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A new fabrication process for ultra-thick microfluidic microstructures utilizing SU-8 photoresist

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Abstract
In this paper we describe a new process for fabricating ultra-thick microfluidic devices utilizing SU-8 50 negative photoresist (PR) by standard UV lithography. Instead of using a conventional spin coater, a simple 'constant-volume-injection' method is used to create a thick SU-8 PR film up to 1.5 mm with a single coating. The SU-8 PR is self-planarized during the modified soft-baking process and forms a highly-uniform surface without any edge bead effect, which commonly occurs while using a spin coater. Photomasks can be in close contact with the PR and a better lithographic image can be generated. Experimental data show that the average thickness is 494.32 ± 17.13 μm for a 500 μm thick film ($n = 7$) and the uniformity is less than 3.1% over a 10×10 cm^2 area. In this study, the temperatures for the soft-baking process and post-exposure baking are 120 °C and 60 °C, respectively. These proved to be capable of reducing the processing time and of obtaining a better pattern definition of the SU-8 structures. We also report on an innovative photomask design for fabricating ultra-deep trenches, which prevents the structures from cracking and distorting during developing and hard-baking processes. In this paper, two microfluidic structures have been demonstrated using the developed novel methods, including a micronozzle for thruster applications and a microfluidic device with micropost arrays for bioanalytical applications.

(Some figures in this article are in colour only in the electronic version)

Nomenclature

GBL	γ-butyrolactone
HF	hydrofluoric acid
LIGA	lithographie, galvanoformung und abformung
PAC	photoactive compounds
PEB	post-exposure bake
PGMEA	propylene glycol methyl ether acetate
PR	photoresist
SEM	scanning electron microscope
TEC	thermal expansion coefficient
UV	ultraviolet
T_g	glass transition temperature

1. Introduction

There is a need to fabricate ultra-deep trenches or ultra-thick structures since these are critical for the performance of microfluidic devices (micro mixers, micro electrophoresis chips for bioanalysis, and micronozzles for generating thrust forces, etc). Traditionally, x-ray lithography is a major technique for fabricating ultra-thick microstructures with millimeter-sized thickness. Very high aspect-ratio microstructures with vertical sidewalls and high resolution have been reported using the LIGA (lithographie, galvanoformung und abformung) technique [1–4]. However, the need for a synchrotron radiation source limits its application for researchers and the process is inherently time-consuming and expensive. Since the EPON SU-8 epoxy-resin negative photoresist (PR) was reported, the fabrication of high-aspect-ratio microstructures by standard UV lithography has become popular in the microelectromechanical systems (MEMS) community. The applications of SU-8 for MEMS devices have attracted great interest [5–8]. Due to the good chemical and mechanical properties of the polymerized SU-8, many novel microfluidic devices have been demonstrated [9–11]. However, the fabrication of high-aspect-ratio microchannels with millimeter depth has rarely been reported and is still under extensive investigation.

The challenges for fabricating an ultra-thick microstructure include PR coating, baking process, PR developing efficiency and tremendous residual stress formed after the curing of the SU-8. Lorenz *et al* [12] have reported that a microstructure with a height of 1.2 mm could be formed by double coating the SU-8 PR layers [12]. However, multi-coating is a time-consuming process. The surface flatness is also a critical issue. The solvent content in subsequent coating layers may be different, resulting in a much more complicated lithography process. Single coating of an SU-8 film thicker than 1 mm is not practical using a conventional spin coater. The high viscosity of the SU-8 will be so prominent as to cause a poor PR coverage for spin coating at a low speed. Another problem caused by spin coating is the so-called ‘edge bead effect’, which occurs for films thicker than 50 μm [17]. The re-flow of the PR also changes the thickness of the coated films during the soft-baking process. The problem becomes more serious when thicker PR layers are coated using substrates with irregular shapes. Besides, photomasks will not be completely in contact with the PR due to the existence of the edge bead. Consequently, this will cause a bad lithography image due to the air gap between the photomasks and the substrates. One possible solution for this problem is to remove the edge bead by standard edge bead removal techniques with γ-butyrolactone (GBL). Alternatively, one can fill the air gap with organic solvents [14]. However, the organic solvent will form a slippery layer and it may diffuse into the SU-8 layer, forming a fragile structure.

