

Microfluidic stickers†

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We present how to make and assemble micro-patterned stickers (μ PS) to construct high performance plastic microfluidic devices in a few minutes. We take advantage of soft UV imprint techniques to tailor the geometry, the mechanical properties, and the surface chemistry of 2D and 3D microfluidic circuits. The resulting microfluidic stickers substantially overcome the actual performance of the very popular PDMS devices for a wide range of applications, while sharing their celebrated fast and easy processing. To highlight the intrinsic advantages of this method, three important applications are detailed: (i) we show that both aqueous and organic droplets can be produced and stored in stickers without any specific surface coating. (ii) We report on the outstanding pressure resistance of the μ PS, which open the way to the transport of viscous complex fluids. (iii) Finally, a simple design strategy is proposed to generate complex flow patterns in interconnected stacks of μ PS.

Introduction

Since the pioneering work of Delamarche *et al.*,¹ elastomer materials have been considered as excellent options for the fast fabrication of microfluidic chips.² Microfluidic devices made of PDMS (polydimethylsiloxane) by replica molding are now part of the standard instrumental toolbox of many research laboratories, ranging from molecular and cell biology to hydrodynamics and analytical chemistry. The reason for this ubiquitous spreading is twofold: (i) replica molding allows for submicron size pattern replication; yet it neither requires expensive facilities and equipments, such as a clean room, nor advanced skills in microfabrication.³ (ii) PDMS combines numerous excellent physical and chemical properties: it is optically clear and has low autofluorescence,⁴ it is permeable to gases,⁵ it has a low elastic modulus and thus allows for simple integration of active elements, such as valves and pumps.⁶ Nevertheless, some of the properties of PDMS strongly limit its range of applications. First, this silicon elastomer swells in most organic solvents,⁷ which mostly limits its use to aqueous solutions. Second, permanent modification of the chemical properties of PDMS surfaces is a challenging task.⁸ This narrows the range of possible formulations in multiphase flows.⁹ Third, due to the low elastic modulus of PDMS, low pressures can significantly alter the geometry of the channels, and the associated relaxation time can reach hours for viscous fluids. This bounds the working pressure typically below ~ 1 bar.¹⁰ In turn, this prevents the accurate transport of viscous fluids, such as concentrated polymer

solutions making rheological measurements almost impossible in PDMS devices.¹¹

Only few other simple prototyping strategies have been proposed to circumvent these limitations. Desimone and coworkers have replaced PDMS by low modulus solvent resistant fluoropolymer.¹² So far this technique is restricted to experts in polymer chemistry. Alternatively, the replacement of replica molding by direct photolithographic patterning of photoresist resins is a promising and straightforward method for the fabrication of single layer polymeric microdevices.^{13–15} But, so far these techniques are restrained to millifluidic channels (typical size: 100 μ m–1 mm).

In this paper, we present a simple prototyping method to make micro-patterned stickers (μ PS) and show how to assemble them to form microfluidic circuits in a few minutes (Fig. 1A). In a first part, we show how to pattern and handle

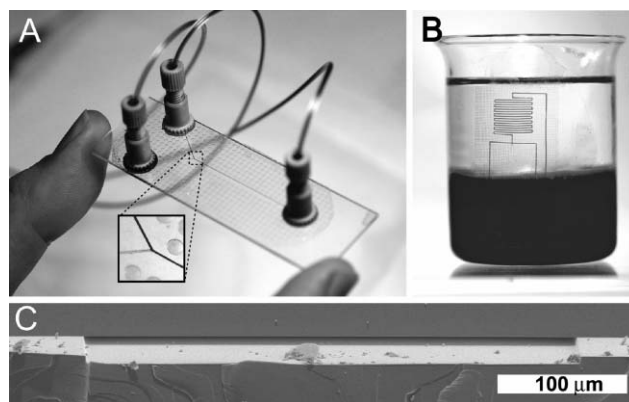


Fig. 1 (A) Microfluidic circuit made of a μ PS sealed by a glass slide. (B) μ PS bound on a curved surface: the outer side of a 50 ml glass beaker containing water and orange G dye and hexadecane. A hole made with a sand blaster connects the inner side of the beaker to the microcircuit. The liquid flow is driven by the hydrostatic pressure. (C) SEM picture of the cross section of a μ PS microfluidic channel (NOA81) bound to a glass slide. Note the absence of sagging, even for this very low aspect ratio channel (width: 350 μ m, height: 7 μ m).

