potential problem with this process is the relatively weak membrane, which is very vulnerable to damage during the peeling step if the adhesion strength at the interface is significant. Although semi-circular and circular channels can be fabricated using this technique, a simple and more flexible process is still needed. Indeed, when creating channels with widths as small as 50 μm, the subsequent membrane deformation was quite small (less than 10 μm) at 25 kPa. Therefore, this technique is more suitable for producing channels with large (hundreds of microns) diameters.

Recently, three dimensional (3-D) printing has been introduced as an easy and straightforward method to make the microfluidic devices in various shapes [15–18]. It can be used to directly create a 3D structure or be used to produce 3D sacrificial template for soft-lithography. However, current printing technique by layers for microfluidic applications still has limitations in relatively larger lateral resolution, and channel roughness problems compared to photo-lithography. Since surface roughness and lateral resolution are important factors in microfluidic devices, 3D printed microfluidics was used for applications requiring relatively large channel dimensions (>100 μm) [15,16]. For finer channel dimensions (<100 μm), a high resolution printer with high price (>$50k) and complex system [18] was required.

In this paper we continued the idea presented in MEMS 2015 conference [19] by adding experiments and analysis. We report a novel, simple method involving only a few fabrication steps and minimal equipment to produce semi-circular or circular microchannels with various diameters.

2. Material and methods

The PDMS (Sylgard 184, Dow Corning, MI) used for this work was a mixture of PDMS precursor and curing agent with volume ratio

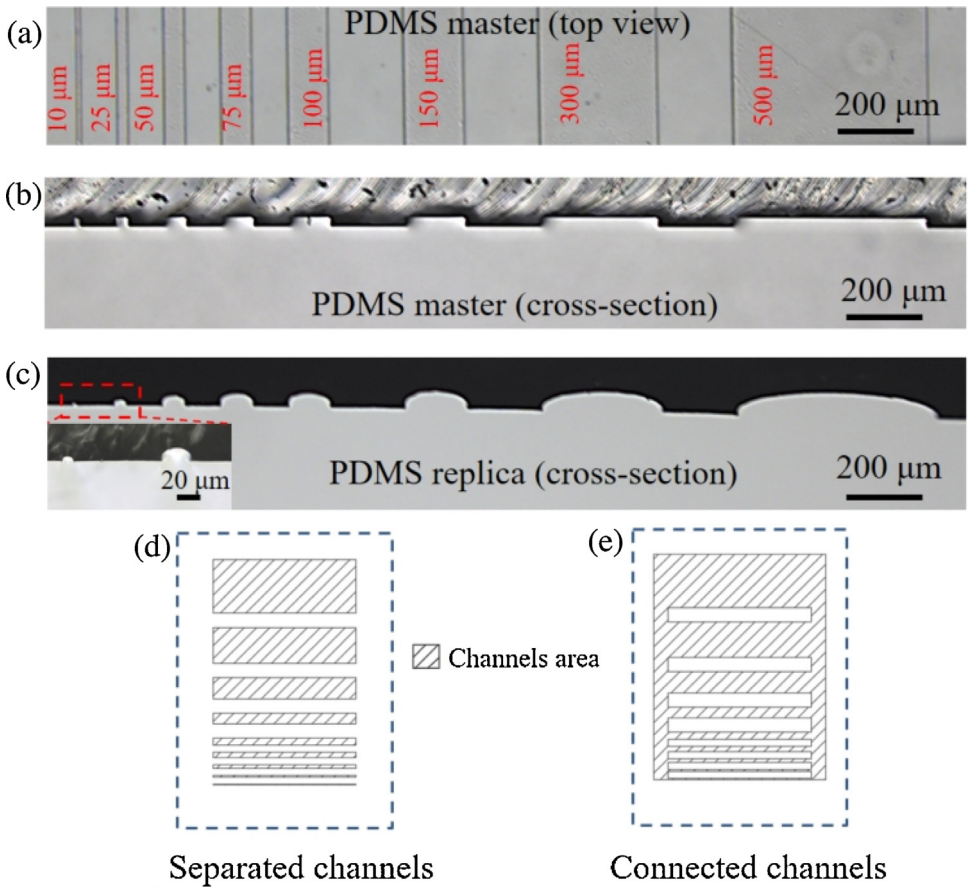

Fig. 2. Bright-field images of PDMS microchannels: (a) top view of the PDMS master, (b) cross-section of the master, (c) cross-section of the replica (channel width ranging from 10 μm to 500 μm), (d) layout of separated channels master, (e) layout of connected channels master.