The baking process is another important issue for the fabrication of ultra-thick SU-8 microstructures. In previous works based on the Taguchi method, it has been reported that the soft-baking conditions of a thick SU-8 film are very critical for its lithographic quality [13, 15, 17] and it has been indicated that an improper post-exposure bake (PEB) procedure tends to distort fine-line features [17]. Normally, it takes tens of hours to bake an SU-8 film thicker than 1 mm at a standard baking temperature of 95 °C. A slow ramping baking process was introduced in order to reduce the internal stress of the thick SU-8 film [16, 18]. However, the glass transition temperature (T_g) of the unexposed SU-8 film is 55 °C. Organic molecules can freely migrate at a temperature higher than T_g, which implies that the internal stress in the SU-8 film is very small. The ramping process and the process of two-step temperature elevation are not necessary during soft baking. In addition, the temperature that activates the photoactive compounds (PAC) of the SU-8 is around 135 °C [15]. The soft-bake temperature can be increased up to 120–130 °C without risk of cross-linking the unexposed SU-8. Soft baking the SU-8 film at a higher temperature can reduce the baking time significantly. Note that care must be taken while keeping the substrate at 55 °C for a period of time during the process of decreasing the temperature in order to re-crystalline the soft-baked SU-8 film around its T_g. It is expected that the internal stress can be reduced after the crystallization.

Another problem commonly encountered during the lithography process is the distortion of the microstructures. The thermal expansion coefficient (TEC) of SU-8 is 52 ± 5.1 ppm °C^{-1} [18]. It has been noticed that unexposed SU-8 might be squeezed out of a channel due to the thermal expansion of the exposed region, resulting in a distorted geometry. The problem caused by thermal expansion becomes more serious when the exposed area is bigger. In this study, we used a temperature of 60 °C for the PEB process, which is higher than T_g of the unexposed SU-8. The unexposed region will be re-softened at 60 °C and the stress formed at the exposed/unexposed interface will be released. Likewise, the large internal stress for the polymerized SU-8 is a big problem for the fabrication of ultra-thick microstructures. Not only does internal stress distort the patterned structures but it also causes some adhesion problems or even cracking failures [12, 16, 19]. Large internal stress will also bow the substrate and collapse an ultra-deep channel. Detailed information regarding the quantity measurement of the stress induced by the polymerization of SU-8 has been reported in the literature [12]. The effect caused by the large internal stress can be alleviated either by reducing the exposed area or by generating a discrete small-sized exposed region.

In the present study we aim to develop an easy and rapid process for fabricating ultra-thick SU-8 microstructures up to millimeter depth. Instead of using multi-coated SU-8 films by conventional spin coaters, a novel ‘constant-volume-injection’ method has been used to coat an ultra-thick SU-8 film on the substrate. The standard process from the SU-8 technical data sheet may not meet the requirement of structures higher than 1 mm. A modified baking process was developed to reduce the process time and to obtain a better lithography quality. In order to overcome the adhesion and cracking problems caused by the internal stress, a new photomask design was proposed. Experimental results have shown that ultra-thick microstructures can be formed without any damage. In this study, two microfluidic structures have been demonstrated using the developed novel methods, including a micronozzle for generating thrust forces and a microfluidic device with micropost arrays for bioanalytical applications.

2. Materials

In this study, a commercially available SU-8 50 negative PR and a propylene-glycol-methyl-ether-acetate (PGMEA) developer were used (MicroChem Corp., MA, USA). Polished 1.1 mm thick soda-lime glass substrates (G-tech Optoelectronics Corp., Taiwan) were cut into pieces to fit the design of the photomasks. Prior to the lithography process,

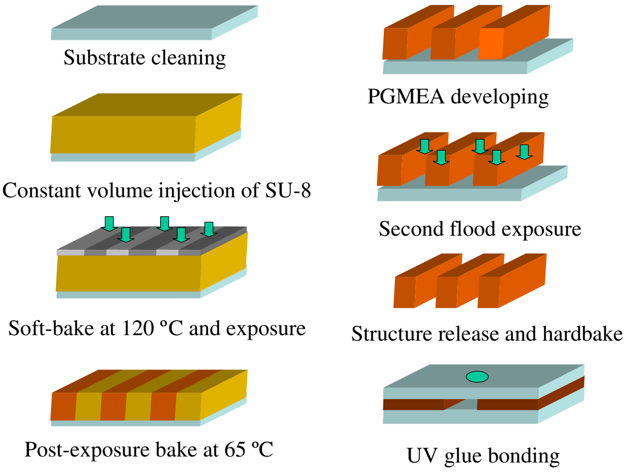


Figure 1. Simplified fabrication process of ultra-thick SU-8 micronozzles for generation of thrust forces.