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† The HTML version of this article has been enhanced with colour images.

thin plastic films with adhesive capabilities using soft imprint lithography.¹⁶ This technique offers the opportunity to tailor not only the geometry but also the mechanical properties and the surface chemistry of the μ PS. We then explain how to stack and seal the stickers to construct simple or complex 2D or 3D microdevices that can combine high spatial resolution, compatibility with organic solvents, and outstanding mechanical resistance. In a second part, we present three important applications in order to highlight some of the advantages of the microfluidic stickers: (i) hydrophilic and hydrophobic devices for digital microfluidics, (ii) pressure resistant microchannels for the transport of complex fluids, and (iii) multilayer 3D microfluidic circuits. Finally, we provide a thorough description of the fabrication protocols of each device.

Fabrication

The stickers are made by soft imprint lithography, as sketched in Fig. 2A. A drop of photocurable monomer (or prepolymer) is deposited on a flat or patterned PDMS substrate. A structured PDMS stamp is then gently pressed onto the drop. To cure the polymer film, the liquid is insolated through the transparent PDMS stamp. Since oxygen inhibits the free-radical polymerization used here to build the polymer network, the permeability to gas of the PDMS ensures that an ultra thin superficial layer of liquid remains uncured.¹⁶ Therefore, both sides of the resulting textured film retain adhesive capabilities. For most imprint processes, the total thickness of the sticker exceeds the height of the pattern due to the usual residual layer remaining below the stamp (Fig. 2A). This residual layer can be suppressed without any further etching process, by matching its height with the thickness of the uncured monomer layer. However, this requires a fine tuning of the exposure dose. To obtain such stencil like stickers, it is actually much more convenient to first contact the two PDMS substrates and then fill the empty space in between with the liquid prepolymer (Fig. 2B). In practice, the gap is spontaneously filled due to capillary forces.³ To speed up the capillary spreading, the parts of the stamp surrounding the pattern can be decorated with a dense network of pillars. This porous structure enhances the local curvature of the liquid meniscus and consequently the Laplace pressure which drives the liquid motion becomes higher.¹⁷

Assembly of microfluidic device: stacking and sealing

We now describe how to handle and assemble the plastic stickers in order to construct microfluidic devices. The main idea consists in *never* handling freestanding plastic films. Such thin sheets would be almost impossible to manipulate because of their very low bending modulus. A first sticker still lying on one of its PDMS mold is gently pressed by hand using a rigid substrate (glass slide, cover slip, metal plate *etc.*) and bound to it by photocuring the remaining reactive layer (Fig. 2C). This straightforward sealing of these adhesive μ PS is one of the main advantages of this technique. Excellent adhesion is achieved without any specific chemical, thermal or mechanical (high pressure) treatment. Furthermore, the high flexibility of the thin stickers enables bonding on curved surfaces (Fig. 1B). The PDMS mold is then removed, which makes the second adhesive side of the sticker accessible. The process can be reiterated to stack multiple stickers made of identical or different polymers. Each layer constitutes a different microfluidic network. These networks can be interconnected by means of stencil like stickers. Eventually, a second rigid substrate can be added on top of the upper layer to strengthen the device. Each rigid substrate can include access holes for the macroscopic fluidic connections.

