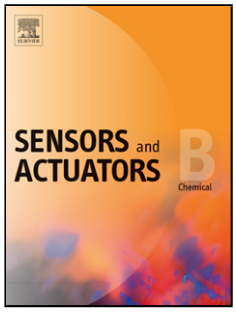

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Epoxy resin mold and PDMS microfluidic devices through photopolymer flexographic printing plate

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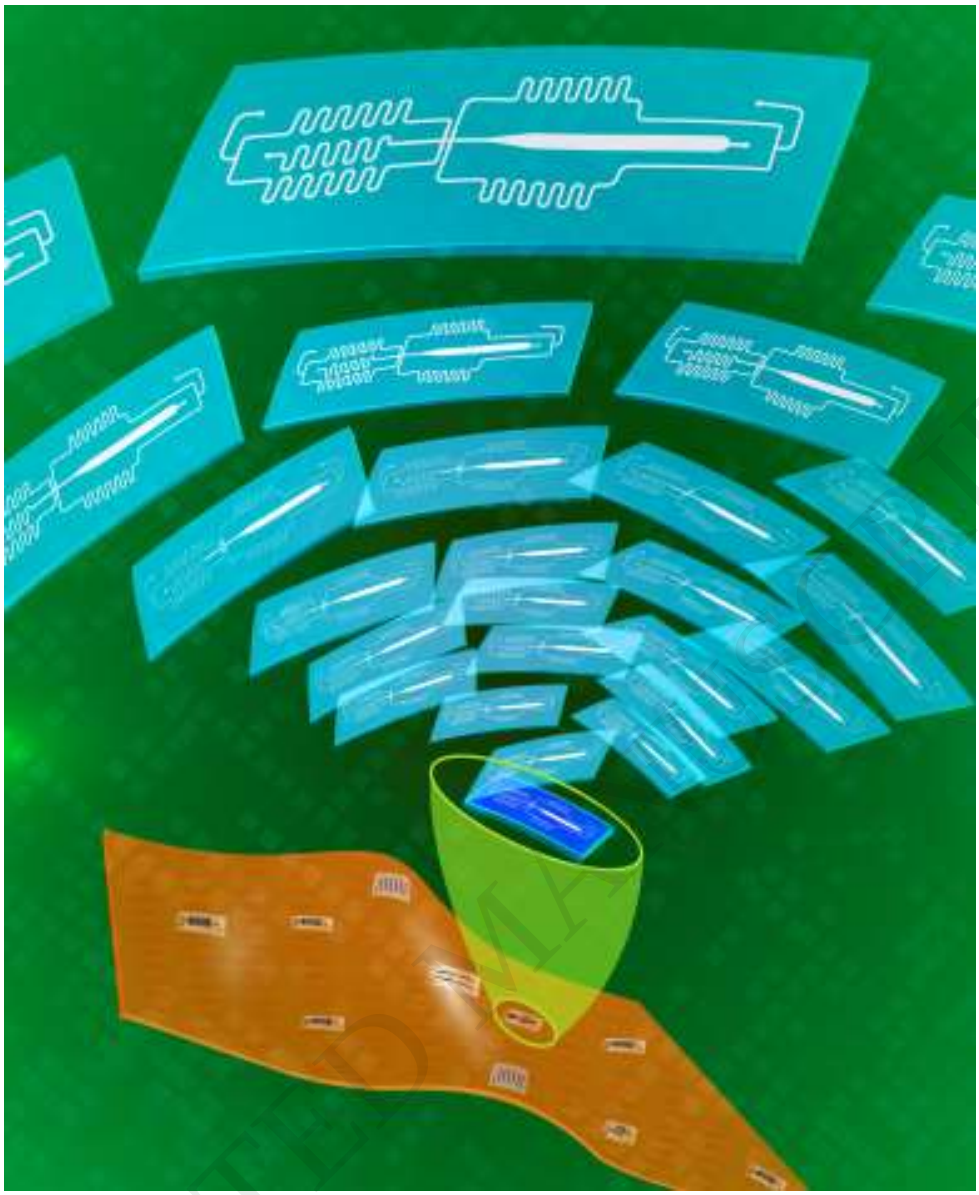
GRAPHICAL ABSTRACT



PDMS (polydimethylsiloxane) microdevice fabrication through photopolymer mold facilitates the scaling-up of microfluidic devices.

