

3D polymeric microfluidic device fabrication via contact liquid photolithographic polymerization (CLiPP)

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Abstract

In this contribution, a new method for the fabrication of complex polymeric microfluidic devices is presented. The technology, contact liquid photolithographic polymerization (CLiPP), overcomes many of the drawbacks associated with other rapid prototyping schemes, such as limited materials choices and time-consuming microassembly protocols. CLiPP shares many traits with other photolithographic methods, but three distinct features: (i) liquid photoresists in contact with the photomask, (ii) readily removed sacrificial materials, and (iii) living radical processes, enable multiple polymeric chemistries and mechanical properties while simultaneously enabling facile fabrication of 3D geometries and surface chemistry control. This contribution details fabrication techniques and methods for the fabrication of high aspect ratio posts covalently bonded to a polymeric substrate, an array of independently stacked bars on top of perpendicular bars, multiple undercut structures fabricated simultaneously, and a complex 3D geometry with intertwined channels.

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1. Introduction

Since the first disclosures of microfluidic devices in the mid 1980s, researchers have found new ways to make device features smaller, materials selection wider, and fabrication faster [1–7]. Initially, the main driving forces for scaling-down macroscopic processes were immediately realized benefits, such as reduced reaction times and limited utilization of expensive reagents. More recently, however, it is the realization that changes in fluidic behavior enable new and efficient microdesigns that have driven research in the microfluidics field. These development efforts have resulted in a remarkable array of techniques for fabrication of microfluidic devices, enabling specialized structures suitable

for countless medical, analytical, and humanitarian needs [8,9]. While the scientific and commercial communities recognize the potential of microfluidics, emergence in the marketplace has been delayed by a lack of standardized fabrication methods. Currently, each device design is dependent on specialized fabrication schemes, leading to long development times and prohibitively expensive devices [10].

While fabrication techniques for silicon and glass microfluidic devices are constantly improving, the present focus has shifted to microdevices made entirely from polymeric materials [10]. The main benefits of polymeric microdevices are expense and durability, which suit field use and point-of-care diagnostic applications, as well as the availability of numerous mechanical and chemical properties achievable based on simple chemical changes in the polymer formulations. Since the development and fabrication of each device requires substantial resources, efforts have

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been made to shorten optimization time through the use of rapid prototyping techniques, where device geometries are quickly evaluated. To date, the most promising techniques for fabrication of polymeric microdevices are soft lithography, developed by Whitesides, and microfluidic tectonics, developed by Beebe et al. [11,12].

Multilayer devices are presently produced with the aid of these techniques within 24 h, substantially reducing development time for microdevices, but several limitations, e.g. limited mechanical and materials properties, have been identified and are well known in the microfluidics community.

To counter problems associated with current state of the art methods, a novel fabrication procedure has been developed [13]. The three main features are liquid monomers polymerized in contact with the photomask, sacrificial materials that are readily removed once the device is finished, and the use of living radical processes [14].

Having liquid precursors polymerized in contact with the mask has many important advantages. It provides for a convenient method to control layer thickness as well as surface geometries, and it enables better feature reproduction since the thin layer of air that is usually present between the mask and photoresist is eliminated. Furthermore, it counters problems associated with atmospheric oxygen inhibition and eliminates the need for flow controlling additives and solvents in the formulation since leveling is provided by contact with the mask. Independence from viscosity and flow properties provides for almost unlimited formulation choices. This ability opens the microfluidics field to devices constructed from polymers with very diverse material properties. Rubbery, glassy, hydrophilic, and hydrophobic polymers are easily accessible, addressing different needs in optimized devices. Uniquely, this complex array of materials and surface chemistries is exploited with multiple materials in one-layer and in complex, multilayer devices.

The sacrificial material is critical in the fabrication of three-dimensional (3D) devices, since it provides a convenient route for exclusion of the liquid monomer from channels and voids. However, complex monolithic 3D device fabrication does not depend on a sacrificial layer alone. The necessary seamless integration of sequentially fabricated polymer layers is achieved through the use of a living radical process. This mechanism provides for covalent bonding between features and also enables surface modifications at different stages of the device fabrication. These surface modifications serve a number of needs, e.g. as flow modifiers or as biologically active detection sites.