Materials

In practice, the choice of the μ PS materials is driven by the application of the microfluidic circuits. The design strategy described above is very general and can be applied to an almost unbound number of combinations of polymers and channel geometries. However, most of the stickers used in the experiments described below have been made of the thiolene based resin NOA 81 (Norland optical adhesive). This resin offers an excellent combination of physical and optical properties complementary to the one of PDMS. (i) NOA 81 has shown a greater solvent resistance to swelling than PDMS.¹⁵ For example, we have flown apolar solvents, such as decaline and hexadecane, in devices made of two NOA 81 stickers for several hours. In PDMS channels of identical geometry, the swelling was so strong that it completely prevented the filling of the device. (ii) NOA 81 has an elastic modulus 3 orders of magnitude higher than PDMS (typically 1 GPa). This avoids sagging effects, even for very low aspect ratio shallow

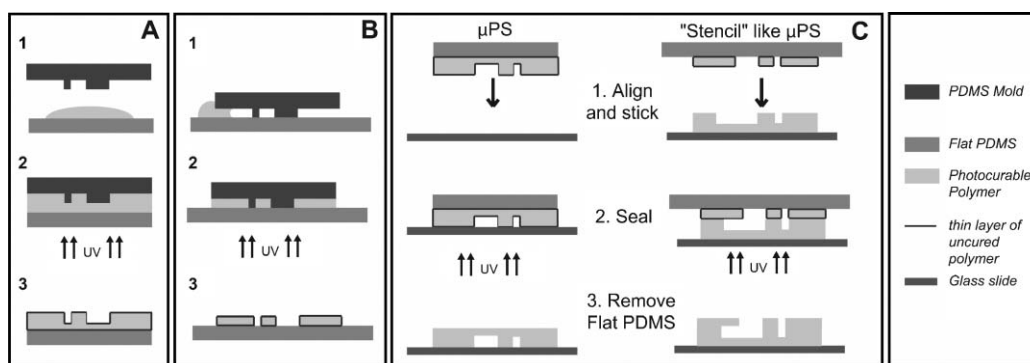


Fig. 2 (A) Sketch of the fabrication process of a μ PS. (B) Sketch of the fabrication process of a stencil like μ PS. For both methods the two surfaces of the sticker still have reactive sites after UV illumination. (C) Construction of microfluidic devices. (Left) One layer device: the circuit imprinted on the μ PS is sealed with a glass slide. (Right) Multilayer devices: the stencil like μ PS is sealed with a sticker previously bound to a glass slide.

channels. To illustrate this important advantage, we show in Fig. 1C a SEM picture of a straight channel with a 1 : 50 aspect ratio. Note that contrary to replica molding, the μ PS fabrication method is especially well suited for making and handling thin polymer films with a high elastic modulus. (iii) NOA 81 enables the replication of submicron features. (iv) NOA 81 is a resin designed for optics applications. An important advantage is its very low autofluorescence which, for instance, makes possible the tracking of single quantum dots in microfluidic stickers.²⁷ More quantitatively, we have compared the fluorescence background of 170 μ m films made of PDMS, NOA 81 and PMMA. The excitation wavelength was 488 nm and the emission was measured at 520 and 580 nm. The fluorescence intensity was normalized by the intensity measured with a glass cover slip of the same thickness. The autofluorescence level measured for the NOA films is 4 (resp. 5) times smaller than for the PDMS (resp. PMMA) for both wavelengths. (v) NOA 81 is biocompatible. Several living cells, including neurons, have been observed over days in microfluidic stickers.²⁷ (vi) Contrary to a PDMS device, NOA 81 stickers are impermeable to air and water vapor, which prevents the evaporation of the liquids flowing in the channels. (vii) Like PDMS micromolding, the soft imprint of NOA 81 films enables the replication of (sub)micron size features. For example, we managed to imprint arrays of cylindrical microwells of radius 1 μ m and 3 μ m depth.

However, this resin has a high shear viscosity (~ 300 mPa·s at 25 °C), this makes the fabrication of stencil like μ PS difficult. To circumvent this limitation, we also detail how to use another photocuring mixture with lower viscosity: tri(methylolpropane)triacrylate (TMPTA) + 1 wt% of photo initiator (Darocur 1173, Sigma Aldrich).

Applications

We now present three microfluidic experiments done in devices made of patterned stickers. These examples are intended to highlight more accurately some of the advantage of the μ PS to make performance devices. For the sake of clarity, all the detailed protocols used to fabricate the corresponding microfluidic circuits are provided in the last section of this paper.