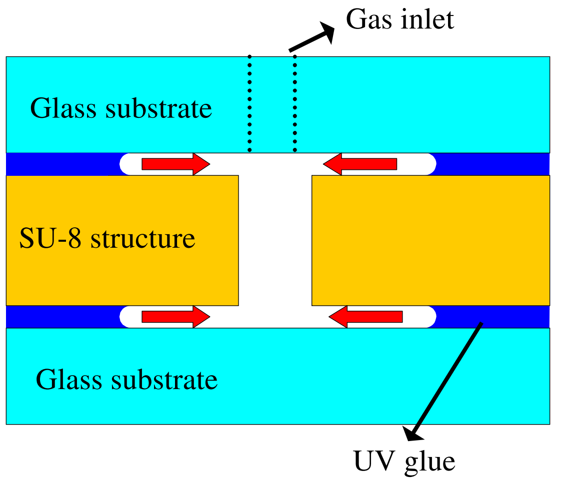


Figure 2. A schematic diagram of UV glue bonding techniques. The UV glue was attracted by the capillary force and filled the gaps between the upper/lower glass plates and SU-8 structures.

the glass substrates were cleaned in a boiling Piranha solution (H_2SO_4:$H_2O_2 = 3:1$) for 10 min. The photomasks were generated using a layout software (AutoCAD) and were printed on a transparent photofilm using a high-resolution laser plotter (10000 dots per inch). A highly transparent UV-sensitive adhesive (UV glue, 1505A, Tex Year, Taiwan) and a UV light source with 365 nm wavelength (Lightex-A20, Tex Year, Taiwan) were used for sealing the microfluidic devices.

3. Methods

The simplified process for fabricating the ultra-thick SU-8 microfluidic devices is shown in figure 1 and is described in detail as follows.

(a) *SU-8 PR coating.* In order to coat an SU-8 film with a millimeter thickness using single coating, a novel 'constant-volume-injection' method was developed. The volume of the PR was first calculated while a constant thickness of the PR was applied on a substrate with a known area. In this study, a commercial plastic 1 ml syringe with 0.01 mL resolution (Terumo Corp., Tokyo, Japan) was used to control the injected volume of the SU-8 PR. Since the solvent content of the PR is drained from the film after soft baking and the thickness of the film decreases by about 20% [20], the SU-8 was injected about 20% higher accordingly. For example, 12.5 mL of SU-8 50 PR on a $10 \times 10\ \mathrm{cm}^2$ glass substrate resulted in a 1 mm thick SU-8 film. A Teflon scraper was then used to spread the SU-8 film at a temperature of 80 °C. Note that the spread SU-8 has to cover the edge of the glass substrate to prevent the film from pulling back during the baking process. The coated SU-8 film does not overflow from the glass substrate for a film with a thickness less than 1.5 mm due to surface tension. However, for a film with a thickness greater than 1.5 mm, overflow of the SU-8 might occur and a casting barrier can be used to solve the problem.

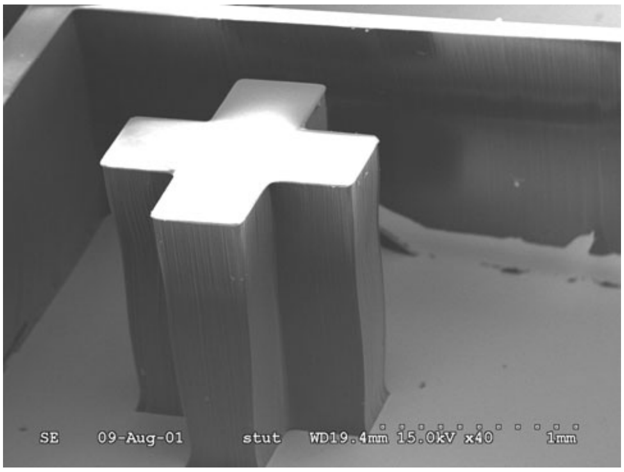
(b) *Baking process and lithography.* The unexposed SU-8 has good mobility at a temperature higher than its T_g. This explains why SU-8 is self-planarized during the soft-baking process. During the baking process, the gravity force can affect the flatness of the SU-8 film. Therefore, the hotplates used in this study were carefully adjusted to a horizontal position prior to the baking process. The baking process proceeded at 120 °C without ramping and the baking durations were 4, 8 and 12 h for films with thicknesses of 500 μm, 1 mm and 1.5 mm, respectively. The substrates were then cooled down and kept at 60 °C for 5 min. After this, the temperature was ramped down to 50 °C within 30 min. Finally, the baked substrates were placed at room temperature for another 30 min prior to UV exposure.

