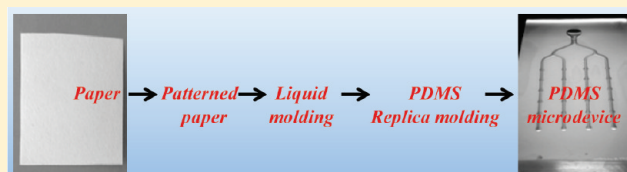


Patterned Paper as a Low-Cost, Flexible Substrate for Rapid Prototyping of PDMS Microdevices via “Liquid Molding”

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ABSTRACT: This report describes the use of patterned paper as a low-cost, flexible substrate for rapidly prototyping PDMS microdevices via “liquid molding”. The entire fabrication process consists simply of three steps: (1) fabrication of patterned paper in NC membrane by direct wax printing (or modified wax printing that we call “transfer wax printing”); (2) formation of liquid mold on wax-patterned NC membrane; (3) PDMS molding and curing on wax-patterned NC membrane anchored with liquid micropatterns. All these procedures can be finished within only 1.5 h without the use of a photomask, photoresist, UV lamp, etc. Through the use of wax-patterned NC membrane coupled with a liquid mold as a template, different PDMS microdevices such as microwells and microchannels have been fabricated to demonstrate the usefulness of the method for PDMS microfabrication. The height of microwells and microchannels can also be tailored flexibly by adjusting the liquid filling volume. This method for prototyping PDMS microdevices has some favorable merits including simple operation procedures, fast concept-to-device time, and low cost, indicating its potential for simple PDMS microdevice fabrication and applications.



PDMS (polydimethylsiloxane) is a widely used material in microfluidics because it is optically transparent, chemically stable, gas permeable, elastic, etc.^{1,2} It has accelerated microfluidics research greatly since its first introduction as a building material for microdevices in the 1990s by G. M. Whitesides and others.³ Currently, most PDMS microdevice fabrication still relies on photolithography methods, which usually include spin-coating, prebaking, exposure, postbaking, and developing steps. These procedures are tedious, time-consuming, and require some expensive facilities such as a clean room, spin coater, UV lamp, etc. Thus, a lot of different methods have been proposed to simplify the PDMS microdevice fabrication process.^{4–13} For example, Kaigala et al. demonstrated a simple procedure for fabrication of PDMS devices using a wax printer in a print, pour, and peel procedure.⁵ Liu et al. fabricated PDMS microdevices with complex structured microchannels by hydrogel micropatterning coupled with liquid molding.⁶ Abdelgawad et al. reported PDMS replica molding on masters formed by laser printing on flexible copper printed circuit board (PCB) substrates.¹¹ These innovative methods have made PDMS microdevice fabrication more accessible for use. However, most of these methods still suffer from high cost and complex operation processes^{6,11} or rough and shallow microchannels.⁵ Here, we report the use of patterned paper as the fabrication substrate to generate PDMS microdevices via “liquid molding”.

This work closely follows our previous work on paper-based microfluidics in which we reported wax printing and the use of nitrocellulose (NC) membrane to fabricate paper-based microfluidic devices.^{14,15} Paper-based microfluidics and related applications were first proposed by G. M. Whitesides et al. in 2007, in which paper was utilized as the substrate to pattern microstructures to generate complex microfluidic functions.¹⁶ As paper material

has many attractive features, such as versatility, low cost, light-weight, disposable, and biodegradable, paper-based microfluidics are quite suitable for the development of simple, inexpensive, and portable diagnostic devices for resource-limited regions. Recently, many studies have allowed the advancement of paper-based microfluidic technology.^{17–38} However, certain merits embedded in this new technology such as simplicity and low cost of fabrication have not been fully explored in previous studies.

In this study, paper substrate (NC membrane) was first patterned with wax microstructures by direct wax printing (or “transfer wax printing”) to form hydrophobic/hydrophilic intersecting regions. Then, liquid (glycerol solution or water) was anchored onto the hydrophilic regions by liquid filling or liquid dip coating. Next, PDMS microdevices were molded and cured on the liquid retained on the patterned paper as the template. This new PDMS fabrication process can be completed within only 1.5 h, is facile, inexpensive, and rapid compared to common photolithography methods, and represents a simple method for the rapid prototyping of PDMS microdevices.