Hydrophobic and hydrophilic drop emitters

The stabilisation of emulsions or foams requires the slow down of the coalescence of the droplets or bubbles. The basic strategy consists of adding a third component (surfactants, polymers or small particles) to kinetically stabilize the liquid dispersion.¹⁸ Given the very high surface over volume ratio of microfluidic channels, another important requirement has to be fulfilled to produce, transport, and store microfluidic emulsions. It is indeed necessary to prevent the partial wetting of the channels walls by the droplets.⁹ For instance, it is impossible to produce and store oil in water emulsions in native hydrophobic PDMS channels, even by adding surfactants to the aqueous phase. To overcome this strong limitation to liquid formulation, the usual strategy consists of changing the wetting properties of the channel walls by modifying their surface chemistry. Again, in the case of PDMS, long term stable surface modification remains challenging. For the most

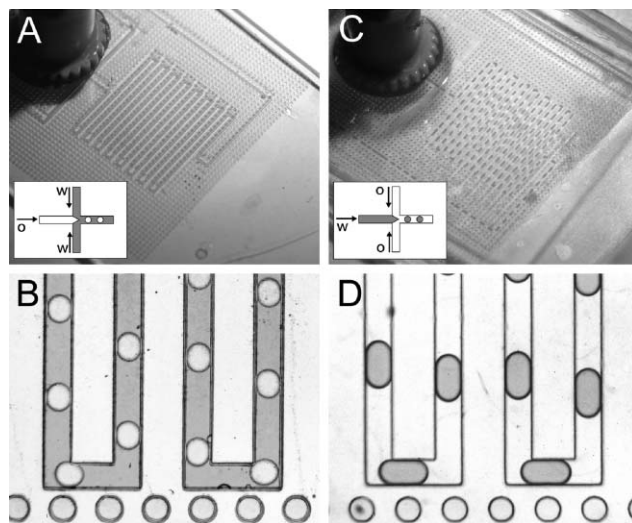


Fig. 3 (A) Picture of a flow focusing drop emitter made with a single NOA 81 sticker. Monodisperse droplets of hexadecane are produced in a solution of orange G dye and 1 wt% sodium dodecyl sulfate in water. (B) Magnified view of the output serpentine channel transporting the direct emulsion. Channel width: 200 μ m, channel height: 80 μ m. (C) Picture of a flow focusing drop emitter made with a single tri(methylolpropane) triacrylate sticker. Monodisperse plugs of an aqueous solution of orange G dye are produced in a hexadecane and 1 wt% span 80 solution. (D) Magnified view of the output serpentine channel transporting the reverse emulsion. Channel width: 200 μ m, channel height: 80 μ m.

conventional oxygen plasma treatment, the original hydrophobicity is typically regained after a few minutes.¹⁹ Moreover, surface modifications are often incompatible with the standard sealing methods.

As the μ PS can be made of hydrophilic or hydrophobic polymers, the wetting properties of the channels walls can be chosen to produce either direct or reverse emulsions. We show in Fig. 3, two identical flow focusing drop emitters made in a hydrophilic (NOA 81) (Fig. 3A), and in a hydrophobic resin (TMPTA) (Fig. 3C). A monodisperse hexadecane in water emulsion is generated in the first device (Fig. 3B), the reverse water in hexadecane emulsion is produced in the second one (Fig. 3D). This demonstrates that specific surface modifications can be bypassed by an appropriate choice of the stickers materials to produce and transport aqueous and organic drops.

Pressure resistant devices for the transport of complex fluids

Microfluidics is a new tool to investigate the flow of complex fluids, such as concentrated polymer or surfactant solutions.^{11,20,21} However, pressure drops induced by the flow of such viscous fluids (up to several Pa·s) in microfluidic devices can rapidly reach several bars. In this high pressure range, mechanical deformation of the channels occurs when working with low elastic modulus material.¹⁰ As a consequence, the change in the geometry and the long transient regimes associated with the mechanical relaxation are two major limitations of PDMS microdevices. In this paragraph, we compare the mechanical performances of PDMS and μ PS channels.