The UV lithography was processed using a mask aligner (OAI Corp.) and the exposure dose was 2.8 mJ $\mathrm{cm}^{-2}\ \mu\mathrm{m}^{-1}$. The PEB was carried out at 60 °C for 30 min with a 3 °C min^{-1} ramping rate starting from room temperature. The substrates were developed using pure PGMEA with gentle ultrasonic agitation and then baked at 90 °C for 10 min to remove the residual organic solvent. A high-dose second exposure (4200 mJ cm^{-2}) was then used to activate the residual photosensitive compounds for cross-linking of the SU-8. Finally, the substrates were immersed into a 20% hydrofluoric acid (HF) solution to release the SU-8 structures from the substrates. The released SU-8 structures were then hard baked at 200 °C for 10 min.

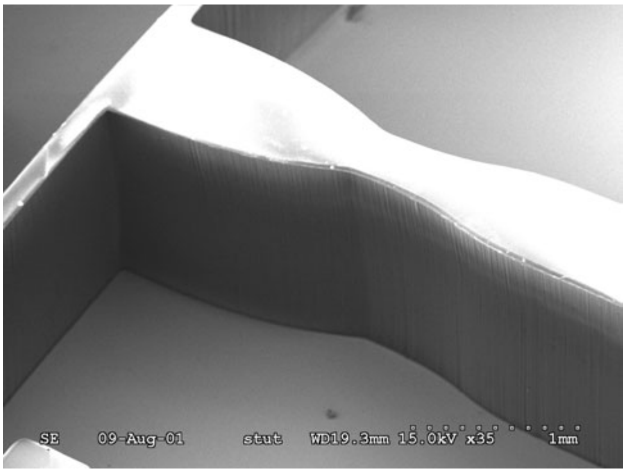
(c) *UV glue bonding technique.* Micronozzles were first fabricated using the method described in the previous section. Glass substrates with a thickness of 1.1 mm were used to seal the microfluidic devices using UV glue binding techniques. Prior to bonding, a gas inlet hole with a diameter of 700 μm was drilled mechanically using a diamond drill bit. Figure 2 shows a schematic diagram of the UV glue bonding techniques. The upper/lower glass substrates and the SU-8 micronozzle were aligned and fixed by a C-clamp. Then a small amount of UV glue was applied at the edge of the sandwiched structures. The capillary force attracted the UV glue to fill the gap between the glass plates and the micronozzle. The sandwiched structure was then exposed using a UV lamp to cure the glue.

4. Results and discussion

Figures 3(*a*) and (*b*) show the ultra-thick SU-8 microstructures fabricated by standard UV lithography with a single coating.



(*a*)



(*b*)

Figure 3. Ultra-thick SU-8 microstructures with a height of 1.5 mm. (*a*) A nearly vertical sidewall could be obtained by standard UV lithography. (*b*) An inverse image of a convergent-divergent nozzle with a minimum feature size of 100 μm and an aspect ratio of 15.