EXPERIMENTAL SECTION

Materials. NC membrane was purchased from Whatman (0.45 μm pore size, Protran, GE). The wax printer was obtained from FUJIXEROX Phaser 8560DN (Japan). The baking equipment was an oven from Shanghai Permanent Science and Technology Company (PH-030A, China). Glycerol was purchased from Amresco (Solon, OH). Sylgard 184 PDMS base and curing

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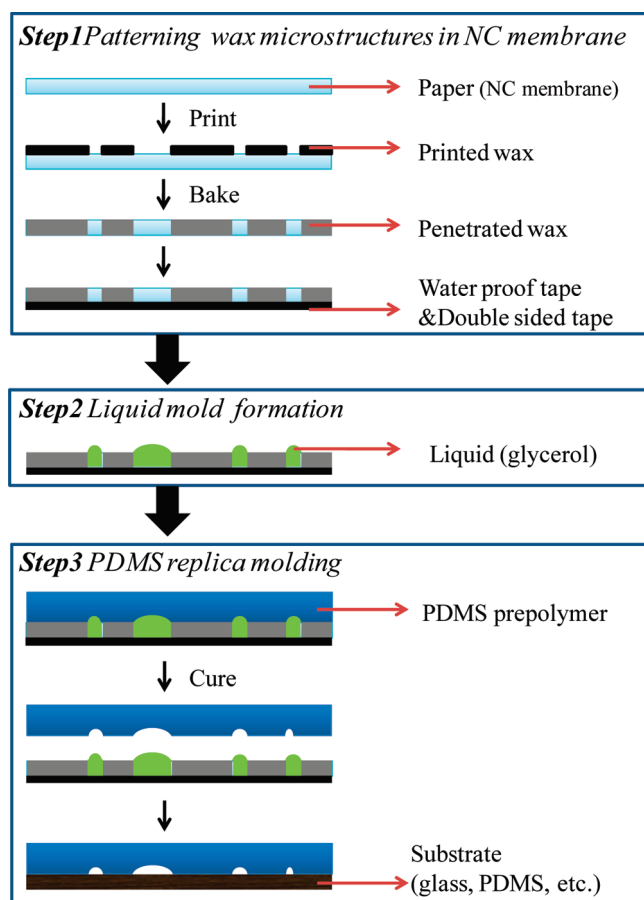


Figure 1. Cross illustration of the PDMS microdevice fabrication process using patterned paper as substrate via “liquid molding”.

agent were obtained from Dow Corning (Midland, MI). Deionized (DI) water was supplied by Wahaha Company (Hangzhou, Zhejiang, China).

Rapid Prototyping of PDMS Microdevices Using Patterned Paper as Substrate. The process to fabricate a PDMS slab with engraved microstructures using a piece of paper includes three main steps as shown in Figure 1.

Step 1: Patterning Wax Microstructures in NC Membrane. The method to generate patterned paper in NC membrane was based on our previously reported direct wax printing method.^{14,15} Briefly, it includes the following: (1) designing the pattern on a computer with mapping software; (2) printing the wax pattern onto the surface of the NC membrane (high resolution printing mode); (3) baking the wax-printed NC membrane to let the wax melt and penetrate through the membrane (125 °C in an oven for 5 min).

Here we also introduced a modified wax printing method called “transfer wax printing” as shown in Figure 2. The method was performed as follows: First, the wax microstructures were printed onto the surface of a transparency film (CG3300, 3M, St. Paul, MN), and it was placed on white paper for visualization in Figure 2). Then wax-patterned film (can be divided into small pieces) was covered with an untreated NC membrane with flexible sizes. The film and NC membrane were sandwiched between two glass sides with clamps and baked in the oven to let the wax melt and be absorbed by the paper substrate. The entire process to fabricate the wax-patterned NC membrane can be finished within 10 min.