of 10:1. After sufficient mixing, the liquid mixture was placed in a vacuum chamber to degas for 5 min. Hydrophobic hydroxypropyl methylcellulose (HPMC) was used as an anti-sticking agent for the temporary PDMS bonding [20]. The anti-sticking agent was made by a mixture of 0.3% HPMC and citric acid buffer in a 1:1 vol ratio.

The depth (d) of the channel was determined by obtaining a cross-section of the replica using an X-Acto knife and measuring the dimensions under an optical microscope (Olympus BX51) and accompanied software (JNOPTIC capture 2.2). The three dimensional geometry of the PDMS microchannel was obtained via scanning electron microscopy (SEM).

The fabrication process is described in Fig. 1(a) First, a mold was produced using SU-8 by standard photolithography. Then, a PDMS master was fabricated by soft lithography to replicate the SU-8 mold. The size of master was 2 cm × 3 cm × 0.2 cm, so the weight of the PDMS master was around 1.2 g. The master was composed of eight channels with a height of 25 μm and widths of 10 μm, 25 μm, 50 μm, 75 μm, 100 μm, 150 μm, 300 μm, and 500 μm (Fig. 2). (b) We soaked the PDMS master with a solution of HPMC for 2 h at room temperature. Then, the PDMS master was removed from the solution, thoroughly rinsed with deionized water, and dried in an oven at 50 °C. Without HPMC, the edges of the channels get damaged during the detachment stage in step. (c) The treated PDMS master was then slowly laminated on the partially cured 'gel-stage' PDMS starting from one edge. Without any additional normal force, the two surfaces join with natural adhesive force between the PDMS master and the sticky gel-stage replica. The joining process was done in room temperature. The gel-stage PDMS was prepared by partially curing at a hot plate (70 °C for 31–34 min) or in the oven (70 °C for 10–12 min) to increase its viscosity and surface tension. This step results in a thin solid layer on the surface of the pre-polymer and increase the surface tension of the partially cured polymer which prevents partially cured PDMS filling into the master channel. If the viscosity of partially cured PDMS is too high, the microchannels of the master is in risk of being filled up with partially cured polymer. (d) After the PDMS master made contact with the partially cured PDMS, the stack was heated on a hot plate at 90 °C for 60 min to create the expanded semi-circular microchannels and solidify simultaneously. The profile of the microchannel depends on the air-trapping cavity volume, the curing temperature, the channel width, and the standing time used to allow the pressure inside the channel to build. (e) The PDMS master was then detached, and the semi-circular microchannel was obtained without any damage, thanks to the HPMC coating. (f) To make circular channels, we used the first PDMS replica as a master and place it on a gel-stage PDMS same as step (c). (g) We then simultaneously expand and solidify the pre-polymer to complete the circular microchannel without any alignment. Because the two replicas were meant to be sealed together, the semi-circular master was not treated with an anti-sticking solution this time. Using this technique, we can obtain various channel dimensions

3. Results & discussion

In conventional soft-lithography, the PDMS pre-polymer is commonly used to replicate the mold structure because at that state, the PDMS pre-polymer behaves as a low-viscosity fluid [1]. As a result, covering the whole mold is easy, and we can exactly replicate micro to nanometer-scale features. In this study, we concentrated on controlling the PDMS polymerization process in the 'gel-stage' for air