Highlights

- High fidelity replication from photopolymer mold to epoxy resin was demonstrated.
- Reusable epoxy resin molds containing structures with different topologies were fabricated.
- Epoxy resin mold was assessed for fabricating PDMS microfluidic devices.
- Microdevices for microdroplets generation were tested.

Abstract

Photopolymer flexographic printing plate is a new photopolymeric material used for microdevices fabrication. This work demonstrates that a photopolymer flexographic master mold can be used for the fabrication of PDMS (polydimethylsiloxane) microdevices by a multi-step manufacturing process. The methodology entails three main fabrication steps: (1) a photopolymer flexographic printing plate mold (FMold) is generated by UV exposure through a transparent film, (2) an epoxy resin mold (ERmold) is fabricated by transferring the features of the photopolymer mold and (3) a PDMS microdevice is manufactured from the epoxy resin mold. The characterization of the manufactured PDMS microdevices was performed using scanning electron microscopy (SEM) and profilometry. Results showed high accuracy in the replication of the profiles. To show the feasibility of the fabrication process a microdevice for microdroplet generation was designed, manufactured and tested. Hence, the manufacturing process described in this work provides an easy, robust, and low-cost strategy that facilitates the scaling-up of microfluidic devices without requiring any sophisticated equipment.

Keywords: epoxy resin, microfluidics, microdroplets generation, PDMS microdevice, photopolymer flexographic.

1. Introduction

The flexography technique is a widely spread printing process which has been used on several substrates like food packages, newspapers, inserts, gift wrap, wallpaper, catalogs, among others[1,2]. In last years, this technique has been employed to produce structures in relief for paper fluidics[3]; and electronic devices like electrodes[4], capacitors[5], sensors[6] and transistors[7]. The

processes involved the use of conventional graphic printing, functional inks and substrates such as glass, paper, PET film, photopolymer flexographic printing plate among others[3,8]. As a result, a variety of patterns with micrometer-scale resolution and uniform thickness can be obtained. The improvements achieved with this technique have led to high performance and complex design devices with a wide variety of applications in different fields [1,9].

The methodology of using flexographic technology for manufacturing microfluidic devices has been recently developed and characterized previously by the authors [10]. It has many advantages over non-traditional methods like CO_2 laser ablation [11], hot embossing [12], stainless steel stamp[13], toner [14], liquid molding [15,16], building blocks[17], laser ablation[18], laser swelling[19], semi-contact writing[20], 3D printing[21,22] and WAX mold[23]. The main advantages of flexographic molds are: (i) short time mold fabrication, (ii) the delamination phenomenon due the structures of the photopolymer mold form a single element is absent, (iii) low development costs, (iv) multiple molds with a variety of dimensions (height, width, length) could be manufactured, (v) high-throughput replication, (vi) minimum channel width of 10 µm and (vii) low surface roughness of the structures [10].

In the previous works, despite all the mentioned advantages of the Fmolds, its service life was limited to the detachment of the SiO_2 layer caused by the replication process. Additionally, for the manufacture of Fmolds is necessary a chemical vapor deposition (CVD) equipment. In this work, an alternative methodology to fabricate PDMS microdevices is developed. It combines the Fmold technique with the ERmold method. The new methodology offers durable molds and does not require SiO_2 deposition as the previously reported methodology, and therefore, its durability is much higher. Additionally, the

ERmold fabrication methodology does not require CVD equipment, thus this mold can be used widely in any laboratory without purchasing expensive equipments. The resulting molds and PDMS replicas were characterized by Scanning Electron Microscopy (SEM) and profilometry techniques. Finally, to prove the usefulness of the methodology, microfluidic devices for microdroplets generation were fabricated and tested.