The structure height is up to 1.5 mm and the sidewall is nearly vertical. It can clearly be seen that the structure contour is well defined at both concave and convex corners. During the PR developing process, ultrasonic agitation was used such that development could be completed in a shorter time. The minimum feature size of the structures in figure 3 is 100 μm with an aspect ratio of 15. Note that there are straight-line fine features along the SU-8 structures, which seem to be a common phenomenon after SU-8 lithography using contact-mode exposure. These fine features have been found in previous works[3] [5, 10, 11] and are thought to be related to the mask problems. Figure 4 shows a SEM image of a microthruster nozzle, with a height of 1 mm and a throat width of 250 μm. For many microsystem applications such as microthrusters, micromixers and microchannels for electrophoresis separation, there is a need to fabricate ultra-deep trenches or ultra-thick structures since these are critical for the performance of the microfluidic devices. The novel

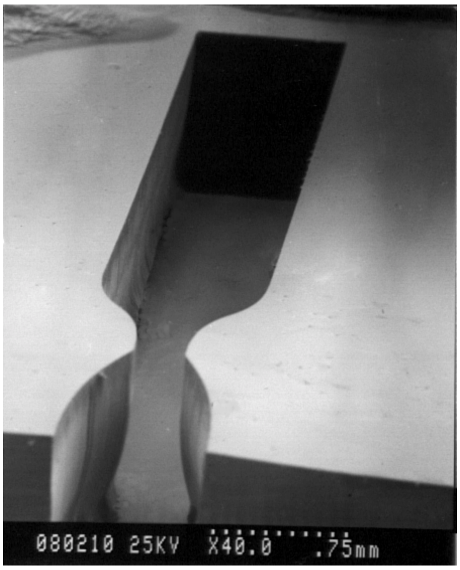


Figure 4. SEM image of a nozzle for the generation of thrust forces. The width of the throat is 250 μm and the height of the SU-8 structure is 1 mm.

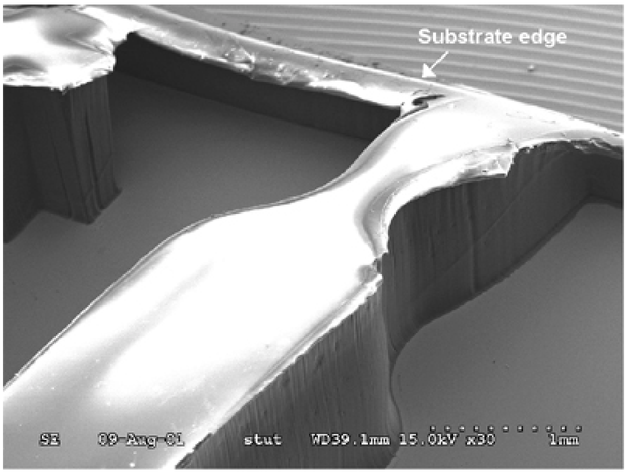


Figure 5. SEM image of an SU-8 structure near the edge of the substrate. No edge bead effect is observed.

SU-8 coating process using the constant-volume-injection method offers a simple way to fabricate microstructures with various thicknesses. In order to evaluate the reproducibility of the developed method, seven pieces of glass substrates with a dimension of 4 $\times$ 4 cm^2 were coated with 1 mL of the SU-8 50 PR. The measured average thickness of the film was 494.32 μm with a standard deviation of 17.13 μm after the lithography process. The experimental results have shown that the constant-volume-injection method can coat films repeatedly with a well-controlled thickness.

Figure 5 show a SEM image of a 1.5 mm thick SU-8 film near the edge of the glass substrate. No edge bead effect was observed. Note that the thickness of the film decreased as it approached the edge. Pattern distortion also occurred at the edge due to the incomplete contact between the photomask and the PR film. The thickness can reach the setting value within 3 mm measured from the edge.

[3] Data sheet for NATO^{TM} SU-8 negative tone PRs, Formulations 50 and 100, released by Micor Chem Corp.

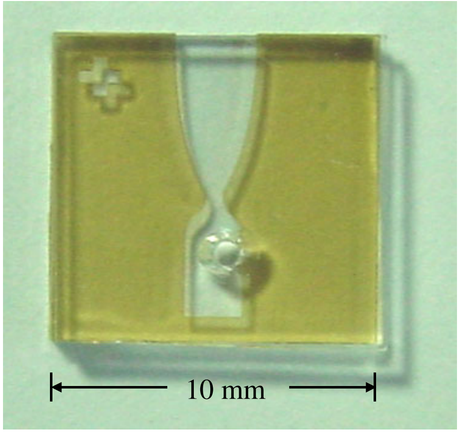


Figure 6. An SU-8 micronozzle packaged by UV glue bonding techniques. The width of the throat is 250 μm and the gas inlet via hole is 750 μm in diameter.