Step 2: Liquid Mold Formation. Initially, wax patterning generated hydrophobic/hydrophilic intersecting regions on paper. Then waterproof tape was attached onto one side of the wax-patterned NC membrane and fixed onto a Petri dish (Greiner, Germany) smoothly with double-sided tape. Next, two methods were chosen to allow liquid adsorbed onto the hydrophilic regions of the wax-patterned NC membrane to form a liquid mold, processes called liquid filling and liquid dip coating. The former involves pipetting the desired volume of aqueous solution to fill the predefined hydrophilic regions on the wax-patterned NC membrane. The latter was conducted as follows: The wax-patterned NC membrane was first dipped into an aqueous solution (dwell in the solution for about 3 min to ensure that the hydrophilic regions were totally wet) to allow the hydrophilic NC membrane to soak in the liquid. Then it was pulled out of the solution slowly and evenly. During this process, the aqueous solution is retained on the hydrophilic NC membrane, which detaches from the hydrophobic wax patterned surface. The interplay of capillary force and shear stress results in the formation of liquid patterns on the hydrophilic area.⁶ The total time needed for this liquid mold formation process is less than 10 min.

PDMS Replica Molding. After the liquid mold was formed evenly on the wax-patterned NC membrane, well-mixed PDMS prepolymer (10:1 mass ratio) was poured onto the substrate. PDMS can be cured in the oven at 80 °C for 1 h and then peeled off the substrate.

RESULTS AND DISCUSSION

Choice of Paper Substrate. In this work, NC membrane was selected as substrate instead of common filter paper. NC membrane has different pore sizes, and the average pore size of the NC membrane used in this work is only 0.45 μm , which is much smaller than the averaged pore size of common filter paper (ranging from several micrometers to hundreds of micrometers). The smaller pore size conveys several advantages: (1) The wax lateral spreading process in the NC membrane during baking can be controlled more precisely and more reproducibly than that on filter paper, and the resulting dimensions of PDMS microdevices molded on it are also more reproducible; (2) the NC membrane surface is relatively smooth, and the surface of the PDMS cured on it is smoother than that cured on filter paper; (3) the fiber is denser in NC membrane than in common paper, which allows the exclusion of excess liquid in the liquid mold formation process when large amount of liquids contact its surface; (4) the large pores in common filter paper can accumulate a large amount of gas, which will generate many gas bubbles in the PDMS during curing process.

Fabrication of Wax-Patterned NC Membrane by Wax Printing. The patterned paper in the NC membrane can be prepared by the direct wax printing method or by a modified wax printing method called “transfer wax printing” (see Figure 2), in which the wax pattern is first printed onto a transparency film and then transferred into the NC membrane by sandwich baking. Transfer wax printing is quite easy to perform and has several favorable features: (1) Transfer wax printing will eliminate the NC membrane jamming during the direct wax printing process (static electricity may sometimes cause a paper jam during NC membrane direct wax printing); (2) the wax-printed transparency film can be divided into smaller pieces when the NC membrane is too small for direct wax printing; (3) this method can form wax microstructures on paper substrates with irregular shapes, such as