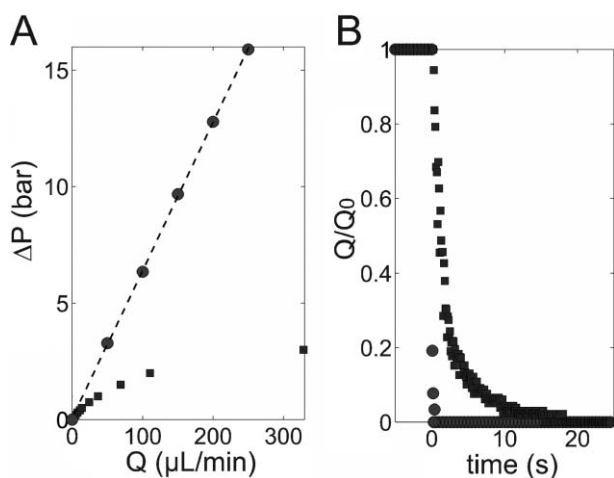


Fig. 4 (A) Pressure drop, ΔP , versus flow rate, Q , curve for 2 cm long ($200 \times 16 \mu\text{m}$ cross-section) PDMS microchannels (squares) and NOA 81 microchannels (circles). (B) Evolution of the normalized flow rate of a glycerin and water mixture after a step of pressure from 3 bars to 0 bar applied at time $t = 0$.

Resistance to deformation. We flow a Newtonian solution in a straight channel ($2 \text{ cm} \times 200 \mu\text{m} \times 16 \mu\text{m}$) and measure the relationship between the imposed flow rate Q and the measured pressure drop ΔP along the microchannel (Fig. 4A). For PDMS microchannels (squares), the ΔP vs Q curve shows non linear variations for pressures higher than a few hundreds of millibars. The hydrodynamic resistance (*i.e.* the slope of the ΔP vs Q curve) decreases as the pressure increases due to the elastic deformation of the channel. On the contrary, with the μPS channel (circles), the pressure increases linearly with the flow rate, even for pressures as high as 15 bars, as expected for a Newtonian fluid in a channel with a constant cross section.

Relaxation dynamic. To study the transient response of the device, we impose a constant pressure flow in the same microchannel filled with a glycerine–water solution (shear viscosity: $50 \text{ mPa}\cdot\text{s}$) containing fluorescent tracers (Fluospheres 500 nm, Molecular Probes). The pressure difference is dropped from 3 bars to 0 bar. PIV measurements are performed to measure the temporal evolution of the flow rate.²¹ The flow in the μPS device stops after a few tens of milliseconds, whereas this

relaxation time is three orders of magnitude higher in the PDMS channel (Fig. 4B).

These two experiments demonstrate that μPS circuits are efficient tools for the study of high pressure flows. Thanks to μPS devices, we have been able to investigate the rheology of semi-dilute micellar fluids with zero shear viscosity of several tens of $\text{Pa}\cdot\text{s}$.¹¹

3D microfluidic circuits and flow patterning

An important drawback of microfluidic devices made using standard lithographic methods is their intrinsic planar geometry. This strongly limits the architectural and the functional complexity of the fluidic circuits. For instance, the crossing of independent channels cannot be achieved in a single layer circuit. So far, only very few methods have been proposed to microfabricate 3D fluidic networks.^{14,22,23} The strategies proposed to assemble and connect multilayer devices are often time consuming and technically involved. As a consequence, most of the microfluidic applications are restrained to single layer devices (or of stacks of such devices).