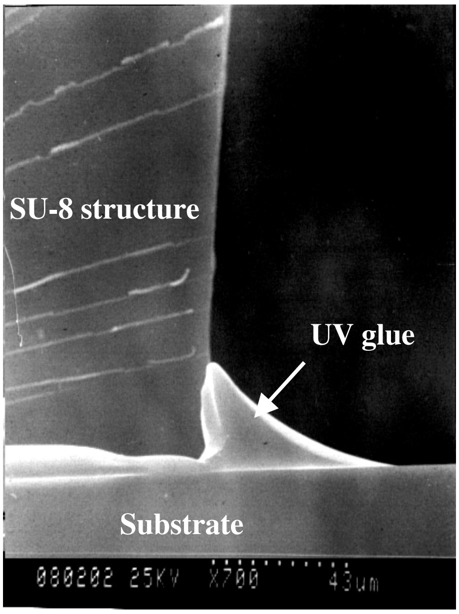


Figure 7. SEM image near the outlet of the micronozzle. The UV glue stopped at the edge of the SU-8 structure and formed a triangular structure. The base of the triangular region is about 50 μm and the height is about 40 μm.

SU-8 is in its liquid phase at a soft-bake temperature of 120 °C. Surface tension and high mobility make SU-8 self-planarized and form a film with good flatness and uniformity. We have measured the uniformity of a 500 μm thick SU-8 film over a 10×10 cm^2 substrate. The variation in thickness is less than 3.1% for twenty measured points. This indicates that the developed method is a reliable method to coat a uniform film.

Figure 6 shows a micronozzle after the packaging process using UV glue bonding techniques. The structure has a depth of 500 μm and a throat width of 250 μm. The proposed UV bonding technique in this study is a simple but reliable method to package the micronozzle with a high bonding strength. The capillary force between the glass plates and the SU-8 structure attract the UV glue to move forward until it covers the whole SU-8 structure. Figure 7 shows a SEM image near the nozzle outlet. The UV glue overflowed at the edge of the SU-8 structures and formed a triangular structure. The UV glue also completely filled the gap between the glass plates and the SU-8 structures and formed an adhesive layer (10 μm) with a high-bonding strength (figure 8).

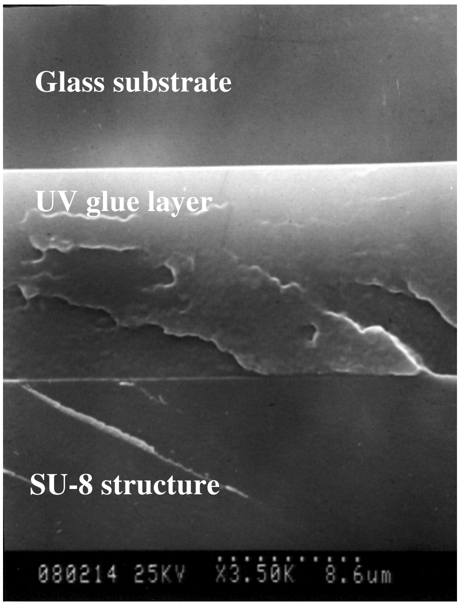
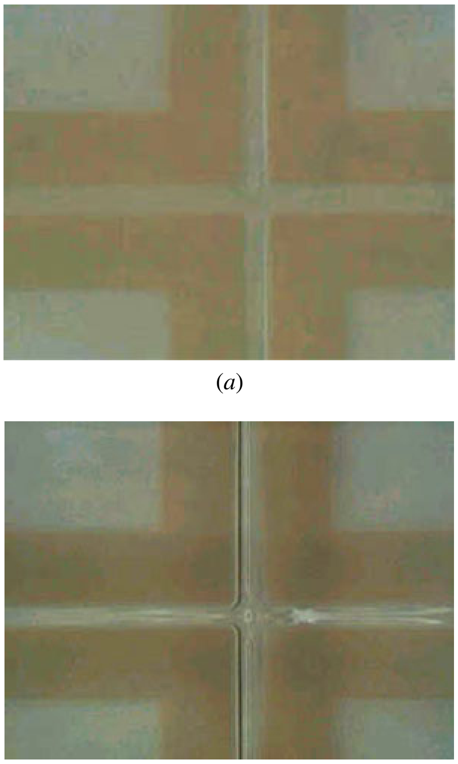