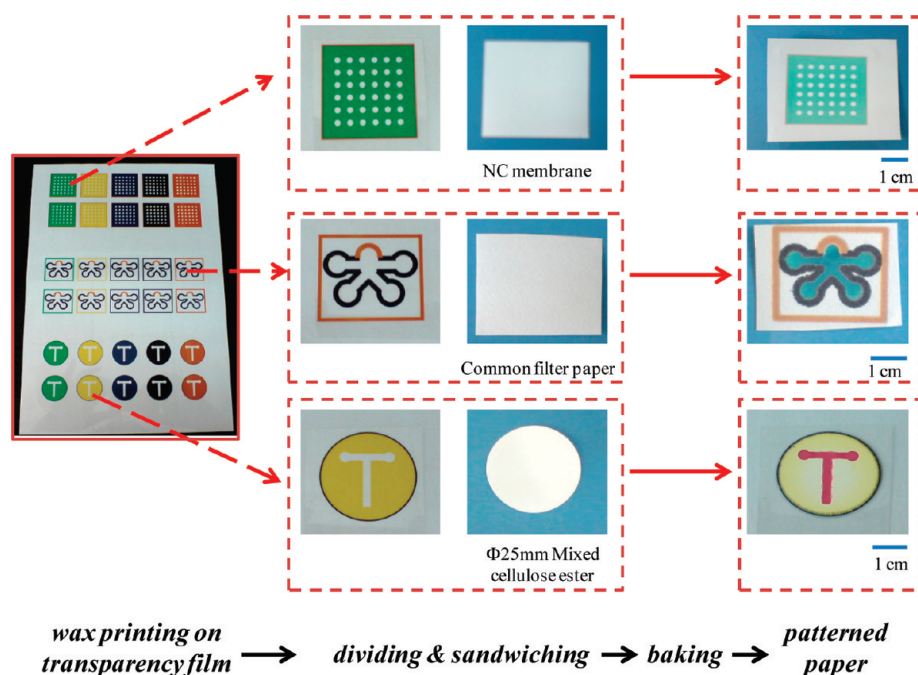


Figure 2. Demonstration of transfer wax printing on different paper substrates with various designs.

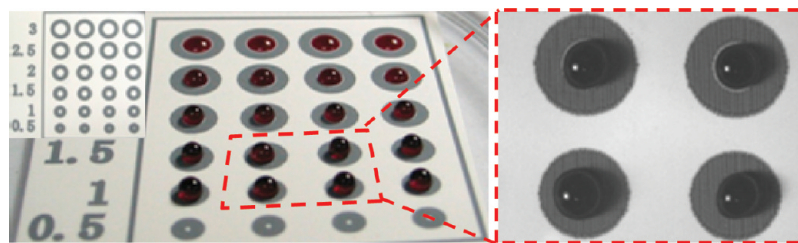


Figure 3. The compatibility between glycerol solution and printed wax. The glycerol solution does not dissolve the printed wax as shown.

round, or on other paper substrates such as mixed cellulose ester membrane; (4) the wax-printed transparency film is convenient to store and transport, so this method can be used in places where a wax printer is not available.

The wax printed onto the paper substrate will spread during the baking step, and this process can be characterized by Washburn's equation which describes capillary flow in porous materials:³⁹

$$L = \sqrt{\gamma D t / 4\eta} \quad (1)$$

where t is the time for a liquid of viscosity η and surface tension γ to flow a distance L into a porous material with an average pore diameter D . From eq 1, we can see that the lateral spread of wax is proportional to the square root of time when all other parameters are constant. Our previous experiments demonstrated that the resulting channel width after baking (both front side and back side) was in a good linear relationship with printing channel width on the NC membrane. Besides, the wax ink (a mixture of hydrophobic carbamates, hydrocarbons, and dyes) used in the wax printer did not melt at 80 °C in the oven (this has been validated in our previous experiment^{14,15}), so wax would not melt during the PDMS curing process (60–80 °C).

Liquid Mold Formation on Wax-Patterned NC Membrane. During liquid mold formation, glycerol solution or water was anchored onto the hydrophilic regions on the wax-patterned NC membrane to form a liquid mold. Then well-mixed PDMS

prepolymer was poured onto liquid micropatterns to be cured. Using glycerol or water as a liquid mold is based on the following considerations: (1) PDMS prepolymer is a nonpolar liquid, so it will be immiscible with glycerol or water; (2) glycerol or water will not dissolve the wax pattern in paper (see Figure 3), which was demonstrated as follows: 3 μ L of red dye solution (10 mg/mL amaranth in 50/50 glycerol–water (v/v) solution) was pipetted into each circular ring (0.5 mm to 3 mm, in increments of 0.5 mm; the hydrophobic wax was 1 mm width). Under normal conditions for 1 h, the glycerol solution did not dissolve the wax ring; (3) glycerol and water are inexpensive, nontoxic, and environmental friendly. Besides, because of the relatively high viscosity of glycerol (20 °C, 1499 mPa·s), glycerol was modified to be 40% water solution (v/v) when it was used to achieve a more suitable viscosity and evaporation rate for ease of operation.