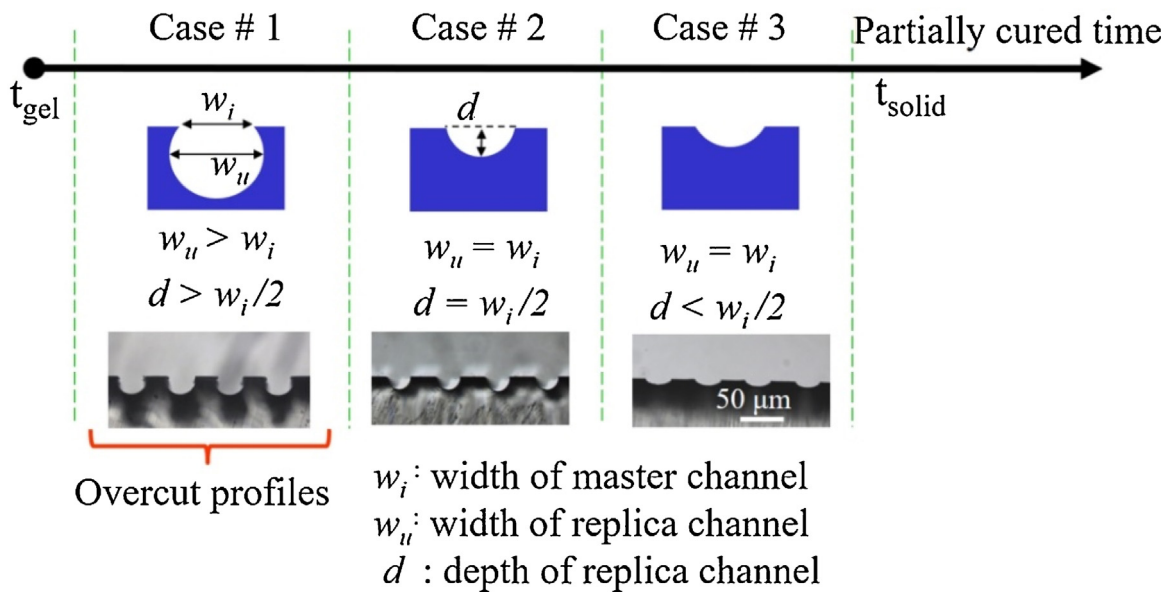

Fig. 3. Schematic diagram of PDMS gelation from the starting point to the fully cured polymer.