2. Material and methods

2.1 PDMS microfluidic device fabrication

The PDMS microfluidic device fabrication process consists of three main steps: (1) fabrication of a photopolymer flexographic master mold (Fmold), (2) manufacture of a reusable epoxy resin male mold (ERmold) using the Fmold by replica molding and (3) transfer of the ERmold features to PDMS. This process is schematically summarized in Fig. 1.

Photopolymer flexographic master mold (Fmold): The photopolymer Flexcel NX and Thermal Imaging Layer (TIL) used in the fabrication of the molds were supplied by Eastman Kodak[24]. The fabrication steps of the Fmold have been described in a previous work [10]. Briefly, microchannels network was designed with Layout editor software[25]; this design was transferred to the TIL with an infrared laser source of 2400 ppi and subsequently, it was laminated onto the unexposed photopolymer plate. In the next step, the photopolymer plate was exposed to UVA light at 0.45 J on the reverse side and then the front side was exposed to UVA light at 19 J for 360 s, after the TIL was removed. Different time periods of UVA exposure on the reverse side were used to control the height of the microchannels. Later, the photopolymer plate was washed with solvent PROSOL N-1 (supplied by Eastman Kodak) at 360 mm/min and it was dried in an oven during 30 min at 50 ºC. Finally, the photopolymer plate was exposed to UVC

light at 10 J for 17 min and UVA light at 4 J for 2 min on the front side. This mold was coded as Fmold. Before its use, the Fmold was placed in an oven at 100 ºC for 12 hours and then it was treated in a vacuum chamber for 1 hour at 25 ºC, followed by a cleaning process in 70 % ethanol solution in an ultrasonic bath for 7 min, dried at 40 °C for 10 min and cleaned by a nitrogen stream.

Epoxy resin mold (ERmold): A commercially available epoxy resin and curing agent (Cristal-Tack, Novarchem - Argentina) were mixed by hand-stirring for 3 min in a 2:1 weight ratio and ultrasonically treated using a bath-type sonicator (TESTLAB Ultrasonic Cleaner) for 7 min to remove air bubbles. Then, the mixture was poured onto the Fmold and cured at room temperature for 72 hours. After curing, the epoxy resin mold was peeled off from the Fmold to form the male mold (Fig. 1d), this mold is referred as ERmold.

PDMS microdevice: Briefly, the PDMS was mixed with curing agent in a 10:1 weight ratio (Sylgard 184 Silicone Elastomer Kit), as previously described by Peñaherrera et al.[26] Then, the mixture was placed under vacuum for 30 min to remove air bubbles, poured onto the ERmold and cured in an oven at 40 °C overnight (Fig. 1f, 1g). After curing, the PDMS replica was peeled off from the mold and holes for inlets and outlets of the channels were punched using a 1 mm diameter biopsy puncher (Integra Miltex®Ted Pella, Inc). Finally, the replica was irreversibly bonded to a glass wafer after exposure to a high frequency generator (BD-10AS, Chicago) for 120 s.

2.2 Characterization

The morphological characterization of the Fmolds, ERmolds and PDMS replicas were carried out using a Field Emission Gun Scanning Electron Microscope (TESCAN FEG SEM MIRA3). In order to avoid damage of samples, the SEM measurements were carried out at voltages of 7 kV. Previously, the molds were metalized with an approximately 20 nm gold layer. Quantitative measurements were made with the MIRA

TC software version 4.2.24.0. Profilometry measurements were performed using Dektak XT profilometer from Bruker, and the analysis was carried out using the Vision 64 software. Linear scans were performed with a 25 µm radius tip, at a scan speed of 10 µm/s and a sampling rate of 0.01 Hz/mm. Before characterization, the molds (Fmold, ERmold) and PDMS replicas were blown with nitrogen gas to remove dust, then were ultrasonically cleaned in ethanol (70 % v/v) for 10 min (this step was repeated 5 times) followed by a drying step in an oven at 40 °C for 1 hour.