Thanks to the replication technique presented here, 3D fluidic circuits, including an arbitrarily high number of connections in the three directions, can be made in a few minutes. Stencil like μPS makes it possible to fabricate vias, where size and geometry are only limited by the lithographic technique used to make the primary molds. We show in Fig. 5A a simple 3D flow patterning circuit made of three μPS . A first sticker, including a single straight channel, is covered with a stencil sticker including five connection holes (Fig. 5B). A second straight channel, perpendicular to the first one, is included in the third sticker that seals the circuits. The direction of flow of the two liquids is schematized in Fig. 5C. A 1 wt% SDS (sodium dodecyl sulfate) solution (liquid A) is injected in the first straight channel. A 1 wt% SDS aqueous solution colored with solvent blue dye (Sigma Aldrich) (liquid B) is injected in the second one. One can observe on Fig. 5B that, downstream of the vias, we manage to impose an alternated pattern of liquid A and B (ABABABABABA) with only two fluid inlets and one outlet. Obviously, this simple example suggests the enormous advantage of such a 3D device for the spatiotemporal patterning of multiphase flows. More specifically, in the case of two fluid flows, the number of maxima of the solute concentrations in 2D circuits scales with the number of inlets. Conversely, using the same strategy as in

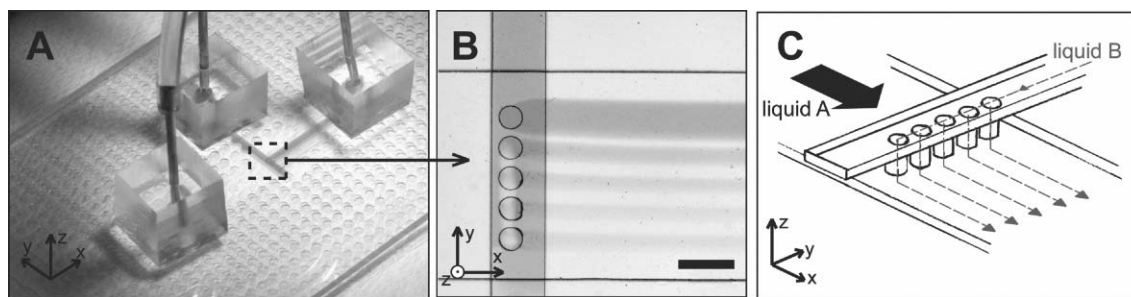


Fig. 5 (A) Picture of a 3D microfluidic device made of 3 stacked μPS . This device has 2 inlets and one outlet. It allows for generating complex flow patterns. (B) Complex stream of blue dye in water solution generated by the 3D microfluidic device. Scale bar 500 μm . (C) Schematic of the 3D device. The stream of dye is represented by dashed arrows.

Fig. 5, only two inlets are required, whatever the complexity of the imposed patterns. A detailed discussion of the spatiotemporal flow patterns obtained with three layer devices will be presented elsewhere.²⁴

Conclusions

We have presented a fast and simple fabrication method to make both 2D and 3D fluidic networks by stacking micro-patterned stickers. This method offers the opportunity to make high performance devices in a few minutes. We show that a wide range of physical and chemical properties of the μ PS enable numerous novel fast prototyping applications. The range of accessible working pressure, combined with the high spatial resolution of the μ PS, should extend the range of microfluidic applications in separation techniques²⁵ and complex fluid rheology.¹¹ Furthermore, the μ PS can be stuck in aqueous media, which offers an interesting perspective in flow patterning for cell and molecular biology.²⁶

Experimental

Hydrophilic flow focusing device (Fig. 3A)

This device has been made by sealing a microfluidic sticker with a glass slide. The sticker is made of NOA 81. A ~ 5 mm thick PDMS mold is made by the replica molding of a primary master obtained by photolithography.³ A ~ 100 μ l drop of NOA 81 is sandwiched between the PDMS mold and a flat PDMS sheet. The liquid is then cured with a collimated UV light source (Hamamatsu LC8), exposure time: 15 s, exposure power: 25 mW cm⁻². The mold is then removed and the plastic sticker is pressed by hand against the glass surface in which connection holes have been made with a sand blaster. A second UV exposure (60 s, 25 mW cm⁻²) permanently seals the device. The fluid is then injected by connecting plastic tubing (Tygon S54HL) to plastic connectors (upchurch nanoport N-333) aligned with the holes on the glass substrate. The drop emitter shown in Fig. 3 has been fabricated in less than 10 min.