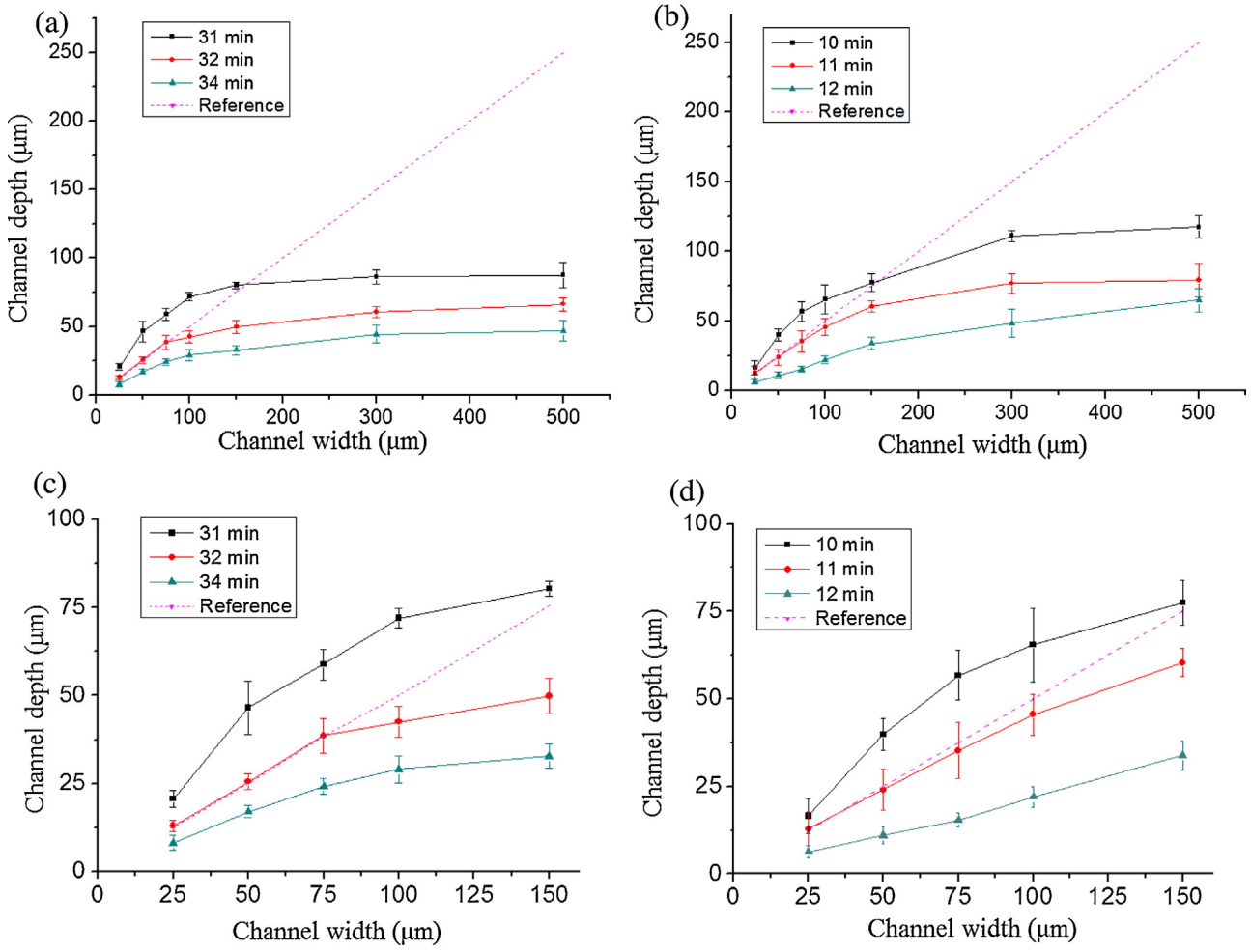

Fig. 4. The correlation between the width and the depth of the replica channel when the time allowed for partial curing was varied. (a) Partial curing in the oven; (b) Partial curing in the hot plate; (c), (d) Magnification of (a), (b) showing channel widths from 25 μm to 150 μm, respectively.

expansion molding. At the 'gel-stage', PDMS is in a transitional form between liquid and solid form. The PDMS at the gel-stage exhibits high surface tension and is more stable than at the pre-polymer stage; hence, the capillary-wetting effect can be reduced substan-

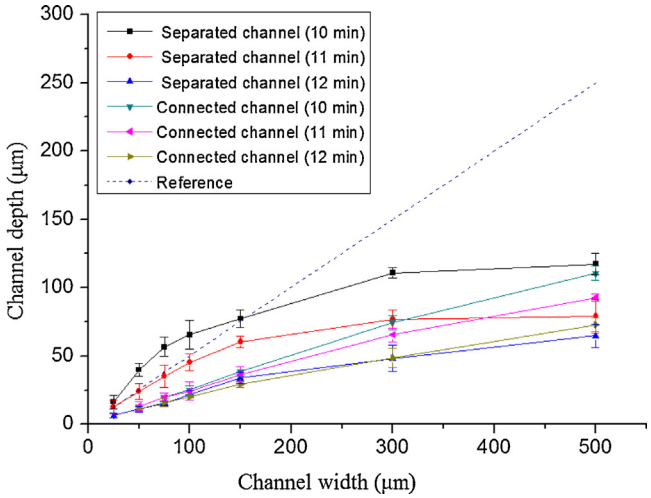

Fig. 5. The comparison of connected channel and separated channel fabricated by air molding technique in various channel widths from 25 μm to 500 μm.