2.3 Application: micron-sized droplets generation

In order to assess the methodology, a flow-focusing single emulsion droplet-generator microfluidic device was designed, fabricated and employed for generation of monodisperse micron-sized droplets. The design is showed in the Supporting Information Fig. S6, it contains two inlet and one outlet channels. The narrow channel that forms the droplet generation nozzle was drawn as 70 µm width. The inner phase, aniline blue solution 2 % (w/v) solution was pumped (AcTIVA Infusion ADOX A22) at a constant flow rate of 0.03 mL h^{-1}, the continuous phase mineral oil (Sigma-Aldrich) with surfactant SPAN 80 (Sigma-Aldrich) (10 % w/v) was pumped at a flow rate of 0.01 mL h^{-1}.

Microdroplets imaging: The Olympus BX40 microscope with 5X and 10X objectives and a Canon T3-I Rebel digital camera attached to the microscope were used to capture the images of the formation of microdroplets. The images were created from a stack of multiple microscope acquisitions over a large surface area of the device. To analyze the size distribution of the generated microdroplets, the area of 100 subsequently generated microdroplets was measured using image processing Fiji Software and the average diameter and standard deviation were reported[27] .

3. Results and Discussion

3.1. Feature replication

Fig. 2 shows Scanning Electron Microscopy (SEM) images of PDMS replicas with different topologies, structures and dimensions. Desired features: circular, curves, serpentine, square, diagonal and linear and no lineal channels, with width dimensions ranging from 80 µm to 2200 µm were obtained. The successful reproduction of microfluidic topologies on the Fmold and the ERmold are shown in the Supporting Information Fig. S1 and Fig. S2, respectively. The results demonstrate that the developed methodology allows obtaining PDMS replicas through photopolymer flexographic printing plate.

3.2. Fmold/ERmold replication fidelity and Fmold durability

The replication fidelity of Fmold to the ERmold was evaluated by comparing the feature dimensions of the molds. For this purpose, a flow-focusing single emulsion droplet generator microfluidic device was designed. Profilometry measurements and SEM images were used to determine depth, height, and width dimensions. Furthermore, the stability and durability of the Fmold was evaluated by comparing three ERmold replicas obtained from the same Fmold. Fig. 3 shows the SEM images of three sections of interest in the Fmold and the first, second and third ERmold replicas. The selected regions are composed of a T-junction (section A), curved (section B), and linear intersection (section C) channel segments.

Table 1 lists the height, depth and width dimensions of the Fmold in regard to the ERmold replicas in the A, B and C sections. The width dimension of the ERmold obtained by SEM shows a slight shrinkage (< 10 %) compared to the Fmold. Further, the profilometry analysis shows that height and depth dimensions in both ERmold replicas and Fmold are very close (144 µm), a tiny shrinkage (< 3 %) is observed. The results proved that Fmold is a reusable mold that allows obtaining successfully

ERmolds. The height measurements recorded by profilometry are shown in the Supporting Information Fig. S3.

Table 1. Fmold/ERmold replication fidelity. Measured microchannel depth, width and height after each molding step.

Section	Design by L-edit software	Fmold	ERmold-1	ERmold-2	ERmold-3
Height / depth (µm)[a]					
-	-	144 ± 1.6	141 ± 1.6	143 ± 2.2	143 ± 1.6
Width (µm)[b]					
A	500	518 ± 2.5	505 ± 0.5	503 ± 3.4	507 ± 7.9
B	400	411 ± 0.5	395 ± 2.1	395 ± 3.1	392 ± 1.1
C	70	83 ± 0.5	75 ± 4.2	78 ± 2.4	79 ± 4.2

a. Height and depth measurements were determined by profilometry technique (n=6)

b. Width measurements were determined by SEM technique (n=3)

Concerning to the comparison between the channel width of the Fmold and the designed by L-edit software, some differences are observed. The channel width was drawn as 500 µm, 400 µm and 70 µm in section A, B and C, respectively. However, the channel widths of the Fmold in these sections is 18, 11 and 13 µm higher than the designed using the software. This can be due to the process of imaging the microstructures on the TIL, since the focused laser has a diameter of 10.5 µm. Consequently, the final channel width measurements obtained on the TIL are higher than the drawn by the software.

