

A rapid prototyping technique for the fabrication of solvent-resistant structures

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Received 28 June 2003, in final form 10 September 2003

Published 14 October 2003

Online at stacks.iop.org/JMM/14/153 (DOI: 10.1088/0960-1317/14/1/021)

Abstract

We demonstrate a rapid prototyping technique for the fabrication of solvent-resistant channels up to and exceeding one millimeter in height. The fabrication of channels with such dimensions by conventional lithography would be both challenging and time consuming. Furthermore, we show that this technology can be used to fabricate channels with a depth that varies linearly with distance. This technique requires only a long-wavelength ultraviolet source, a mask made by a desktop printer and a commercially available optical adhesive. We demonstrate two lithographic methods: one that fabricates channels sealed between glass plates (close-faced) and one that fabricates structures on a single plate (open-faced). The latter is fully compatible with silicon replication techniques to make fluid handling devices.

1. Introduction

There is a large demand for rapid prototyping fabrication techniques that enable design, fabrication and testing of a fluid handling device in a time span of hours such that several iterations can be evaluated in one day [1]. Toward this end, there has been success using elastomer-based molding techniques for aqueous applications [2–9]. The process typically starts with a flat substrate, such as a silicon wafer or a glass plate onto which a resist is coated and patterned using conventional photolithography. The pattern is transferred onto the substrate by various etching processes or used as is, depending upon the resilience of the resist. A siloxane-based elastomer such as polydimethylsiloxane (PDMS) is cast on the substrate, resulting in an inverse mold (or negative) of this pattern. PDMS offers many advantages for fluidics, such as transparency, flexibility, strong adhesive properties, low cost and some degree of durability. However, PDMS-based fluidics is incompatible with most common organic solvents (e.g., toluene) due to its strong tendency to absorb and swell. Additionally, after removal of the PDMS mold from the master, re-adhesion of the mold to a chosen substrate can be

problematic. Hence in recent years researchers have begun to broaden their search for additional fabrication processes that yield fluidic devices without these shortcomings. Beebe and co-workers have recently addressed some of these issues with a clever lithography process, denoted ultra rapid prototyping [10]. This process fabricates fluid handling channels between two glass plates separated by a 250 μm thick gasket. An epoxy-like resin is injected between two plates and patterned by contact photolithography. Washing away the uncured resin leaves well-patterned fluidic channels sealed between glass plates. Commercially available glass plates can be obtained with predrilled holes, greatly simplifying fluid connections to an external device. While this modification of standard patterning methodologies eliminates difficulties with sample sealing, channels fabricated in this manner do not provide sufficient solvent resistance to be suitable for the broad range of solvents used, for example in polymer formulations.

Our development of an alternative fluid handling scheme was largely inspired by this work. We built upon this technique to fabricate fluidic channels with a thiolene-based optical adhesive. Unlike the resin used by Beebe and co-workers, this adhesive is resistant to commonly used solvents for polymer solutions, such as toluene, tetrahydrofuran and ethyl alcohol. Channels can be easily fabricated that are much deeper (up to several millimeters) than is possible without

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resorting to difficult and time-consuming anisotropic plasma etching processes [11]. The fabrication of both close- or open-faced channels can be achieved with this technology. Close-faced channels are fabricated between two glass plates while open-faced channels are fabricated as a relief structure on one plate, the latter being completely compatible with common PDMS molding technologies. The great depth of these channels could be particularly interesting for preparation of bioreactor channels for the growth of cells seeded onto a scaffold [12, 13]. These dimensions are also ideal for investigations of the stability of emulsions and colloidal dispersions under flow, a topic of current research [14]. While micro-fluidic applications have received a great deal of academic and commercial attention in recent years, lab-on-a-chip applications can often be achieved with fluid handling capabilities on the millimeter length scale. Channels of these dimensions can also be more suitable for manipulation of fluids with higher viscosities than those typically accessed in micron scale fluidic systems [15].