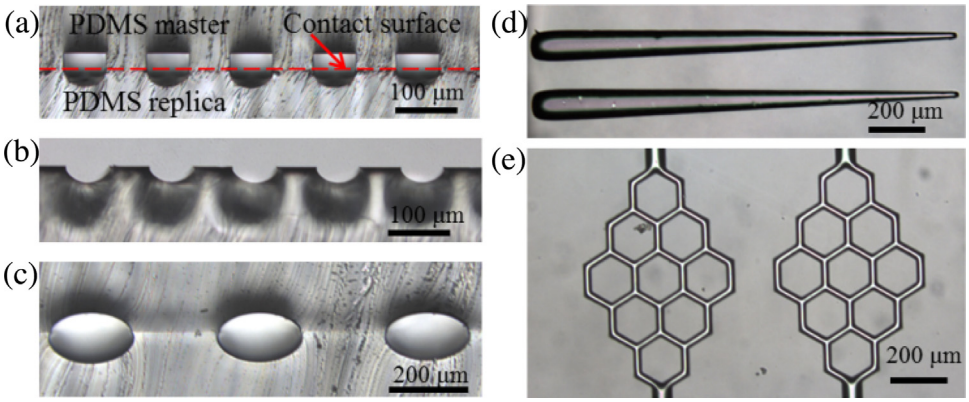

Fig. 6. Bright-field images of PDMS microchannels: (a) PDMS master with channels with rectangular cross-sections on top side and a stamp with semi-circle channels (b) Cross-sections of semi-circular channels after detaching the master. (c) Uniform circular channels. (d) Angular channels where the left end is 100 μm and the right end is 10 μm in diameter. (e) A complex planar microfluidic network fabricated in a single replica.

tially and thus prevent the gel pre-polymer from filling the stamped channels. Therefore, the gel point, at which PDMS first transitions from a liquid to a solid, is an important parameter for our molding process. From the start of the gelation, the surface tension exponentially increases, resulting in solidification of the PDMS. Wong, in his Ph.D. thesis, analyzed the curing conditions, and categorized the different gelation stages of PDMS [21].

The master consists of separated channels as shown in Fig. 2a,b,d and the connected channels as shown in Fig. 2e. The master channels have rectangular cross-section (Fig. 2b) and the depth is the same because it is replicated from the single layer SU-8 mold. Therefore, the volume of each master channel in the separated case will be proportional to the width. In the experiments, we used a series of channel widths from the 10 μm to 500 μm. However, the 10 μm width replica channel was not always successful due to partially cured polymer wetting the master channel, so we just showed the results of the replica channel width from 25 μm to 500 μm.

The deformation of a polymer by gas cavities depends on the surface tension of the partially cured polymer, the incubation temperature, and the dimensions of the master channels, including the height and width. The cross-section shape of the master does not affect the shape of the expanded channel because the shape does not depend on the master shape, but rather the width or volume of the master. When PDMS was partially cured on a hot plate, a heat gradient was created from the bottom to the top of the PDMS. As a result, the polymerization of the surface of the replica PDMS varies according to the thickness of the PDMS layer. The 1.8 mm thick PDMS stamp used in this work started gelation after 31–34 min of heating at 70 °C on a hot plate and 10–12 min of heating in the oven. The depth and lateral profile of the replicated channel depends on the gelation time. The profiles can be generally divided into 3 cases, as shown in Fig. 3 and the resulting width and depth of the replica channels with varying curing time was measured and plotted in the graph in Fig. 4.

Case 1. Overcut structures (with $w_u > w_i$ & $d > w_i/2$)

Case 2. Nearly semi-circular structures (with $w_u = w_i$ & $d = w_i/2$)

Case 3. Oval-shaped channels (with $w_u = w_i$ & $d < w_i/2$)

Where w_i is the width of master channel, w_u is the width of replica channel, and d is the depth of replica channel.

Fig. 4a and b show the relationship between the master channel width and the depth of the replica channels for different partial curing time in the case of separated channels. The pressure induced in each closed channel with different width is different between each channel. This is because the enclosed gas volume is different. Therefore, the profile of each replica channel will be different, as shown in Fig. 2c. The reference line is the case of 'perfect circular channel' ($d = w_i/2$). Fig. 4a is the case of using a hot plate for heating, and Fig. 4b is the case of using a convection oven for heating. In general, both cases have the same trend in deformation. When the master channel width increased from 25 μm to 75 μm, the resulting