3.3. ERmold/PDMS replication fidelity and ERmold durability

Table 2 shows SEM and profilometry measurements of channel dimensions of Fmold, ERmold and first, fifth and the tenth PDMS replicas of a flow-focusing double emulsion droplet generator microfluidic device. It is shown that channel dimensions (width, height, depth) vary less than 10 % for the entire range, demonstrating that PDMS can be replicated with high fidelity. Height measurements recorded by profilometry and SEM

images of the Fmold and ERmold have been shown in the Supporting Information Fig. S4 and Fig. S5, respectively. Fig. 4 shows the SEM images of three sections in first, fifth and the tenth PDMS replicas. The microchannel geometry did not change over the multiple molding steps. From these results, it was demonstrated the feature transfer precision from the ERmold to the PDMS. In addition, the stability and durability of the ERmold were determined.

Table 2. ERmold/PDMS replication fidelity. Measured microchannel depth, width and height after each molding step.

Section	Design by L-edit software	Fmold	ERmold	PDMS-1	PDMS-5	PDMS-10
Height / depth (µm)[a]						
-	-	227 ± 1.6	227 ± 2.3	246 ± 1.9	230 ± 2.6	229 ± 3.6
Width (µm)[b]						
A	2100	2118 ± 4.6	2112 ± 6.0	2085 ± 2.3	2098 ± 5.6	2104 ± 9.8
C	70	85 ± 1.2	86 ± 0.6	83 ± 0.6	86 ± 2.5	85 ± 2.3

a. Height and depth measurements were determined by profilometry technique (n=6)

b. Width measurements were determined by SEM technique (n=3)

The results evidence that the ERmold can be employed multiple times to obtain PDMS replicas. By other hand, the precision is comparable to typical photolithographic tolerances (10 %) [28], making of the proposed methodology an ideal candidate for the fabrication of microfluidic devices designed for different purposes, and with microchannel dimensions relevant to a wide range of microfluidic applications. Besides, the re-usability and durability of the ERmold was demonstrated; the selected commercial epoxy resin generates rigid masters that can be used many times to get PDMS replicas with minimal changes in feature dimensions.

3.4 PDMS device for microdroplet generation

Microfluidic devices for microdroplet generation are of great significance in many chemical, biomedical and industrial applications[29–32]. Here, we fabricated a PDMS

device using the proposed methodology for microdroplet generation. The design of the microdevice is shown in the Supporting Information Fig. S6. A representative microscope image of the microdroplet formation is shown in Fig. 5a. The histogram of the droplet sizes is show in the Fig. 5b, the statistical analysis shows sizes ranging from 47.4 μm to 63.1 μm with a standard deviation lower than 4 %, indicating a high level of droplet monodispersity. The experimental data show that this microfluidic device is capable of generating controlled size microdroplet. The Supplementary Video S1 shows the microdroplet generation.

Based on the results listed above, the described methodology offers advantages such as: (1) non-cleanroom facilities, (2) low cost, (3) high durability of molds, (4) the possibility of having a high mass production of epoxy resin mold and PDMS replicas with high precise replication, (5) ERmold as a monolithic mold, hence delamination between the features and the substrate is not a limiting factor, (6) the fabrication of heterogeneous structures having different dimensions allow the manufacture of microdevices that can be used in many applications, being easy to implement as an alternative to conventional soft lithographic approaches. In addition, ERMold is more durable than FMold and does not require deposition of SiO_2, thus the fabrication method can be achieved at institutions where plasma enhanced chemical vapor deposition (PECVD) is not available. All these attributes turn this methodology into a promising tool to manufacture microfluidic devices.

4. Conclusions

For a variety of research areas, a critical aspect is selecting a methodology for building molds to fabricate PDMS microdevices. We proved that the ERMold could be considered a good option to obtain PDMS replicas with desired topologies. Remarkably, the methodological approach proposed here allows manufacturing reusable molds with high capability of replication and containing structures ranging from microns to

millimeters. The functionality and versatility of the methodology has been successfully demonstrated fabricating various microfluidic topologies and employed on the fabrication of microdroplets generator device with a flow-focusing droplet generation model.