2. Methods

A series of thiolene-based optical adhesives (Adhesive no 61, no 68, no 81, Norland Products) were used as purchased³. These adhesives function as a negative resist in the parlance of conventional lithography. While all varieties produced satisfactory results, no 81 was preferred due its faster curing rate. The adhesive was typically patterned onto glass plates of one millimeter thickness (Corning Microslides, 75 mm × 50 mm plain, Model no 2947). Curing was performed under a fume hood and care was taken to avoid breathing the adhesive vapors during sample preparation. The glass plates were separated by a well-defined spacing (typically 600 μm thick silicon wafer pieces, Wafer World). PDMS (Sylgard 184, Dow Corning) was used as received with a 10:1 mass ratio of base to curing agent. The prepolymer mixture was poured into the casting basin and allowed to degas and pre-cure overnight (or under vacuum for about 1 h). The materials were cured at 75 °C for one or more hours in a forced-air oven.

Three illumination configurations were tested: a Thermo Oriel (no 87532–1000) flood illumination system optimized for deep ultraviolet (UV, 220–265 nm), the same system optimized for long UV (350–450 nm, requiring Thermo Oriel mirrors no 80112 and 80512) and a Spectroline SB-100P flood lamp (Spectronics) optimized for 365 nm. All three illumination configurations produced satisfactory results. While the manufacturer of the optical adhesive reports that optimal curing is obtained with longer wavelength light (365 nm), we also obtained good results with shorter wavelengths, due to either the sensitivity of the adhesive or to a small amount of long wavelength light produced by the illumination system. However, given the reduced transmission of conventional glass for short UV wavelengths, higher UV doses are required to produce similar results. Exposure times were typically of several minutes

³ Certain commercial equipment, instruments or materials are identified in this paper in order to specify the experimental procedure adequately. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology, nor is it intended to imply that the materials or equipment identified are necessarily the best available for the purpose.

with the deep UV Thermo Oriel configuration for samples of thickness 600 μm or less. For long wavelength UV exposures we measured light intensities with a Spectroline DRC-100X digital radiometer with a DIX-365A UV-A sensor (Spectronics) which is sensitive to wavelengths ranging from 320 to 400 nm and covers an intensity window of 0 to 20 000 $\mu\text{W cm}^{-2}$ with 10 $\mu\text{W cm}^{-2}$ resolution. We found that typically the standard uncertainty in the dose measurements (dose = light intensity × exposure time, in J cm^{-2}) is less than 5%. When using the collimated Thermo Oriel flood illumination system the sample was typically placed about 5 cm from the lens. For the Spectroline flood lamp, the sample was typically placed about 50 cm away from the bulb to achieve a degree of beam collimation during pre-cure. The dose was selected such that UV exposure fully cured the adhesive. Shorter lamp-to-sample distances (5 to 10 cm typically) were employed during post-cure (after washing the uncured resin) to reduce overall exposure times. A more detailed discussion of variations on the curing process of the optical adhesive will be presented in a separate publication [16].

Masks were typically designed with a graphics program and printed on transparencies (3M, model CG3300) with a 1200 dpi laser printer (Hewlett Packard, model 8000N). To fabricate channels with structures greater than one millimeter in depth, two or more copies were aligned in register to increase the optical density of the darkened regions and taped to a glass plate. Structures were characterized with optical microscopy and with a profilometer (Veeco/Dektak 8) using 0.2, 2.5 and 12.5 μm tips. In our experience, the standard uncertainty for the maximum thickness is less than 5%. The width of the structures was taken to be that at half-height and the associated uncertainty is less than 5%.

In some cases a PDMS replica of the fabricated structures was chemically bonded to a glass substrate. To achieve this, the glass was first exposed to UV light with a Jelight 342 UVO cleaner for 1 h. The distance from the glass to the UV bulb was approximately 2 cm. The PDMS replica was then immediately placed next to the glass with the patterned side exposed to the UV source and the two samples were further exposed for 90 s. Upon completion of this second step, the two samples were immediately brought in contact and then baked overnight at 75 °C. We found that this technique reliably bonded PDMS to glass or silicon as long as the two surfaces were brought together within seconds of completing the UV dose. This bonding technique is analogous to that reported by Duffy *et al* [8] but does not require an oxygen plasma.