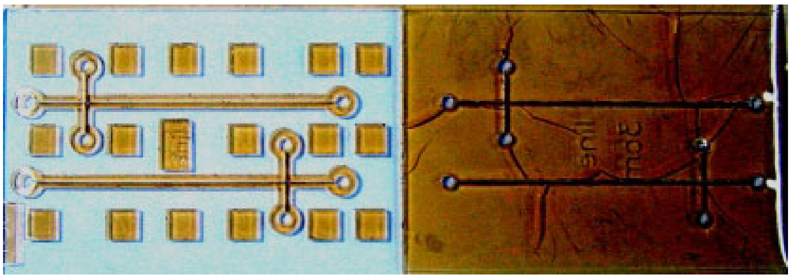
Figure 8. Cross-section image of the UV glue bonding region. The patterns on the UV glue and SU-8 layer were caused by the cleavage of the sandwiched structures. The thickness of the UV glue is about 10 μm.



(*a*)

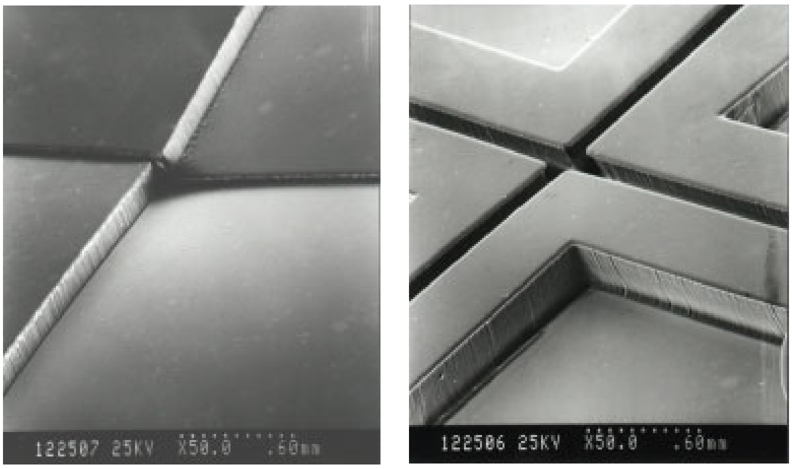
(*b*)

Figure 9. Photographs of baked SU-8 resist at different PEB temperatures: (*a*) 60 °C; (*b*) 95 °C.



(*a*) (*b*)

Figure 10. Pictures showing the new pattern design concept. The depth of the trench is 1 mm and the width is 100 μm. The glass substrate is 8 cm in length and 3 cm in width. It can clearly be seen that there are serious cracking problems for patterns with larger exposed areas while a good shape definition is obtained for the new pattern design.



(*a*) (*b*)

Figure 11. SEM images of a cross-shape channel on the same substrate. The channel collapsed for patterns with a continuous exposed area (*a*). With a proper design to divide the exposed area into several blocks, the well-defined channel without any collapse was obtained (*b*).

SU-8 is a negative PR which needs a post-exposure baking process to enhance the cross-linking of the exposed PR [21]. The recommended PEB temperature is 95 °C from the technical data sheet. A PEB at 95 °C may not be a problem for the fabrication of island structures. However, it cannot meet the requirement for the fabrication of SU-8 trench structures with millimeter depth. It has been noticed that the unexposed SU-8 might be squeezed out of a channel, causing the distortion of the structures. Figures 9(*a*) and (*b*) show top-view photographs of 500 μm thick SU-8 films baked at 60 °C and 95 °C for 30 min, respectively. The substrate baked at 60 °C shows a better shape definition for the exposed area (figure 9(*a*)). Conversely, for the substrate baked at 95 °C, further cross-linking of the exposed SU-8 occurred while the unexposed SU-8 inside the channel was distorted and the surface no longer remained flat. These problems become more prominent when fabricating ultra-deep trenches with a large exposed area. Furthermore, the internal stress formed in the SU-8 structures after the PEB process usually causes the cracking of an ultra-deep structure. Using a smaller exposed area or dividing the patterns into several discrete areas is an efficient method to solve the problem. Figure 10 shows pictures of two trenches with the same patterns using different mask design concepts. It can clearly be seen that there are serious cracking problems for patterns with a larger exposed area while a good shape definition is obtained for the new pattern design. The minimum feature of the discrete block is about 100 μm for a 1 mm deep trench.