This work details the use of contact liquid photolithographic polymerization, hereafter referred to as CLiPP, and the use of sacrificial materials in the fabrication of seamless, monolithic 3D devices with complex geometries. Specifically, the challenging task of producing 3D tortuous channels with complex features in each layer is detailed. This device is an important model for improved, integrated designs where specialized detection structures, such as diode laser arrays, are accessed numerous times from arbitrary sections of the

device and for applications where liquid streams need to cross each other without risking cross-contamination. The 3D ability minimizes the number of individual layers needed to solve a specific task, reduces cost, and improves performance while keeping the device footprint to a minimum.

2. Experimental

The basic component of the experimental set-up is a modified mask alignment system where an in-house built reaction chamber is incorporated to make polymerizations of thick, planar layers of liquid polymer precursors facile. Photolithographic features such as masks in conjunction with collimated UV-light and micromanipulators for mask alignment are retained.

2.1. Experimental set-up

As the basis for the experimental set-up, a Hybralign 200 mask alignment system (OAI, Milpitas, CA) with a 3 in. diameter UV-light source (Oriel Instruments, Stratford, CT) is used. Shown in Fig. 1 is a drawing of a custom designed polymerization chamber. The system provides a 2 in. \times 2 in. polymerization area and bottom plate that is vertically mobile up to 5 mm. Micromanipulators for alignment in the x - y plane were retained from the original mask alignment system, and rotating the mask holder itself corrects for angular alignment errors.

2.2. Materials

A typical polymer precursor formulation for device fabrication consists of 1.5% (w/w) 1-hydroxycyclohexyl phenyl ketone (tradename: Irgacure 184, Ciba, Tarrytown, NY) as the photoinitiator, 1.0% photoiniferter precursor, tetraethylthiuram disulfide (TED, Aldrich Chemical Co., Milwaukee, WI), and 1.0% acrylic acid (Aldrich), in a mixture of 50% (w/w)

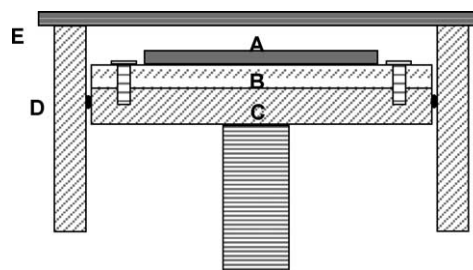


Fig. 1. Schematic of a custom-built fabrication chamber. A polycarbonate substrate (A) is glued onto a moveable bottom portion comprising a flat stainless steel structure (B), and a removable aluminum plate (C) secured with four screws. An o-ring provides a seal between the moveable bottom portion (B and C) and the chamber walls (D) to prevent leakage of liquid monomer into the inner mechanism of the fabrication chamber. The reaction environment is completely enclosed with a photomask supported on a glass plate (E). An LVDT sensor tracks the absolute position of the bottom section (not shown).

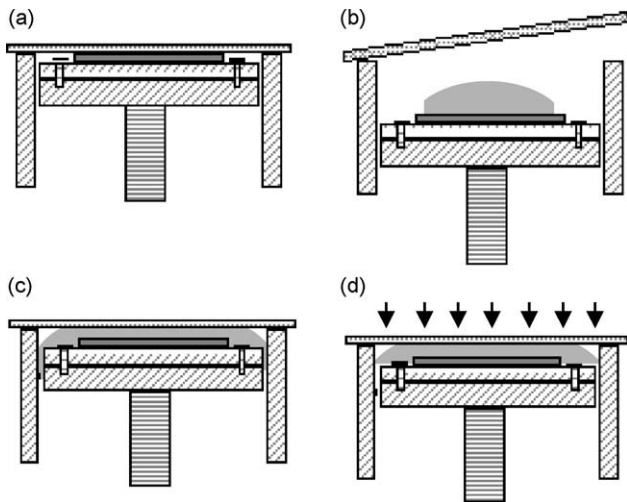


Fig. 2. Schematic of the procedure for the fabrication of a single-layer device.

triethyleneglycol diacrylate (SR 272, Sartomer, Exton, PA), 10% hexavinyl aromatic urethane acrylate (EBECRYL 220, UCB), and 36.5% of a divinyl aromatic urethane acrylate (EBECRYL 4827, UCB).