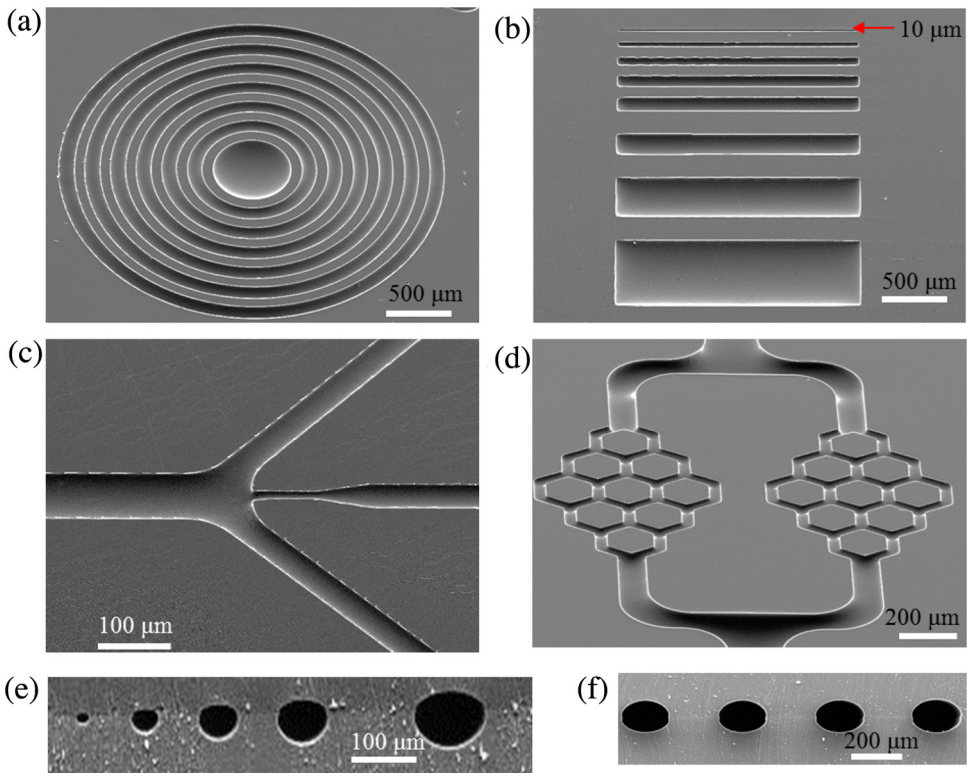

Fig. 7. SEM images showing the micro-features of semi-circular channels: (a) concentric microchannel rings, (b) multidepth microchannels with different widths, (c) micronozzle for a droplet focusing flow device or 3D focusing flow, (d) microcapillary network, and (e, f) cross-sections of circular microchannels.

channel depth increased linearly. However, for channels exceeding 150 μm in width, there were little or no differences in the replica depth for master channel. The difference between the trends in Fig. 4a and b, is due to the difference in the heating method. The heat flux direction created by the hot plate is from the bottom to the top, whereas the heat flux direction by the oven is from the surrounding to the center. Therefore, the curing time using the oven is much shorter than the hot plate. Also since the oven heating polymerizes the center later than the surrounding, this allows for the large diameter channels to expand more for the oven heating case. The small diameter channels in the oven heating behave similar to the hot plate method because the gelation state is already reached for the shallow channels. The polymerization of the PDMS starts right after it is mixed with the curing agent and the process is accelerated during the heating process. The deformation characteristic is equally attributable to the increasing gas pressure from the thermal air expansion and the surface tension of the polymer. Gas expansion into the polymer will cease when the pressure in the closed cavity is equal to the surface tension of the polymer. The replica channel cross-section is defined at this point.

Fig. 4c,d are zoomed in plots in the range of 25 μm–150 μm. The data above the reference line indicate overcut channels. The channels with a partial curing time of 31 min and 10 min showed overcut features that were clearly higher than the reference line, except for a channel width of 150 μm ('Case 1'). Using hot plate heating, at 32 min partial curing time, the height of the semi-circular replica was close to the reference line ('Case 2'). At this condition, the surface of partial cured PDMS will deform during air expansion and the deformation was stopped when the internal air pressure was equaled to surface tension of the partially cured polymer. The equivalence took place at the point when the replica channel got the nearly semi-circular features. After 34 min, surface tension of partially cured polymer was increased to near solid phase, and the air expansion could not so much push the volume into the polymer, so the channels were oval or elliptical shape ('Case 3'). Therefore, the partial curing time should be carefully selected to set the 'gel-stage' of polymer and obtain the desired channel geometry. Gel-stage duration depended on the curing temperature. At high temperature such as 70 °C, the gelation time is short (3–5 min). To increase the gelation duration, we can partially cure the polymer at lower temperatures, such as 60 °C or even 50 °C, then the gel-stage can be longer (10–15 min). Therefore, we can reduce the effect of the temperature fluctuations during the process and improve increase the process condition margins.