Supporting Information

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Prof Luis Cumbal. Prof Cumbal received his PhD from Lehigh University, USA. He works in the development of novel materials to clean up contaminated soils and water. He is the co-inventor of the LayneRT adsorbent used for the selective arsenic removal from water. He has published more than 60 articles in peer-reviewed journal and participated as a speaker in about 100 national and international scientific events. Dr. Cumbal is Associate Editor of the Journal Groundwater for Sustainable Development and peer-reviewer of several scientific journals. Currently, he is the head of the Center for Nanoscience and Nanotechnology and professor of the Graduate Program in Nanotechnology and of the Department of Life Sciences at the Universidad de las Fuerzas Armadas, Quito, Ecuador.

Figure captions

Fig. 1. PDMS microdevice fabrication. (a) Photopolymeric flexographic master mold (Fmold). (b,c) The epoxy resin is cast on the Fmold and cured at 25 °C (d,e) After 72 hours the ERmold is peeled off to form the male mold. (f,g) The PDMS is cast on the ERmold and cured at 40 °C overnight. (h) The PDMS replica is peeled off. (i) The fluidic connection ports were punched and then the replica was irreversibly bonded to a glass wafer by plasma exposure. The PDMS replica corresponds with a design of microdevice to microdroplet generation.

Fig. 2. SEM images of PDMS microfluidic topologies molded from ERmold. Microfluidic topologies reproduction ranging from lineal and curved patterns: A: circular, B: serpentine, C: diagonal, D: curve, E: square, F: linear intersection. (scale bar: 100 µm)

Fig. 3. Feature fidelity precision from Fmold to ERmold. (a) SEM images of selected sections of the fabricated Fmold (blue row), ERmold-1 (green row), ERmold-2 (green row), and ERmold-3 (green row). Section A: T-junction, section B: curved, Section C: linear intersection channel segments. (b) Flow-focusing double emulsion droplet generator microfluidic device design. Fmold fabrication conditions: First step - UVA exposure time on reverse side = 70 s, UVA exposure time on front side = 360 s, second step - UVA front exposure = 2 min, UVC front exposure = 17 min. (Scale bar: 200 µm)

Fig. 4. Feature fidelity precision from ERmold to PDMS replica. (a) SEM images of selected sections of the PDMS replicas. (b) Flow-focusing single emulsion droplet generator microfluidic device design. Fmold fabrication conditions: First step - UVA

exposure time on reverse side = 50 s, exposure time on front side = 360 s, second step: UVA front exposure = 2 min, UVC front exposure = 17 min. (Scale bar: 1000 µm)

Fig. 5. Microdoplet generation using a microfluidic flow-focusing device. (a) Representative optical microscopy images of the microdroplet (magnification: 5x). (b) Representative size distribution of 100 subsequently generated microdoplets obtained from a single experimental run. The dispersed and continuous phase flow rates were kept constant at 0.03 $mL.h^{-1}$ and 0.01 $mL.h^{-1}$, respectively.