2.3. Fabrication

2.3.1. Fabrication scheme of one-layer structures

A typical process for constructing a single layer starts with a substrate (usually a thermoplastic sheet cut to size, e.g. PMMA or polycarbonate) glued in place on the moveable bottom plate and a photomask secured on the planar top using transparent wax between the mask and the glass (see Fig. 2).

In the first step, depicted in Fig. 2(a), the substrate is raised until the top of the substrate contacts with the mask and a baseline measurement is performed utilizing an LVDT sensor connected to the moveable bottom section. This measurement gives the absolute position of the top of the substrate. The substrate is lowered until there is a gap formed between the top of the substrate and the mask plane, thus defining and allowing control of the thickness of the layer that is to be produced. The mask is attached on a hinged mask holder so it can be temporarily removed from the curing chamber. When the mask holder, with the mask firmly

secured, is removed, the liquid polymer precursor is poured onto the substrate (Fig. 2(b)), and subsequently, the mask is lowered in place. The layer thickness is adjusted by vertically positioning the bottom portion of the fabrication chamber until the desired layer thickness is achieved (Fig. 2(c)). The polymer layer thickness is determined by the distance between the mask and the top of the polycarbonate substrate, i.e. the difference between the LVDT readout in steps (c) and (a). The monomer contacts the mask and spreads over the entire substrate surface in the course of raising the substrate to the desired position. The monomer is now confined between the mask plane and the substrate plane, defining the layer thickness as well as enabling level surfaces. When exposed to collimated UV-light, monomer in sections where the mask is transparent polymerizes, forming an insoluble polymer network. The polymeric precursor is effectively a negative-type photoresist where masked regions remain in the liquid state. The mask is removed and excess material, i.e. non-cured polymer precursor, is removed by washing with methanol, resulting in a layer of predetermined thickness having features corresponding to the negative image of the photomask (Fig. 2(d)).

In Fig. 3, examples of one-layer devices fabricated using the methods described in the previous section are presented.

2.3.2. Fabrication scheme of multilayer devices

In the CLiPP process, multilayer devices are fabricated from individual layers formed sequentially on top of each other. To prevent blocking of channels and other voids in the previous layers, a sacrificial layer currently consisting of a histology wax is used (Paramat Extra, Electron Microscopy Sciences, Fort Washington, PA). After each layer is polymerized, the device under construction is removed from the curing chamber and put on a hot plate where the temperature is set to 10 °C above the melting point of the wax, i.e. 60 °C. When the device is thoroughly heated, melted wax is poured onto the surface. The wax quickly fills the voids, creating volumes that are inaccessible to the liquid monomer. A razor blade is contacted to the surface and quickly moved over it, leaving only a thin wax film on planar surfaces while wax in channels and voids is unaffected (Fig. 4(a)). When cooled down to room temperature, the surface is solvent polished until only trenches and voids are filled with sacrificial material (Fig. 4(b)). It is imperative to remove wax

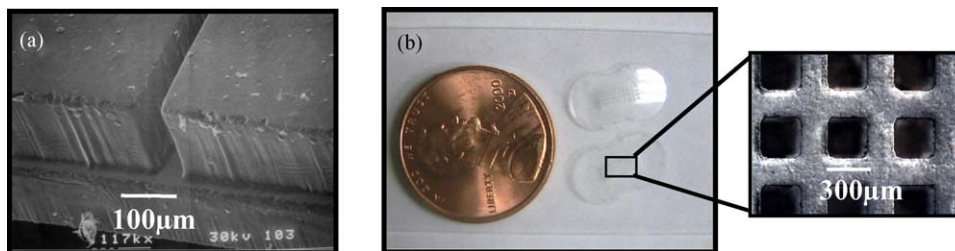


Fig. 3. Examples of one-layer devices. (a) A 65 μm channel with a height of 200 μm (aspect ratio of 3). The striations in the picture are due to the 15 μm pixels from which the mask features are constructed (5080 dpi printer) and (b) 240 μm thick cell scaffolds for cell culture in 300 μm wells.

