Fabrication Chain for Prototyping of Microfluidic Chips in Polymers

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Abstract

We established an integrated prototyping chain for rapid fabrication of microfluidic chips in polymers comprising fabrication of masters made from elastomers, replication into polymers by soft embossing, surface modification and thermal sealing. Our techniques enable rapid and precise fabrication of fully functionalized microfluidic chips featuring typical minimum lateral dimensions of 50 µm and aspect ratios smaller than one.

Keywords: prototyping, soft-embossing, surface-modification, sealing, COC

1. Introduction

The trend to use polymer chips in microfluidic applications results from the wide spectrum of available polymers (COC, PMMA, PS etc) with different characteristics and the general option of cost-efficient mass-fabrication techniques, e.g. by injection molding. For an efficient development of microfluidic chips at the prototyping phase, it is important to possess comprehending, high-precision polymer fabrication techniques to rapidly iterate designs. All techniques, including structuring of the substrate, surface modification and sealing must be compatible to an integrated prototyping chain (Fig. 1).

Master structures are generated by photopatterning multilayers of thick photoresist SU-8 [1] which are then replicated into cyclo-olefin copolymer (COC) disks by soft embossing [2]. Surface functionalization of channels is achieved by subsequent coating procedures followed by a thermal sealing of the disk by a designed 2-composite foil.

In the following, the processes of the fabrication chain are explained in detail.

Figure 1. Fabrication chain for prototyping microfluidic channels in polymer disks comprising mastering of microstructures for subsequent replication into polymers, surface modification and the final sealing. The overall process time starting from a mask design to a fully functionalized disk is less than four working days.

2. Fabrication of PDMS-Master

Using mold inserts made of PDMS (polydimethylsiloxane) is a powerful technique for rapid and precise fabrication of microstructured embossing tools for
replication in polymers. Since PDMS is temperature stable up to \( T = 250^\circ \text{C} \), it stands the high temperatures during hot embossing for relevant polymers such as COC, PMMA, PC or PS. We fabricated PDMS mold inserts by casting the liquid material over a multi-step SU-8 master. The SU-8 master structures are realized by successive deposition, pre-baking and exposure of several SU-8 layers on top of each other followed by a final, simultaneous development. The process parameters outlined as follows are valid for thicknesses of composite layers of SU-8 2100 and SU-8 2025 [3] up to 300 \( \mu \text{m} \). The process starts with the deposition of a thick SU-8 2100 bottom layer. Using a standard spin coater, the resist is spread on a 4” silicon wafer at a frequency of 500 rpm for 15 sec followed by spin coating at higher frequencies according to the data sheets defining the actual layer thickness. After subsequent pre-bake on a hotplate at \( T = 95^\circ \text{C} \) for 30 min, the first layer is exposed to 365 nm UV at 600 mJ/cm\(^2\). Since the minimum feature sizes in our structures range above 50 \( \mu \text{m} \), transparency masks are used. Pulsed exposure enables fair dissipation of generated joule heat during irradiation and prevents from damage of the resist. The crosslinking of the resist is induced during the subsequent post-exposure bake at \( T = 95^\circ \text{C} \) for 30 min. Deposition of further layers follows the same protocol at adapted spinning rates for SU-8 2025 layers. All SU-8 layers are simultaneously developed at room temperature for about 20 min. Next, the SU-8 structures are cast into a negative PDMS mold insert. We used Elastosil RT 607 A + B (Wacker Chemie, 81737 Munich, Germany) where the compounds A and B are thoroughly mixed at a ratio of 1:9. The mixture is evacuated before and after casting for 5 min at a time to avoid air pockets in the cured silicone leading to a porous matrix and defects in the later replicas. The silicone cures at 70°C within 3h and can easily be peeled off from the SU-8 master.

3. Replication by Soft-Embossing

The PDMS mould inserts are replicated into polymer disks (TOPAS 5013, Ticona GmbH, 65451 Kelsterbach, Germany) by the technique of embossing. The disks are assembled with the PDMS mold insert in a closed cavity made of brass consisting of a round bottom plate, a frame and a cover plate. Tight fit between mold and the framing holder is important to avoid deflection of the elastic PDMS mold during embossing. After applying the cover plate, the entire assembly is heated under vacuum using a hot press, to a temperature well above the glass transition temperature (\( T_g = 130^\circ \text{C} \) for TOPAS 5013). Beyond the material specific glass \( T_g \), the melted polymer fills the cavities of the mold insert. Applying a relatively small load of \( F = 3 \text{kN} \) for 5 min at 175°C supports the filling. Vacuum conditions during the whole process prevent trapping of air pockets in the cavities. Planarity and parallel alignment of the pressing plates of the hot press is important to avoid wedge-errors in the replicated piece. After solidification of the polymer at room temperature, the structured disk can easily be peeled off from the elastic PDMS mold insert (Fig. 2).