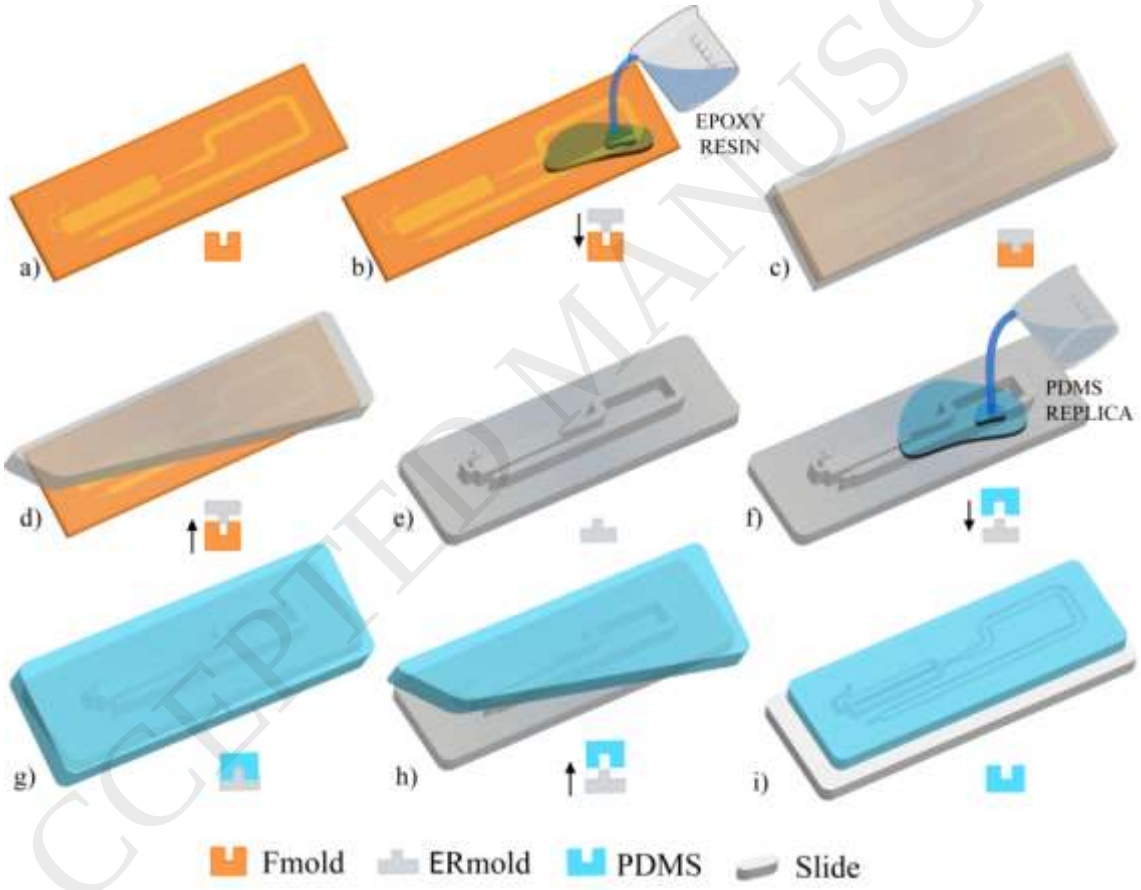
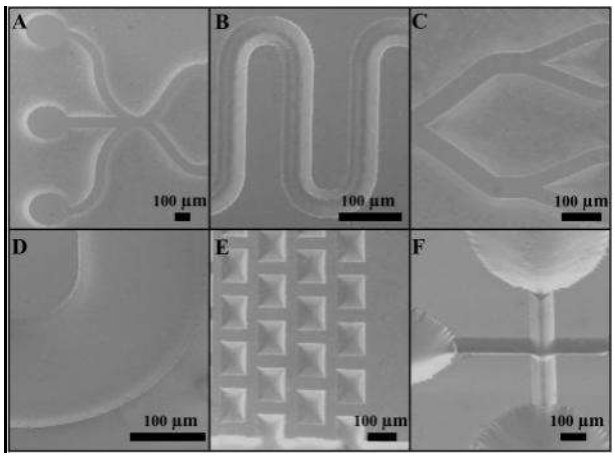


Fig. 1.



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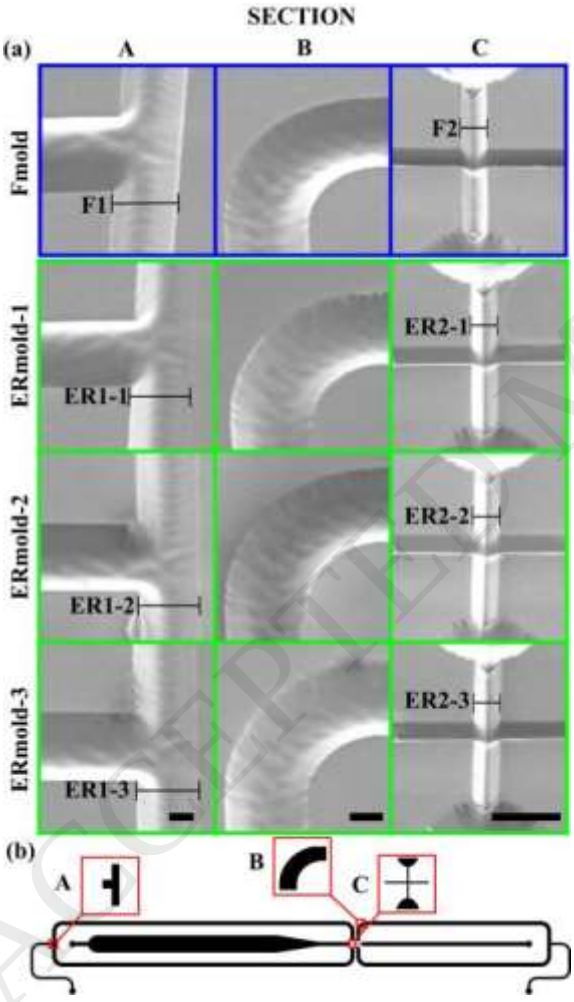