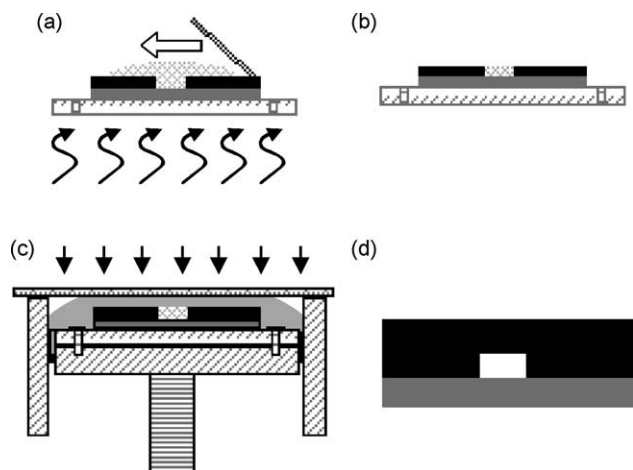


Fig. 4. Schematic of the procedure for the fabrication of multilayer devices. (a) The aluminum plate, with the substrate and the first polymerized layer, is detached from the fabrication chamber and placed on a hot plate. Melted wax is poured on top of the heated assembly and quickly fills voids and cavities. The surface is leveled with a razor blade, leaving only a thin wax layer on top of the structure while cavities are completely filled. (b) A solvent polish step removes the thin wax layer, ensuring good adhesion to the subsequent layer. (c) The aluminum plate is repositioned in the fabrication chamber and steps described in Fig. 2 are performed to build the top layer. (d) The final structure is released when the detached substrate is subjected to hot solvent and pressure to remove the wax, leaving a monolithic polymeric structure.

from surfaces where subsequent layers are to be attached since residues prevent covalent bonding between the layers, resulting in poor device integrity. Following the removal of excess wax, a new layer is polymerized according to the procedure outlined in Section 2 (Fig. 4(c)). These steps are repeated as many times as necessary to form the desired polymeric structure. The final device is realized through removal of all sacrificial structures by immersing the device in Citrisolv solvent (Fisher Scientific, Pittsburgh, PA) heated to 10 °C above the wax melting point. Positive pressure is used to remove the melted wax, resulting in a finished device with unobstructed channels (Fig. 4(d)).

2.4. Polymerization conditions

Polymerizations were conducted at ambient conditions using a medium density Hg-light source with a spectral output between 300 and 450 nm. No cut-off filters were used. The light intensity was set at 50 mW/cm². Typically, the curing time was 350 s, yielding a dose of 17.5 J/cm². This dose was used irrespective of layer thickness, yielding good patterning results in the thickness range of 100–500 μm.

3. Results and discussion

3.1. Pattern reproduction

The quality of the pattern transferred from the photomask to the polymer is determined by many factors: the resolution

of the photomask, degree of UV-light collimation, diffraction, refraction, and the polymerization mechanism of the polymer precursor. Generally, good pattern transfer requires control over all these factors, but several factors are more important than others. In the following sections, two phenomena are highlighted: diffraction and polymerization kinetics, and their effect on the quality on pattern reproduction is discussed.

3.2. Diffraction

The most desirable masking techniques are those that eliminate diffraction between the mask and the resist. In CLiPP, this feature is the case, since the mask features are in contact with the polymerizable liquid, eliminating the thin layer of air that usually is present when solid resists are used. Contact yields the sharpest possible light intensity transition between transparent and masked regions, thus enabling very high aspect ratios and sharp angles between a planar surface and feature walls. While minimized in CLiPP, some portion of the light is diffracted in the material during the curing process, due to small air bubbles present in the liquid and from changes in refractive index when the liquid is transformed to a solid. These phenomena lead to a finite light intensity in masked portions of the liquid. Therefore, to achieve desired feature quality, polymerization control is imperative.