3. Methodology 1: close-faced

We describe here a method of patterning channels of UV-curable adhesive between two glass plates, producing channels sealed to the upper and lower plates. First, a glass plate is set onto a flat work space and several milliliters of adhesive are deposited onto the plate from a syringe or poured directly (figure 1). Glass plates are used since UV cleaning did not show appreciable enhanced adhesion. Given the high viscosity of the glue (about 3.0 Pa s or 300 centipoise) it must be applied carefully to avoid introducing air bubbles. Spacers are placed on either side of the glass plate, far away from the area to be patterned. For convenience we use as spacers pieces cut from

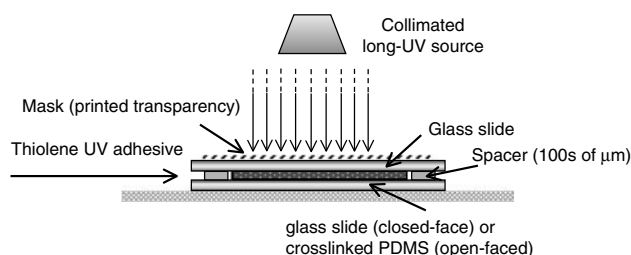


Figure 1. Cross-sectional view of the process to fabricate micro/milli-fluidic channels with either open- or close-faced methodology. In the close-faced methodology, optical adhesive is placed between two glass plates separated by spacers. A mask is placed on top and an appropriate UV exposure is performed. The adhesive is cross-linked in the exposed regions and the unexposed adhesive is washed away by injecting solvents. With the open-faced methodology, the bottommost glass plate is replaced with a sheet of cross-linked PDMS. UV exposure cross-links the adhesive and binds it to the glass plate. The glass is then peeled off of the PDMS and washed with solvents to remove unexposed adhesive.

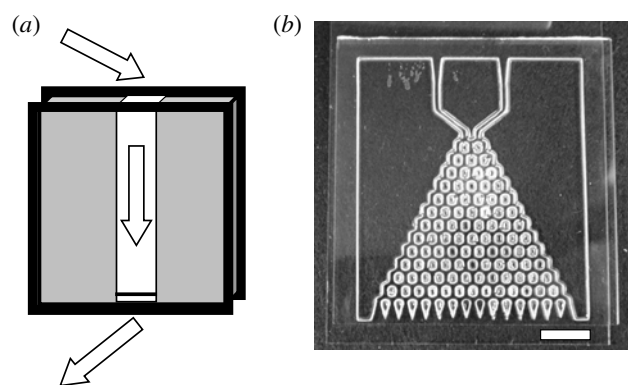


Figure 2. (a) After UV exposure with the close-faced methodology, the unexposed regions are washed away by injection with solvents. This leaves a fluidic channel (light) in a thiolene matrix (dark) which is further annealed to make it impervious to a range of solvents. (b) An example of a passive diffusion array sealed between glass plates. Width of channels is approximately $500 \mu\text{m}$ (obtained with a 365 nm UV dose of 30 mJ cm^{-2}). Bar = 1 cm .

silicon wafers, thereby controlling the thickness to typically $600 \mu\text{m}$. A second glass plate is lowered at a shallow angle onto the liquid adhesive and gently placed on top of the first. The adhesive is allowed to flow and stabilize. A third glass plate, onto which the transparency mask is attached, is then applied on top with the mask side down. This plate (and mask) is typically larger than the other plates so as to shield stray light from reaching the sides of the glue. An exposure is performed to cross-link regions of the adhesive not shielded by the mask. Subsequently, the mask is removed and a syringe is used to wash out the unexposed adhesive regions by injecting with solvent (figure 2(a)). Ethanol is typically used first to wash out the majority of the uncured adhesive, and acetone is used sparingly as a final step. After a satisfactory pattern is obtained, the entire sample is flood exposed with a UV dose 20 to 30 times that of the pre-cure. The post-cure has several beneficial effects: increased solvent resistance, increased adhesion to the glass and increased mechanical stability via a higher cross-link density. The sample is finally baked for 12 h at $50 \text{ }^\circ\text{C}$ to improve the adhesion of the UV-curable adhesive with the glass slide. The overall process

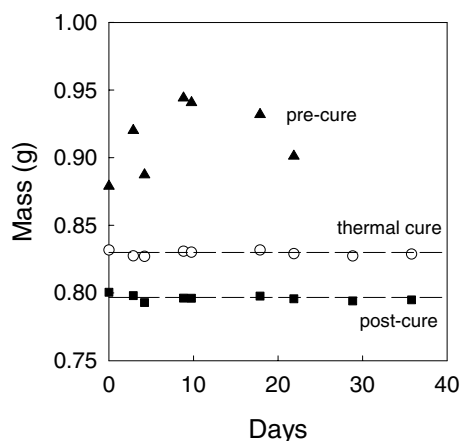


Figure 3. Weight variation of (open face) adhesive stamps upon prolonged immersion in toluene. Three UV and thermal treatments were investigated: (\blacktriangle) pre-cure only, (\blacksquare) pre- and post-cure and (\circ) pre-, post- and thermal cure (details in the text).