Fabrication of PDMS Microdevices Using Wax-Patterned NC Membrane as Substrate. With the established method presented here, microwells and microchannels structures were fabricated to demonstrate its usefulness in PDMS microdevice prototyping. Microwells with different sizes and shapes were first fabricated. When the pattern dimension is relatively large (larger than 1 mm) and the resulting height is higher, the liquid filling method can be used to generate PDMS microwells. Figure 4A shows the process to fabricate 6 × 6 2 mm diameter PDMS microwell arrays, which are quite simple to prepare. Especially,

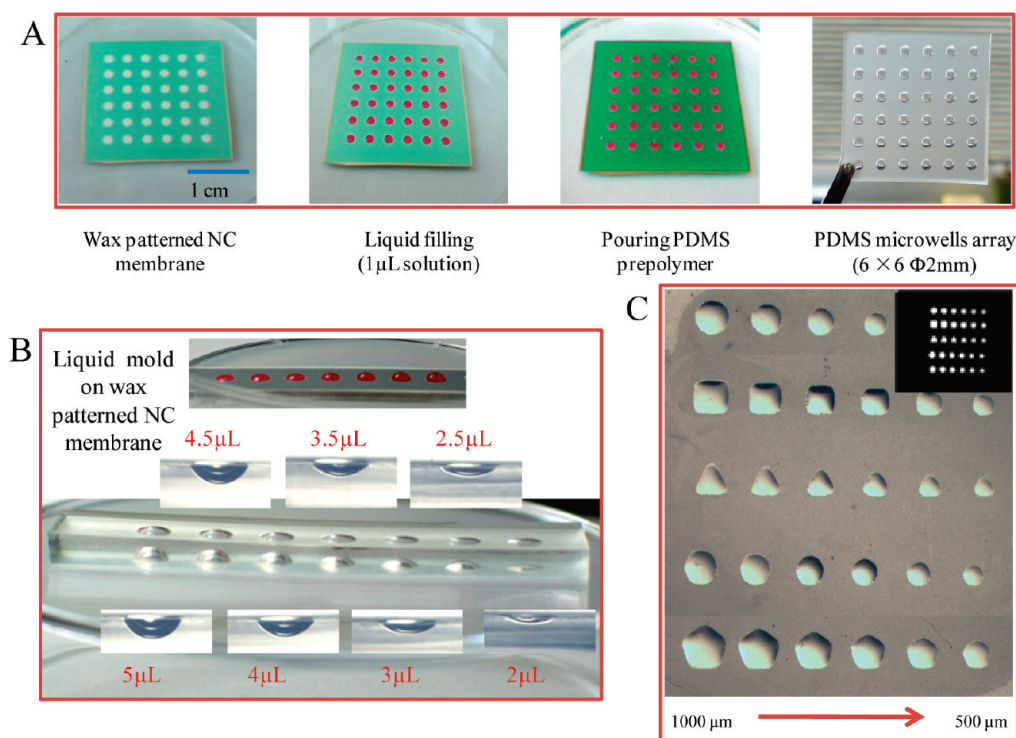


Figure 4. Fabrication of PDMS microwells using wax-patterned NC membrane as substrate (glycerol solution was mixed with amaranth dye for visualization). A, PDMS microwell array fabrication process. B, PDMS microwells of the same base size (3 mm) but with different heights. C, PDMS microwells of different diameters and different shapes, such as round, square, triangle, pentagon, hexagon, etc.

different volumes of liquid can be flexibly pipetted to generate adjustable heights with the same base dimension. For example, in Figure 4B, different volumes of 40% glycerol/water solution were pipetted into seven 3 mm diameter round hydrophilic patterns on the wax-patterned NC membrane, and PDMS was molded on it. This yields concave microwells of the same base dimension but with different heights, which may be useful in PDMS lens fabrication research.⁴⁰ For those pattern dimensions smaller than 1 mm, liquid dip coating can be used to form a liquid mold on wax-patterned NC membrane to fabricate PDMS devices with different shapes (see Figure 4C), such as round, square, triangle, pentagon, hexagon, etc.

We also demonstrated the use of wax-patterned NC membrane in microchannel fabrication. Figure 5A shows a PDMS microchip made from wax-patterned NC membrane. The cross-sectional view indicates that microchannels fabricated in this manner have a round profile (aqueous solution normally tends to become spherical to achieve the smallest surface area), which could assist in the building of pneumatic valves on a microfluidic chip. The relationship between the resulting width of the PDMS microchannels and its designed width was also evaluated. Figure 5B exhibits a good linear relationship between printing width and resulting width. Note that the resulting width is a bit larger than the designed width because the weight of the PDMS can compress the liquid mold.