3.3. Polymerization control

To counter polymerization in unwanted regions, control over propagation and/or termination reactions becomes critical. Radical chain growth polymerization is difficult to control since termination of chains is dependent on the square of the radical concentration, meaning that a low radical production rate invariably leads to longer chains in the absence of efficient chain transfer mechanisms. Therefore, network formation typically remains possible and even likely in masked regions. Since the liquid polymeric precursor has a low viscosity, diffusion of radical scavengers such as O₂ and hydroquinone mono methyl ether (MEHQ) is rapid in unpolymerized regions [15]. Diffusion competes effectively with radical production where the radical production rate is low, preventing polymeric network formation in unwanted areas. Where the radical production rate is high, diffusion of terminating species is unable to compete with radical formation, which leads to cross-linked polymer with a high conversion in unmasked regions.

While the presence of dissolved oxygen in the polymer precursor is advantageous to control pattern fidelity, it causes problems if the oxygen diffusion rate is of the order of the radical production rate. In systems open to the atmosphere, oxygen replenishment is sufficient to prevent complete polymerization at the top of the sample [16]. Partial polymerization leads to tacky and soft surfaces, causing handling and structural integrity problems, particularly in films less

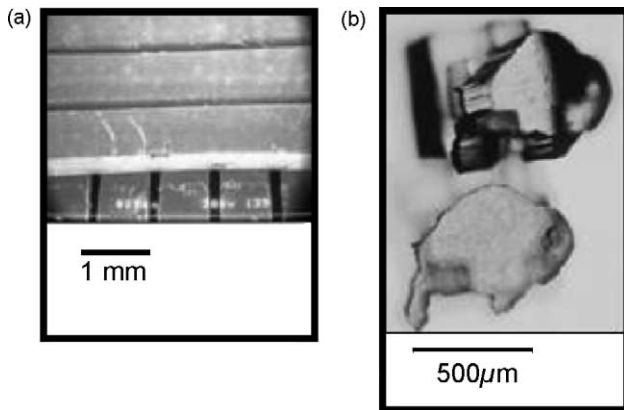


Fig. 5. Structures obtained using the sacrificial wax technique. (a) An SEM picture of a 500 μm thick structure composed of 800 μm wide bars on top of similar but perpendicular bars. This geometry would require multiple assembly steps if fabricated from premade structures. (b) An optical microscopy picture showing 700 μm long buffaloes on pedestal structures. Four hundred of these undercut microstructures were fabricated simultaneously.

than 50 μm thick. Numerous methods have been developed to counter oxygen inhibition, but with the exception of lamination techniques, they invariably affect not only the atmospheric oxygen but also the desired dissolved oxygen. The CLiPP scheme is similar to lamination techniques since it provides a solid barrier to atmospheric oxygen, countering surface inhibition while retaining the positive effects of oxygen dissolved in the polymer precursor.

3.4. Multilayer devices

Microfluidic applications generally require extensive channel systems. Channels are 3D objects, and depending on the fabrication method, at least two to three polymerized layers are needed for their construction. Multilayer devices present unique fabrication challenges. State of the art processes typically employ fabrication of individual layers out of thermoplastics, glass, silicon, or PDMS, and subsequent alignment and bonding of the layers to form functional 3D geometries. However, the CLiPP scheme allows for covalent bonding between adjacent layers as the liquid polymer precursor is cured, rendering a separate bonding step superfluous. The use of a curable liquid formulation directly in contact with a layer that has trenches and voids poses significant challenges. One possible way of solving this problem is to mask the liquid so that it is prevented from curing in the void sections. While this is sufficient in some cases, it is impossible to form enclosed channels or other undercut geometries in this manner. The CLiPP method solves this problem through the use of sequential fabrication of layers and sacrificial materials, as described in Section 2. In this manner, it is possible to fabricate multiply parallel undercut structures and criss-cross patterns of bars that would require multiple assembly steps using any other technique (Fig. 5).