is therefore divided into three steps: (i) pre-cure (typically tens to hundreds of seconds), (ii) post-cure (hundreds of seconds) and (iii) thermal cure (specimen ageing overnight which extends the device's lifetime). Figure 2(b) shows an example that demonstrates the effectiveness of this patterning technique. A passive diffusion array is shown consisting of two inlets (image top) that are used to feed fluids past a series of semi-circular obstacles such that the number of obstacles increases linearly with distance. At the bottom of the structure there are 15 outlets from which 15 fluid compositions can originate.

Structures fabricated in this manner have been demonstrated to be impervious to solvents such as toluene during prolonged immersion. The solvent resistance of the devices was investigated by immersion in toluene for several weeks while monitoring the weight. Using the open-faced methodology (described in the following section), which offers large surface exposure, three stamps of adhesive (with dimensions $2.0 \text{ cm} \times 2.0 \text{ cm} \times 1.1 \text{ mm}$) were fabricated on a glass slide. Each specimen received a different treatment: (i) only pre-cure, (ii) pre- and post-cure and (iii) pre-, post- and thermal cure. Thorough post-cure (100 times the pre-cure UV dose) and thermal cure (two days at $50 \text{ }^\circ\text{C}$) were carried out. The mass of the specimens was monitored after the removal of toluene under vacuum at $120 \text{ }^\circ\text{C}$ (above the boiling point of the solvent). Results are shown in figure 3. Both the fully cured and the post-UV cured specimens show no resolvable weight change after several weeks of immersion, while the pre-cured only specimen (with incomplete network cross-linking) increased mass due to solvent uptake and delaminated after three weeks. Similar results have been found with methanol, hexane and methyl ethyl ketone, demonstrating the resilience of this patterning technology to solvents commonly used in polymer formulations.

4. Methodology 2: open-faced

We describe here a method of fabricating patterns in an adhesive which is coated onto a single glass plate. As the fabricated structures are on an open glass plate they are readily

replicable by PDMS mimicking technologies. This is a simple modification of methodology 1, where the bottom plate is now a large (20 cm × 30 cm) tray in which a one millimeter deep layer of PDMS has been cast (figure 1). Because the adhesive does not bind to cured PDMS, this surface makes an ideal work space for manipulating the adhesive and patterning into channels. Several milliliters of UV-curable adhesive were applied to the PDMS work space and two silicon wafer spacers were placed on either side. Alternatively, a preformed gasket of PDMS was used to fabricate deeper structures. A middle glass plate and a top glass plate (with attached mask) were applied as before. Note that the mask is slightly larger than the area to be exposed so as to minimize light impinging on the adhesive from the sides. After limited exposure, the top glass plate with attached mask was removed and set aside. Next, the middle glass plate was slowly removed from the PDMS slab, leaving the majority of the uncured adhesive on the slab while the cured adhesive adhered to the glass plate. The uncured adhesive can be reused by re-centering the uncrosslinked fluid on the PDMS slab with a straight edge⁴. The glass plate was carefully rinsed with ethanol, acetone and ethanol again to remove uncured adhesive while minimizing the removal of pre-cured structures. After rinsing, the sample was post-cured as described above and the glass plate was annealed at 50 °C overnight as a final step to optimize the bonding of the adhesive to the substrate. Figure 4 shows several examples of structures fabricated by this technique. Panel (a) shows sets of parallel lines and panel (b) shows an array of solvent-resistant wells. Panels (c)–(d) show relief structures in the form of diffusion arrays that were fabricated employing the same design used to fabricate the structures shown in figure 2(b). The two masks are the optical negative of each other, resulting in topographically inverse structures. For example, panel (c) consists of a relief structure of hexagonally arranged lines (darker) whereas panel (d) consists of the same, but hexagonally arranged channels (darker). Panel (e) shows a magnified view of four channels that were fabricated with the open-faced methodology in a separate sample. We found that we could reliably fabricate structures with line widths as small as 150 μm. The periodic protrusions of these channels are designed to enhance mixing in polymer formulations by providing a torturous path. The ultimate lateral resolution of this method is limited by the collimation of the UV light, printing resolution and washing efficiency, as we investigate in the following section.