The height of the liquid pattern formed on the wax-patterned NC membrane can also be flexibly tailored by adjusting liquid filling volume, as demonstrated in Figure 6A. First, 500 μm microchannels connected with 3 mm diameter round reservoirs were wax-patterned onto NC membrane. Then the wax-patterned NC membrane was dip-coated with water to form a thin layer water mold. Different volumes of water were pipetted into the

round reservoirs. The round reservoirs act like an impounding region that directs the liquid height balance between the reservoir and its connecting microchannel. Finally, the PDMS prepolymer was poured and cured on the water mold. As shown in Figure 6A, from left to right, the resulting height/width ratios were 0.119, 0.169, 0.212, 0.226, and 0.260, respectively. Compared with the reported data (0.052–0.063 in ref 4 and 0.097 in ref 6), the resulting height/width ratio shows about a 3–4 fold improvement over that of other liquid mold-based PDMS fabrication methods. This improvement could be very useful in some applications such as droplet generation, as droplets are difficult to generate in low height/width ratio microchannels. As shown in Figure 6B, a PDMS microchip was molded for droplet generation by tailoring the height of liquid mold, which demonstrated the usefulness of tailoring the height of microchannels.

During PDMS baking process, the liquid pattern was replicated into the PDMS slab. The rest of the PDMS can be solidified on the wax-patterned NC membrane surface; thus, the microstructures of the NC membrane would also be replicated. These microstructures will not interrupt its sealing with other substrates (PDMS, glass, etc.) by plasma oxidation. However, there is some difficulty in reversible sealing with these substrates, which can be alleviated by the following method: An even, uncured PDMS layer around 1 μm thick was formed by spin-coating or wiping methods. The PDMS chip was then attached onto the uncured PDMS. The uncured PDMS fills the microstructures in the PDMS surface replicated from NC membrane. After curing, the reversible sealing with other substrates such as PDMS, glass, and polystyrene is possible.

Advantages of PDMS Fabrication Using Patterned Paper as Substrate via “Liquid Molding”. The proposed method in this work to fabricate PDMS microdevices has several favorable

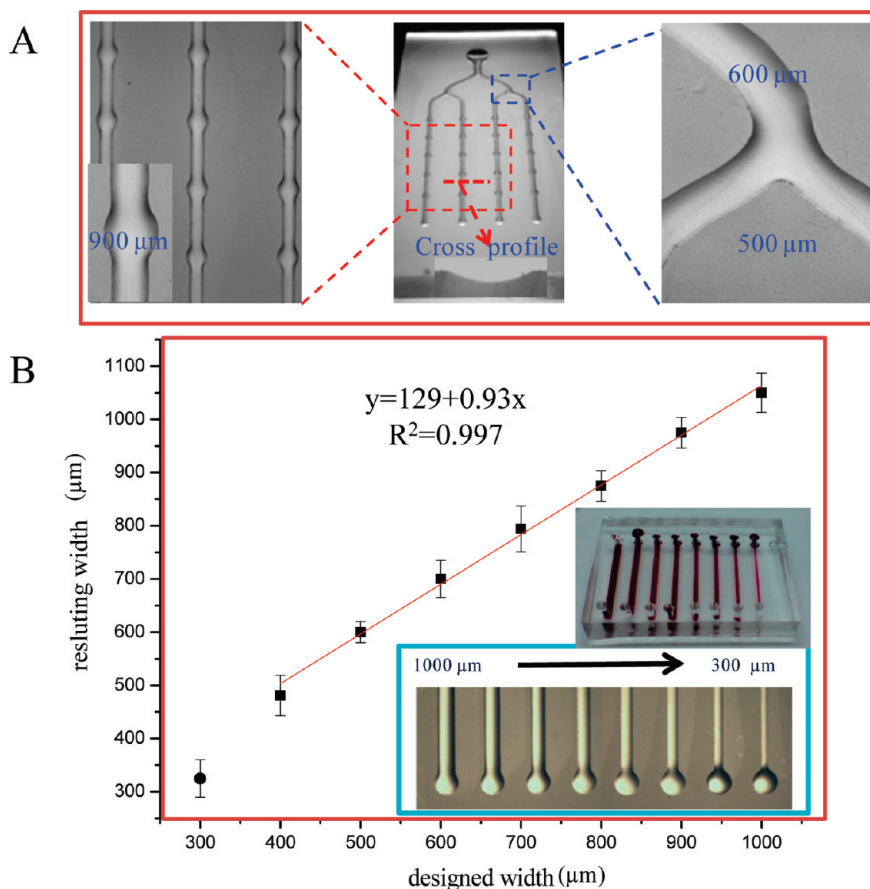


Figure 5. Fabrication of PDMS microchannels using wax-patterned NC membrane as substrate. A, PDMS microfluidic chip fabricated on wax-patterned NC membrane and its enlarged view. B, Characterization of PDMS channel width molded from a liquid mold anchored on wax-patterned NC membrane ($n = 4$).