**Figure 2.** PDMS mold insert (above) and replicated COC disk featuring negative structures of the PDMS stamp. Note that the inner hole in the disk is milled separately from soft embossing.
4. Surface Modification

The wettability of the surface is crucial to the functionality of microfluidic devices. Untreated COC surfaces suffer from rather hydrophobic contact angles (95° for DI water) and thus do not display any self-priming properties. To overcome this, the microstructured surface of the disk is selectively modified by attaching hydrophilic and hydrophobic polymers. For the hydrophilization of the plain disk, hydrophilic polymers are attached to an O$_2$-plasma activated substrate either by spin or dip coating techniques. Upon UV irradiation, photo-crosslinking occurs between the photoactive benzophenone groups within the polymer and the COC surface.

![Diagram showing hydrophobic patch](image)

**Figure 3.** Segment on the disk showing a hydrophobic patch in a channel featuring several depths $d_1$, $d_2$, $d_3$ connected to a reservoir. The structures are replicated in COC by soft embossing from a PDMS mold which is cast over an SU-8 multilayer master. Note the exact positioning of the hydrophobic patch covering only the shallow channel segment.

Next, a hydrophobic fluoropolymer-based solution is applied to a specific channel segment. A high positioning accuracy of the hydrophobic patches is important to stop liquid flow at defined positions in the channel. Spreading of the solution along the hydrophilic channels must be prevented by accurate positioning and control of the dispensed volume. Among various approaches, wetting the tip of a felt pen with hydrophobic solution and manual “writing” the coating into the channels shows the most satisfying results for a prototyping technique (Fig. 3). This way, only little amount of solution wets the surface and the spreading of solution stops at designated corners or steps in the channel height. The affect of the coating on the channel dimensions are negligible since the hydrophobic patch measures a few nanometers, only.

5. Sealing

The challenge in sealing microchannels is to achieve a pressure-tight bond between substrate and cover which does not affect the geometry by channel deformation or sagging of cover lid. Furthermore, compatibility of materials and processes with surface coatings and waiving unstable reagents (e.g. outgassing glue or solvents) are important to devices for bioanalytical applications. Various approaches for sealing microstructured polymer substrates with cover plates have been reported [4,5,6]. Temperature diffusion bonding of two polymer parts is based on the interlink of the polymer chains when the surfaces are closely put together and heated beyond their glass transition temperature $T_g$. The contact pressure and the process temperature sensitively affect the bonding strength. The right choice of both parameters is a compromise between weak bonding strength and deformed channel cross section by molten substrate or sagging of cover lid due to thermal expansion.

In order to minimize the thermal expansion of the cover lid, we developed a composite foil featuring two layers of different $T_g$ and thickness. A thin polymer layer of micron size of low $T_g$ is coated on a foil of several 100-µm thickness of high $T_g$. When the composite foil is pressed on the substrate at temperatures fairly above the $T_g$ of the thin layer but smaller than the $T_g$ of the thick foil, the thin polymer layer melts and is strongly bonded to the substrate. The temperature-stable thick layer gives good mechanical
stability and prevents the foil from collapsing. We processed the sealing on a lamination machine featuring two heated cylinders which apply a defined pressure at a specific feed rate to the foil and the substrate. The cross-section of a sealed channel is depicted in Figure 4.

**Figure 4.** Cross section of a microchannel with 100 µm in height and 1.2 mm in width which is thermally sealed by the two composite foils. The technique provides both a tight bond between cover and substrate and unaffected cross section due to the mechanically stable foil.

6. Conclusion

We developed a compact prototyping chain for fabrication of microfluidic chips comprising tooling of PDMS masters, replication by soft-embossing, surface-modification and sealing. The techniques are characterized by their high precision, simplicity and capability to rapidly generate fully functionalized structures in a few days.

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References