3.5. Sacrificial material

A mainstay in the integrated circuit industry is the use of sacrificial layers. The basic idea is to exclude certain volumes in the device with sacrificial material, deposit structures around them, and subsequently, free the desired structures by preferential removal of the sacrificial layer. In this manner, channels and other void volumes are easily obtained. For the CLiPP method, the best sacrificial material was found to be Paramat Extra. This histology wax was selected for the low melting temperature, a very low melt viscosity, and the relatively smooth surfaces that are formed when the wax has solidified. While this scheme allows for rapid fabrication of 3D structures, there are problems associated with it. The primary issue is channel depth variability. These variations have two sources: (i) the wax shrinks when cooled, thus creating a wax layer with a surface that is at a lower level than the sides of the polymerized void and (ii) the channel roof becomes slightly uneven since the wax does not solidify into a completely even surface. For the successful use of the CLiPP process for devices with small tolerances, i.e. $\pm 10 \mu\text{m}$, the issues associated with the wax must be resolved, either through substitution of the sacrificial material or a new way of controlling the shape and surface roughness of the sacrificial material.

3.6. Covalent bonding, adhesion

Interlayer bonding is a key feature in any multilayer microdevice. Many different schemes have been developed to ensure good adhesion between prefabricated layers, but they invariably involve one or more extra fabrication steps, e.g. surface treatment, deposition of a bonding material, and subsequent activation with heat, light, etc. It has also proven to be difficult to ensure leakage free seals between the top layer and channel structures while simultaneously ensuring that the channel features are intact. CLiPP makes use of the active iniferter surface groups to ensure good adhesion wherever liquid monomer precursor is in contact with previously polymerized structures. In this manner, it is possible to fabricate posts that are covalently bonded to flat surfaces, as well as criss-cross patterns of bars that would require multiple assembly steps using any other technique (Fig. 6).

3.7. Complex 3D microfluidic devices

Integrated microfluidic devices have received considerable interest as a means of providing lab-on-a-chip applications tailored to point-of-care testing and medical diagnostics [17,18]. CLiPP is very compatible with the trend of increasing device complexity by providing the microdevice designers with many more materials and geometrical options, enabling more functional components for any given device volume.

Using traditional methods, fabrication of complex, 3D devices with structural features in each layer is a very labor intensive and demanding task. Current state of the art

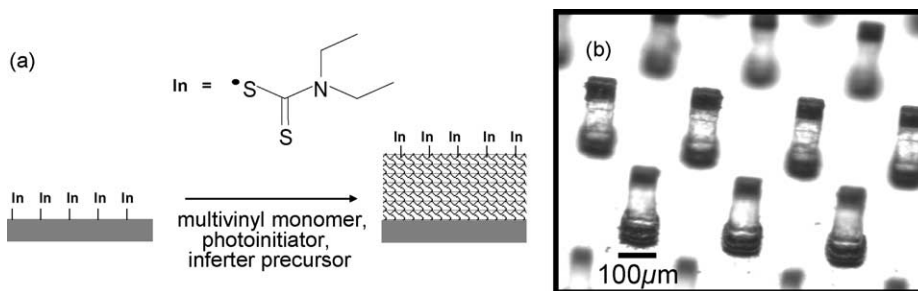


Fig. 6. The living radical process as an adhesion promoter. (a) The structure to the left depicts a previously polymerized layer with reinitiable groups (In) attached to surface. When exposed to UV-light, these groups cleave, leaving active radicals tethered to the polymer matrix. These radicals initiate reactions with monomers in the immediate vicinity, i.e. the polymer precursor forming the next layer of the device, ensuring good covalent bonding between the layers. (b) Covalently bonded posts with an aspect ratio of 4, used in a tissue perfusion microdevice.

processes typically require mechanical micromachining of channel connections between layers, which affect geometrical complexity and limit the designer's freedom [19]. Soft lithography, it should be noted, alleviates these concerns, but introduces other problems, as has been previously described.