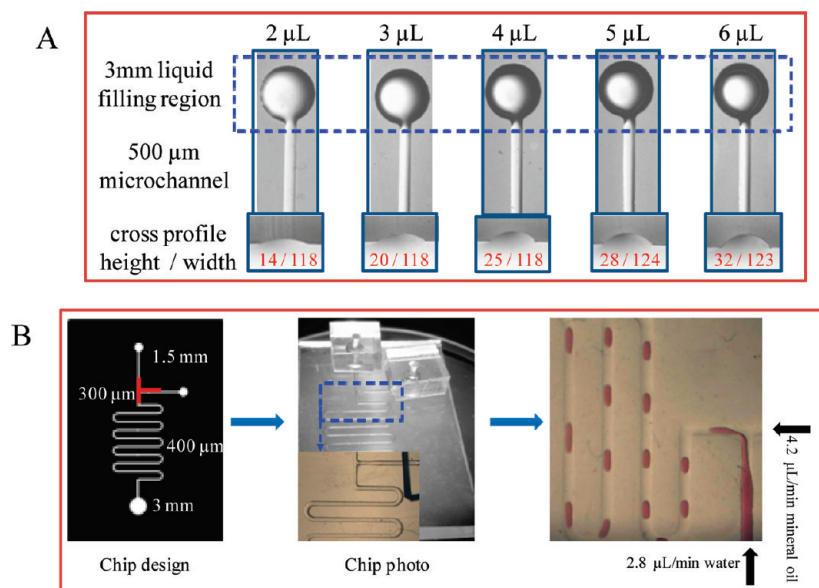


Figure 6. Tailoring the height of microchannels molded from a liquid mold anchored on wax-patterned NC membrane. A, PDMS channels with different height/width ratios (the numbers in the cross profile were measured in Imagepro software (Media Cybernetics, Bethesda, MD)). B, A droplet generator chip molded on wax-patterned NC membrane. (The microchannel width was designed to be 400 μm except for the cross parts labeled with red (300 μm)).

characteristics compared with those of commonly used photolithography methods: (1) Fast and easy-to-perform fabrication

process: The entire three-step process can be finished within 1.5 h. Note that the time needed to fulfill the first two steps takes

only about 20 min to get the liquid mold ready for replica molding while photolithography methods usually take several hours to a day to finish spin-coating, prebaking, exposure, postbaking, developing, and hardbaking steps. The improvement in production speed can ensure the flexible design and fabrication of different designs in a short period of time. (2) Low cost: The entire fabrication process can be performed without a photoresist, UV lamp, and photomask, which are indispensable in photolithography methods. (3) Environmentally friendly: The entire process obviates the need for any toxic organic solvents. Compared with some other simple, low-cost PDMS fabrication methods, the main advantage in our method lies in its flexibility and ease to tailor microchannel heights (demonstrated in Figure 6), which has not been previously reported.

Despite its advantages, this fabrication method has a potential limitation in resolution (the smallest PDMS microchannel width achievable with this method is around 300 μm) due to the confinement of wax printer resolution and wax spreading during baking process. However, this resolution is not entirely satisfactory. Considering the advantages of easy and rapid prototyping, this fabrication method will be especially suited for microdevices with simple channel designs and low resolution requirements.⁴

CONCLUSION

This report presents a simple method for the rapid prototyping of PDMS microdevices using patterned paper as substrate. Microwells and microchannels with a flexibly tailored aspect ratio were fabricated with this method. The method not only provides a complement to the current PDMS fabrication methods but also expands the utility of paper-based microfluidic devices. We believe it will be very useful for a wide range of microfluidic applications based on PDMS microdevices.

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