Hydrophobic flow focusing device (Fig. 3B)

A ~ 100 μ l drop of TMPTA and 1 wt% of photo initiator (Darocur 1173, Sigma Aldrich) is sandwiched between the same PDMS stamp as above and a flat PDMS sheet. The liquid is cured after a 30 s UV exposure (exposure power: 12.5 mW cm⁻²). The PDMS mold is then removed and the plastic sticker is pressed by hand against a flat plastic substrate (petri dish) covered by a thin film of the same polymer. This film is prepared by pressing a drop of monomer with a flat PDMS surface. The resulting film is photopolymerized (exposure time: 30 s, exposure power: 12.5 mW cm⁻²). A second UV exposure (exposure time: 60 s, exposure power: 25 mW cm⁻²) permanently seals the device. For this device, the four walls of the channels are made of the same hydrophobic material and thus have identical chemical properties. Since the TMPTA has a low shear viscosity, the thickness of the residual layer is very thin. To strengthen the device, a second plastic substrate has been bound to the plastic sticker after the sealing step. The same tubing and connector are used to inject the

liquid and connection holes have been drilled in the plastic substrate prior to sealing.

Pressure resistant device (Fig. 4)

We use a quite similar protocol to make this straight microchannel. Here, a drop of NOA 81 is imprinted directly on a glass slide (15 s, 25 mW cm⁻²). After removing the PDMS mold, the channel is sealed with a glass cover slip pressed by hand on the sticker, in order to obtain a perfectly rigid chip (made of one glass slide and one glass cover slip sandwiching a plastic sticker). To improve the adhesion to the glass surfaces, the second UV illumination (60 s, 25 mW cm⁻²) is followed by a heating of the whole system during 1 h at 150 °C. Note that if the connection holes are to be on the glass slide, the residual layer of adhesive polymer must be punched through with a needle after the first UV curing and before the device is sealed.

3D microfluidic circuit (Fig. 5)

The 3D microfluidic circuit presented in Fig. 5 is composed of 3 different μ PS. One μ PS is fabricated for each of the two rectangular channels. For the large rectangular channel (2 mm wide, 50 μ m high and 20 mm long), a ~ 5 mm thick PDMS mold is made by the replica molding of a primary master obtained by photolithography.³ A ~ 100 μ l drop of NOA 81 is sandwiched between the PDMS mold and a glass microscope slide, in which 3 connection holes have been made with a sand blaster. The liquid polymer is then cured with a collimated UV light source (Hamamatsu LC8), exposure time: 15 s, exposure power: 25 mW cm⁻². The mold is removed and the connecting holes are re-punched with a gauge 21 needle. The second channel (500 μ m wide, 50 μ m high and 10 mm long) is fabricated the same way on a clean 22 \times 32 mm #1 cover slip (ESCO, USA) without holes. The mold is removed. Eventually, a stencil like μ PS containing the 5 cylindrical (diameter 200 μ m) vias connecting the two perpendicular channels is fabricated: a PDMS mold decorated with a dense network of 600 μ m diameters pillars and 5 cylinders (for the vias) is placed over a flat sheet of PDMS. The gap between the mold and the flat PDMS sheet is filled by capillarity with a ~ 100 μ l drop of TMPTA and 1 wt% of photo initiator (Darocur 1173, Sigma Aldrich). The liquid is cured after a 30 s UV exposure (exposure power: 12.5 mW cm⁻²) (Fig. 2B). The flat PDMS sheet is removed and the stencil like μ PS is aligned and pressed with hand against the second channel. The mold of the stencil μ PS is removed, as shown on Fig. 2C (right). The two stacked μ PS are then aligned and pressed by hand against the first channel. The entire stack is sealed with a collimated UV light source (Hamamatsu LC8), exposure time: 60 s, exposure power: 25 mW cm⁻². Three pierced cubes of PDMS (5 mm \times 5 mm \times 5 mm) are plasma sealed on each hole of the microscope slide for macroscopic fluidic connections (Fig. 5A).

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