The use of sacrificial materials allows for the fabrication of very complex designs since all cavities and voids in previous layers are filled and inaccessible to liquid monomer. It is, therefore, possible to fabricate devices that contain complex shapes in each layer. A 3D geometry with intertwined channels was manufactured using the steps detailed in Section 2, i.e. a successive polymerization of stacked layers and the introduction of sacrificial material after each polymerization step. The mask sequence used for the fabrication is shown in Fig. 7(a–c). The sequential build-up of the structure is indicated by the schematic drawings shown below the masks, where dark features represent the void sections formed by the corresponding mask and the wax-filled features in underlying layers are shown in gray. The final structure resulting from this fabrication scheme is shown with dyed liquids added to highlight the independent channels that are intertwined in three dimensions.

While the structure presented in Fig. 7 is not a device per se, the ability to manipulate liquids in three dimensions is desirable in multiplexed applications. A potential application for 3D features is cell-sorting. Here, cells are characterized spectroscopically and, depending on the cell type, are diverted to different channels using the optical forces of a laser. Finally, the cells are counted and collected for further evaluation. Since the laser used in the sorting step has the ability to very rapidly switch chip-location, parallel channels containing separate samples could be sorted simultaneously, drastically reducing the time required for multisample tasks. Currently, this result is difficult to achieve since an optimized device has a minimized footprint, and thus only contains one waste channel common to all sorting streams. Hence, streams need to cross without risk of contaminating each other. Using the abilities of CLiPP, a 3D device, similar to that shown in Fig. 7, which allows for crossing streams without the risk of dilution and cross-contamination, is easily realized.

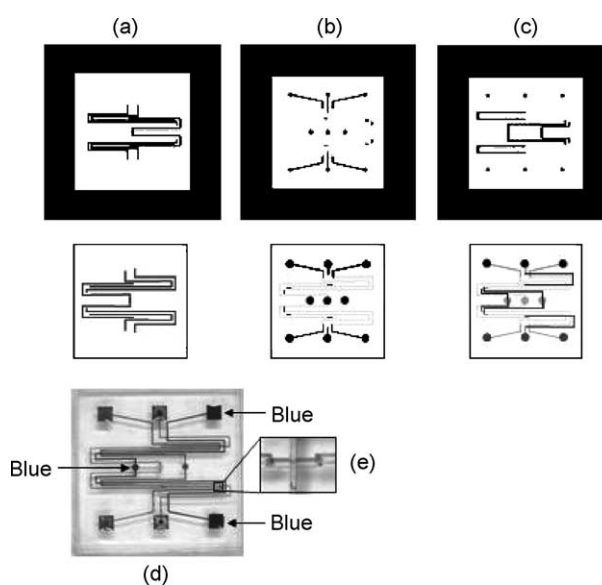


Fig. 7. Schematic of the mask sequence used to fabricate a complex 3D device where three separate channels form an intertwined geometry. The work flow is indicated by the mask sequence (a–c), where the fabrication sequence is indicated by the schematic under the masks where newly formed features are dark and features formed in lower layers are indicated in gray. (d) A finished 3D device with three intertwined tortuous channels, filled with dyed liquids. The arrows point to an inlet and an outlet located on the right hand side of the finished device. The fluid passes around two separate streams as indicated by the arrow on the left. The device illustrates the concept of access to multiple points in a device from an arbitrary location. (e) A detail of the 400 μm wide channels, where it is clearly seen how the channels cross without connecting.

4. Conclusions

In this contribution, a novel technique for the fabrication of three-dimensional polymeric microdevices, contact liquid photolithographic polymerization, is presented. This new technology overcomes many problems associated with other rapid prototyping techniques in that geometries and polymeric materials both are optimized simultaneously and independently. The fabrication of a complex 3D device with

intertwined channels is detailed as are the methods used to covalently bond high aspect ratio posts to polymeric substrates and, using the batch processing capabilities of CLiPP, the process of massively parallel fabrication of undercut microdevices is demonstrated. This 3D microfluidic device illustrates the ability to access any portion from other parts of the device, resulting in optimal use of the allotted volume while reducing the cost of addressing demanding microfluidic operations.

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