The surface roughness of fabricated structures was investigated with a Dektak 8 (Veeco) profilometer. An arithmetic average roughness (R_a) of 2.5 μm was measured where the lower limit of the confidence interval was 1 μm and the upper limit was 5 μm. Similar values for roughness were measured on the side wall as well as the top of the fabricated structures. This roughness level is intrinsic to the cross-linking of the Norland 81 adhesive and is probably a signature of the propagation of the cross-linking front during UV exposure. The measured roughness level was largely insensitive to the line height or feature size.

⁴ After more than five iterations, however, the performance of the re-used adhesive suffers from deleterious effects, probably due to partial cross-linking of the unexposed regions due to insufficient optical density of the darkened portions of the mask.

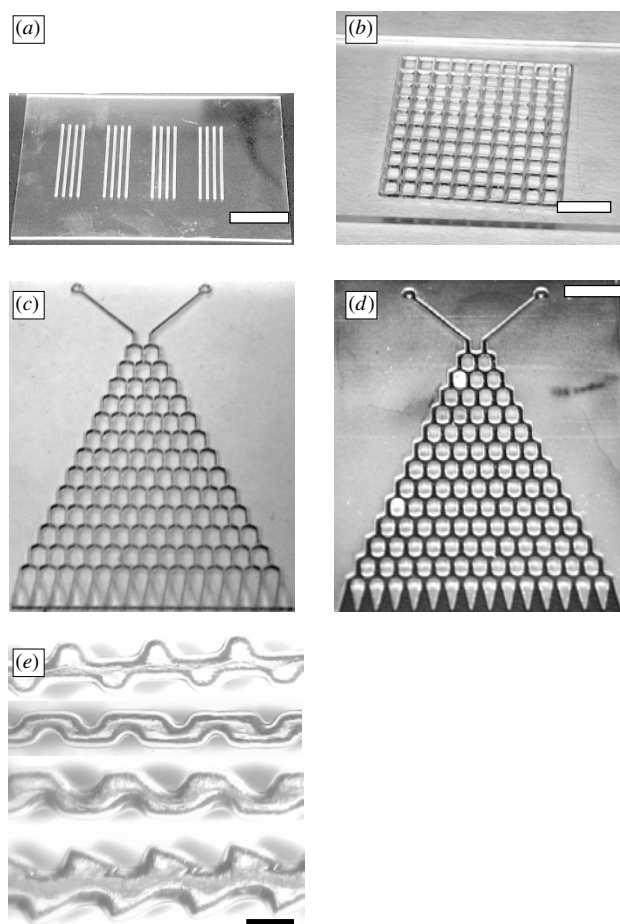


Figure 4. Several examples of fabricated structures using open-faced methodology. (a) Millimeter wide and 500 μm tall lines of adhesive on a glass slide. Bar = 1 cm. (b) Array of 10 × 10 square wells (with 300 μm wide walls) fabricated with open-faced methodology. The height of the wells can be easily tuned with UV dose administered; features shown here are 400 μm tall. (c)–(d) Two open-faced diffusion arrays made with the mask (and its inverse) used in figure 2. The darker regions of panel (c) correspond to relief structures on a glass slide, the darker regions in panel (d) correspond to channels in a cross-linked adhesive matrix. The structures have lateral dimensions of 600 μm and a height or depth of 500 μm (obtained with a 365 nm UV dose of 25.3 mJ cm⁻²). (e) Four channels with designs to enhance mixing that were fabricated with this methodology. The smallest channels which could be reliably fabricated had feature sizes of the order of 100 μm. Bar = 300 μm.

5. Spatial resolution

The spatial resolution of the contact lithography described in this work was tested using a standard United States Air Force (USAF) 1951 negative test pattern (chromium on glass with an optical density greater than three purchased from Edmund Optics)⁵. The target pattern was impressed on a 100 μm thick glass slide (cleaned with UV ozone for 15 min) using a 50.8 cm lamp-to-sample distance and a 60 s exposure (10.6 mJ cm⁻² UV dose), resulting in a 250 μm tall features

⁵ The 1951 USAF standard test pattern consists of groups of bar patterns whose spatial frequency increases as the sixth root of two, i.e. $2^{n/6}$, with n ranging from 0 (or -12) to 47. Optical density is expressed by $OD = \log(1/T)$ where T is the transmittance.