We conducted experiments to show the difference between separated channels (Fig. 2d) and connected channels (Fig. 2e) using a convection oven as shown in Fig. 5 . The connected channels, which are connected by two large channels (200 μm wide), are at the same pressure level throughout the whole volume. From the results in Fig. 5, we were able to see that with the same channel width, the replica depth in connected channels have smaller depth than the depth in separated channels (at 10 min, 11 min). We also can see the partial curing time variation in connected channels has not much effect on the replica channel depth when the channel width was less than 150 μm. We think that for separated channels, the deformation depth mainly depends on the volume of each channel, while for connected channels, it depends mainly on the overall shared volume of the connected channels. For the connected channels, initially the partially cured polymer in the larger channels will be pushed down more than the smaller channels due to the lower surface tension, this will then let the overall pressure be reduced as the overall volume is increased. The smaller channels will also simultaneously expand until the gas pressure gets equivalent to the surface tension of polymer. Compared to the separated case, the smaller channels in the connected case will be under lower pressure due to the pressure relief by the overall volume expansion of the larger channels. This can explain why the small channels

(<200 μm) in connected case have smaller deformation than the separated case.

In Fig. 6a and b, the air molded semi-circular channels are shown after the molding process (a), and after the master was detached (b). Self-alignment mechanism was used to create smooth channel profile shown in Fig. 6c. The separated channels with different width (10 μm and 100 μm) were successfully fabricated in Fig. 6d. In Fig. 6e, we fabricated a connected channel structure for mimicking the artificial blood capillary network.

Based on our method, we could fabricate both separated-channel device and connected-channel device. In Figs. 6 d and 7 a we show the separated-channel device examples. Angled channels and micro ring channel that can be fabricated by our method. In some previous research [14,22], where the channel formation uses external pressurized gas, the air cavities had to be connected with the pressure regulator to control the pressure inside the cavities, so it was only suitable to fabricate channels that are all connected to this pressurized gas. For separated channels, it was difficult to fabricate, or could not be produced by a single step.

To summarize the advantage of our method: (1) We have developed a low cost and straightforward way of fabricating PDMS circular channels with good repeatability (2) A single master can be used to create channels with variable depths by adjusting the fabrication conditions, such as the partial curing time. (3) A master with a single channel depth can be used to generate multidepth channel devices. (4) No alignment is needed. The first replica can be used as a master to create a second replica, and the self-alignment property facilitates sealing the two parts of the device together to form continuous and perfect channel profiles (5) The replica features do not depend on the cross-section of the master channel, and thus, many options are available for master fabrication other than photolithography, such as laser writing on metal sheets and micromilling.

4. Conclusions

We have invented a novel PDMS-molding method to rapidly fabricate circular microfluidic channels that has many advantages over previous approaches. We provided the control parameters to create oval or circular microchannels in the range of 25–150 μm diameter. Such a straightforward and low-cost fabrication process can create complex channel topologies for various biomedical applications. Fig. 7 shows preliminary results of fabricating concentric microchannel rings, micronozzles for droplet-generation devices or 3D focusing flow devices, and complex microcapillary networks. Especially, we believe that this technique's ability to produce circular channels can be used to mimic the human blood vessel. Our technique may also be used for other lab-on-a-chip applications that need circular channels, such as patch clamp devices to simultaneously measure multiple cells on a single chip.

Acknowledgements

This work was partly supported by the Basic Science Research Program through the National Research Foundation of Korea (NRF) funded by the Ministry of Science, ICT and Future Planning (NRF-2013R1A1A1012616) and under the framework of the international cooperation program managed by the National Research Foundation of Korea (NRF-2013K2A1B8054280). We thank Prof. Young-Hak Cho and his students for helping with clean room fabrication.

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Biographies

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