In addition to the cracking problems, the collapse of the channel is another common problem in the fabrication of ultra-deep trenches. Figure 11 shows pictures of a cross-shape channel on the same substrate. The channel collapsed for patterns with a continuous exposed area (figure 11(*a*)). With a proper design to divide the exposed area into several blocks, a well-defined channel without any collapse was obtained (figure 11(*b*)).

SU-8 has been known to have excellent chemical and mechanical properties. In addition, the surface of SU-8 presented some unreacted epoxy groups that could be used for further derivatization of the interior walls of the fluidic channel [11]. Figure 12 shows a micropost array and a microfluidic channel fabricated by the process described above. The post is 210 μm in height and 40 μm in diameter. Micropost arrays outside the channel can work as a barriers for microbeads (or bioparticles) with immobilized materials. Therefore, beads can be trapped in the cavity for ligand/receptor binding. Micropost arrays can also greatly increase the surface area such that the efficiency of the chemical reaction can be improved. Alternatively, the micropost array can be used as a microreactor with filtration microdevices. Reactants can flow through the particle-filled cavity and then the products

Figure 12. A microfluidic device for bioanalysis with micropost arrays. The post is 210 μm in height and 40 μm in diameter.

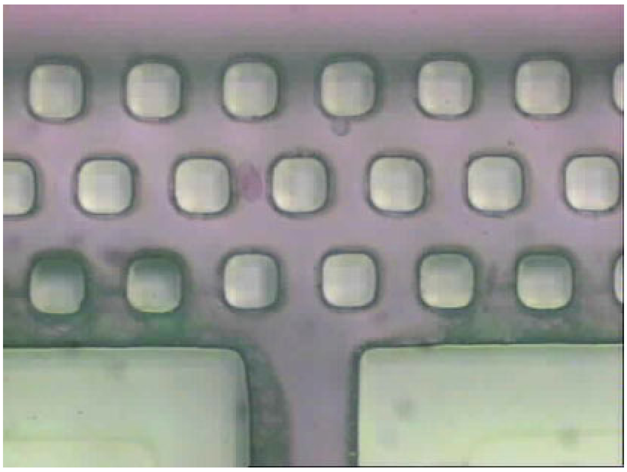


Figure 13. An optical microscopic image of a microfluidic device which was bonded with a flexible transparency using an SU-8 adhesive layer. The microfluidic channel was filled with rhodamine fluorescent dye.

can be injected into the microchannel for subsequent chemical analysis such as separation and detection.

Sealing the SU-8 microfluidic device with another SU-8 layer is a way to create microfluidic devices with consistent surface characteristics. The process for SU-8 bonding has been described in detail in [11]. However, sealing a microfluidic device with hard substrates such as glass or quartz substrates usually causes a low yield for microdevices with large areas. In this study, a simple but reliable process to spin the SU-8 on a flexible transparency film has been developed. Good bonding with high yield was observed. Figure 13 shows an image of the microfluidic device bonded by a flexible transparency with an SU-8 adhesive layer. The microchannel was filled with rhodamine fluorescent dye. No leakage between microchannels and microposts was observed. This indicates that a flexible cover substrate with a thin SU-8 layer more easily contacts the surface of the microdevice, resulting in a higher yield rate.

5. Conclusions

In this paper we have described a novel process for fabricating ultra-thick SU-8 structures using standard UV lithography. Instead of using conventional spin coaters, a constant-volume-injection method was developed to coat an ultra-thick SU-8 film up to 1.5 mm by single coating. The PR film coated by the developed technique was highly reproducible with reasonable uniformity. Without using spin coating methods, less PR was used such that the fabrication cost was reduced. More importantly, an exact contact printing was feasible since there was no edge bead effect. We have also reported on a modified baking process to greatly reduce the processing time and improve structure quality. A new mask design concept for fabricating an ultra-deep trench has been proposed and has proven to be an efficient way to avoid cracking problems. In this study, two microfluidic devices have been demonstrated using the developed fabrication process, including a micronozzle for thrust generation and a microfluidic device with micropost arrays for a high-performance microreactor. Two major bonding techniques for reliable sealing of microfluidic devices have also been reported. This novel process by standard UV lithography could be used as a simple way to fabricate microstructures as thick as those fabricated by delicate LIGA techniques.

Acknowledgments

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