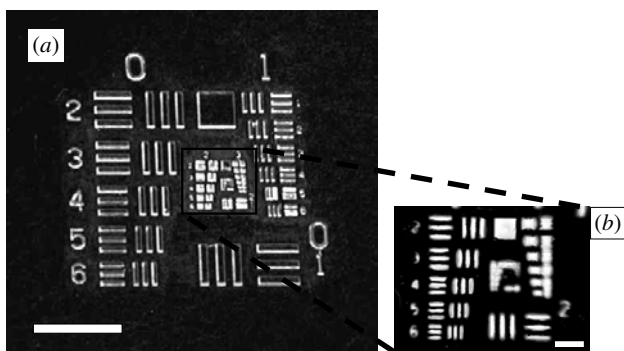


Figure 5. (a) Pattern obtained upon curing the optical adhesive using the negative standard USAF 1951 test pattern as a mask (cured with 10.6 mJ cm^{-2} UV dose). The smallest dimensions reproduced were $70 \mu\text{m}$ wide bars (separated by $70 \mu\text{m}$ intervals), given the impediments in washing uncured resin in the interstices of higher frequency features (Bar = 5 mm). (b) Micrograph of the central region of the pattern (groups 2 and below). The larger bars on the top left are satisfactorily patterned with a spacing of $125 \mu\text{m}$, but the smaller structures on the top right are below the resolution limit of this technique. Bar = 1 mm.

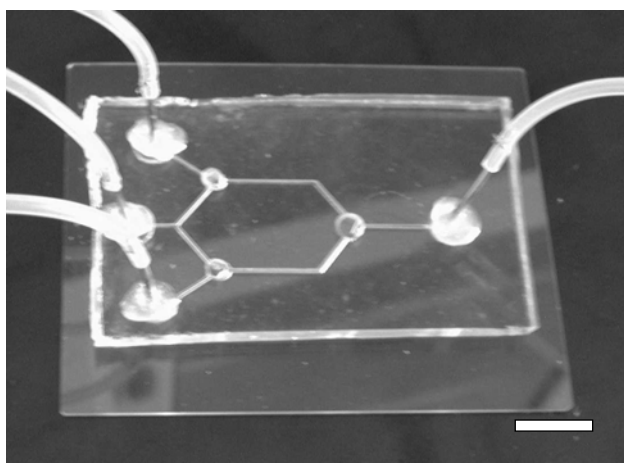


Figure 6. PDMS replica of relief structure fabricated with open-faced methodology. A multicomponent mixing device is fabricated by attaching flexible tubing and inserting micro-stirring bars in channel junctions. Bar = 1 cm.

(figure 5). The uncured resin was carefully washed away using a mixture of ethanol and acetone (mass fractions of 0.6 and 0.4, respectively) and the test pattern was post UV and heat cured as described above. With this enhanced collimation and gentle rinse, we were able to reproduce features with a frequency down to $7.13 \text{ cycles mm}^{-1}$ ($n = 17$, group +2, element 6), corresponding to $70 \mu\text{m}$ wide lines. Narrower line widths were obtained in isolated features. We found that the viscosity of the uncured resin is the largest limitation in the washing of high-frequency (or ‘crowded’) patterns. Isolated lines do not suffer from this difficulty, enabling us to make line widths as narrow as $50 \mu\text{m}$.

6. Fluid handling device

To demonstrate that structures made with the open-faced methodology can be replicated for PDMS-based fluidic apparatus, we show a simple device in figure 6 with three inputs, one output, four T-junctions and three mixing stages