Fig. 3.

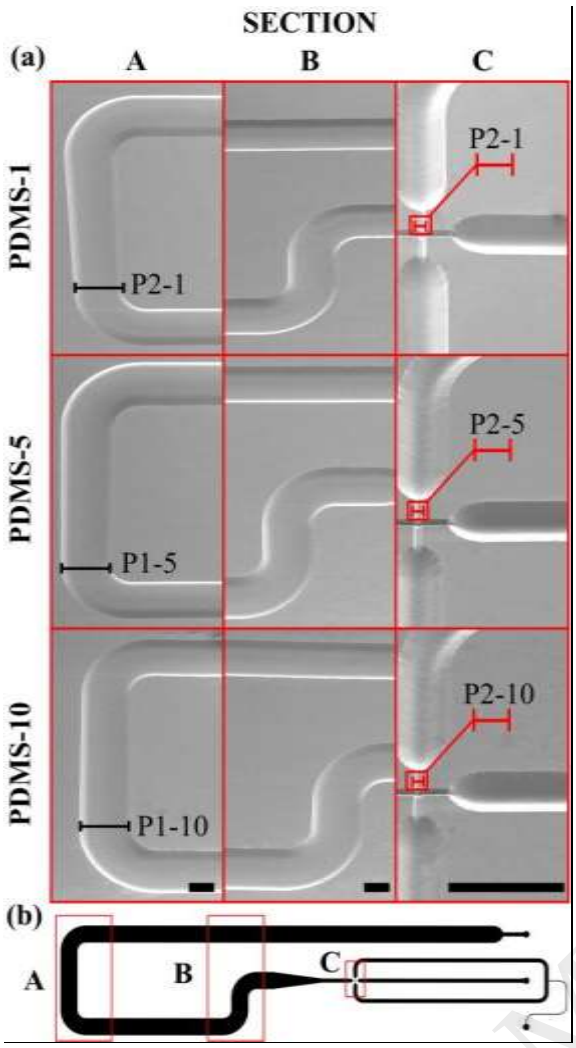

Fig. 4.

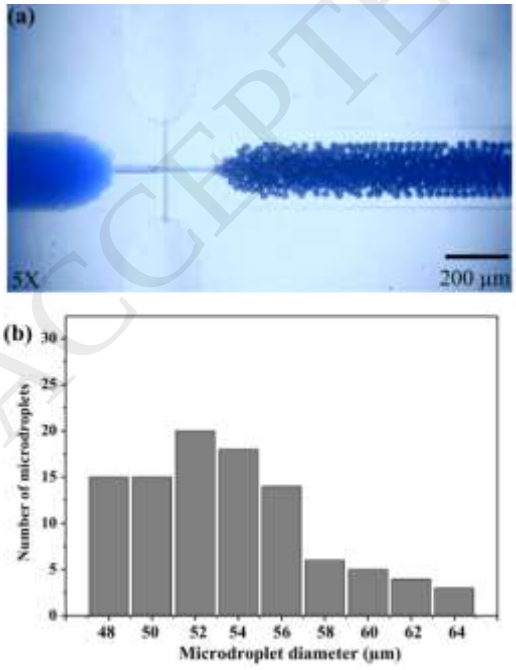

Fig. 5.