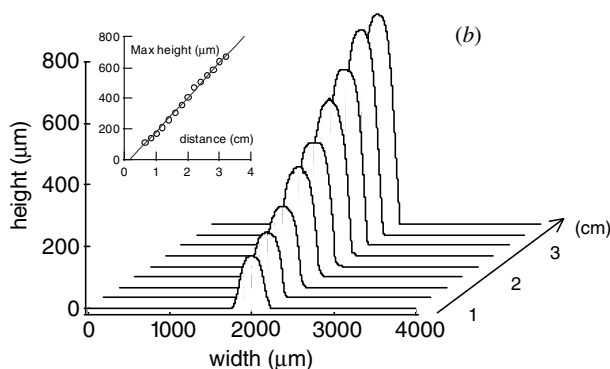
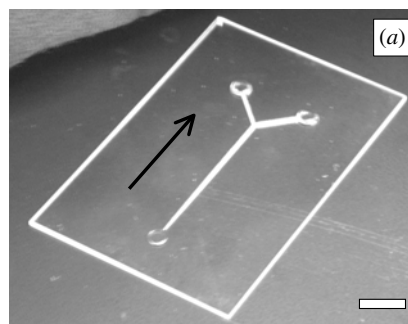


Figure 7. Example of structure with height gradient. The open-faced methodology was used with a spacer (thickness of 1 mm) only on one side to make a wedge of optical adhesive. (a) Optical image of T-junction relief structure on glass plate. Distance on graphs in panel (b) coincides with arrow on image. Bar = 1 cm. (b) Profilometer scans as a function of distance along structure. The rounding of the structure edge is in large part a limitation of the scanning ability of the profilometer. Inset shows maximum height of structure along longer portion (ranging from $100 \mu\text{m}$ to $700 \mu\text{m}$). The line is a linear fit to the data. The height of the taller circular pads was $850 \mu\text{m}$ (UV dose of 140 mJ cm^{-2}).

(where micro-stirring bars were placed). Structures were fabricated with the open-faced methodology and replicated with PDMS. After curing the PDMS, it was chemically bonded to the glass as described earlier. Fittings were attached and the integrity of the seal was demonstrated by pumping aqueous fluids through the device. While the focus of this paper has been on the patterning technology, a subsequent paper will describe the use of these structures in devices in studying fluid properties [16].

7. Height or depth gradients

While conventional photolithography is well suited for the fabrication of structures with uniform height or depth, it is particularly challenging to fabricate structures with height or depth gradients. This limitation stems from the spin coating process, which strives to apply the resist as uniformly as possible. Since our technique does not rely upon spin coating, we suffer from no such limitations. In contrast to spin coating, the adhesive fluid will adopt the shape of any volume that confines it, whether between parallel glass plates (as shown up to now) or between plates that are not coplanar, the latter forming a wedge. Such confinements are easily created by following either methodology but with spacers of unequal thicknesses. We show an example of this in figure 7 where

the relief structure of a T-junction has been fabricated using the open-faced methodology. The longest section has a height gradient ranging from 100 μm to 700 μm and the circular pads reach heights of 850 μm .

While spacers of unequal thickness produce structures with simple one-dimensional height gradients, this technique can be further extended by the pre-molding of geometric structures into the PDMS casting basin. For example, if the PDMS were cast to be slightly convex, the application of methodology 2 centered on the symmetry point would result in relief structures which were thinner in the middle than at the edge. The only limitation then to this technique is the slight bowing of the glass by capillary forces (the adhesive wets clean glass). We find that the millimeter thick glass slides minimize this to an acceptable level whereas cover-slips (about 170 μm thick) experience an unacceptably high degree of bowing.

8. Conclusions

We have demonstrated a rapid prototyping technique for the fabrication of fluidic channels with an inexpensive, UV-curable adhesive. This technique is significant as it can produce millimeter-deep channels (or structures) without the need for expensive masks or deep etching techniques. Two fabrication methodologies were demonstrated—close-faced, which fabricates channels that are sealed between glass plates, and open-faced, which fabricates structures on a single piece of glass. The latter is amenable to PDMS replication techniques. The resist material was shown to be resistant to a variety of solvents, including toluene. This patterning technique has a lateral resolution of approximately 70 μm , but we emphasize that isolated structures can be fabricated with smaller dimensions. Furthermore, we demonstrated the fabrication of a structure with a height gradient of 600 μm over three centimeters. We intend to use this rapid prototyping technology in the development of measurement tools for the study of polymeric and colloidal formulations.

Acknowledgments

This work was partially inspired by a presentation of Professor David Beebe at NIST. We thank him and C Khoury for useful advice. We thank Dr Scott Kennedy for useful conversations

and the mask used to fabricate the structures shown in figure 2. CH and CMS acknowledge support from the NIST National Research Council Postdoctoral Fellowship Program.

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