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A versatile and flexible low-temperature full-wafer bonding process of monolithic 3D microfluidic structures in SU-8

J Steigert[1], O Brett[1], C Müller[2], M Strasser[1], N Wangler[1], H Reinecke[2,3], M Daub[1] and R Zengerle[1,3]

[1] Laboratory for MEMS Applications, Department of Microsystems Engineering (IMTEK), University of Freiburg, Germany
[2] Laboratory for Process Technology, Department of Microsystems Engineering (IMTEK), University of Freiburg, Germany
[3] HSG-IMIT, Wilhelm-Schickard-Str. 10, D-78052 Villingen-Schwenningen, Germany

E-mail: steigert@imtek.de

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Abstract
We present a versatile fabrication process for the precise fabrication of embedded three-dimensional microfluidic structures in SU-8 photoresist. The full-wafer bond process based on a polyester (PET) handling layer enhances the previous low-temperature bonding technology. We achieved an extremely high bond strength of 45 MPa while requiring only small anchoring structures. Small channel structures with an aspect ratio >2 as well as wide membranes with an aspect ratio <0.02 were successfully bonded to realize precisely defined channel structures. Furthermore, the developed process features high yields (>80%) and enables the integration of microelectronics. The flexibility of the fabrication process is presented in two contrary applications. A completely freestanding and transparent SU-8 foil with a thickness of 225 μm featuring embedded 3D microchannels was fabricated. Also, high quality ink-jet dispensers were successfully fabricated whereas the dispenser quality mainly depends on the channel quality.

Introduction

The impact of microfluidic technologies has dramatically increased during the last few years since microfluidics is considered a key technology within the field of life-science [1]. Various powerful tools have been developed for biological applications (e.g., lab-on-a-chip systems) and medical applications (e.g., implantable devices) [2–4]. These applications are based on the implementation of versatile fluidic unit operations, e.g. in lab-on-a-chip systems sample preparation, metering, mixing or separation. For the monolithic integration of those unit operations on one single chip, complex three-dimensional (3D) fluidic structures are required. This way, flexible technologies for the precise fabrication of complex microfluidic structures combined with a rapid and cost-efficient fabrication processes are required. Furthermore, the fabrication process has to be compatible with the integration of microelectronics which is needed in many applications for actuation or detection.

So far, many microfluidic devices are fabricated using complex, time consuming and expensive processes in silicon or if transparent setups are necessary in glass. Also the integration of microelectronics could only be ensured by using low-temperature bonding that has high requirements on substrate properties and quality [5]. Normally, a low surface roughness is needed which could not be obtained after metallization. Other polymer microfabrication strategies are based on casting, hot embossing, thermoforming or injection molding [6]. Various low-temperature bonding techniques of polymer substrates are available such as adhesive bonding or lamination. Nevertheless, the integration of microelectronics in polymer substrates is quite complex. Especially, polydimethylsiloxane (PDMS) is often used in microfluidics for the construction of multilayer devices [7, 8].

Nevertheless, PDMS is a flexible material and mechanically not very stable.

An alternative presents the use of an SU-8 resist. SU-8 is a negative tone epoxy photoresist developed in the mid 1990s by IBM in Zurich [9, 10]. It enables the fabrication of high-aspect-ratio microstructures via UV-lithography and shows a high mechanical reliability, good chemical resistivity as well as excellent optical properties. Combined with its good biocompatibility [11–13], SU-8 is well suited for microfluidic devices in the field of life-science: examples are single cell genetic manipulation [14], microneedles [15] and lab-on-a-chip devices [16]. The main challenge for the realization of microfluidic devices is the integration of precisely defined 3D microchannels in SU-8 and particularly their encapsulation.

So far, various approaches for the fabrication of enclosed channels in SU-8 have been reported based on (i) adhesive bonding [17], (ii) sacrificial layers [18–22], (iii) embedded metal masks [23–25], (iv) exposure dose controlled lithography [26, 27] and (v) low-temperature bonding [13, 16, 28–35]. One strategy for the fabrication of enclosed channels in SU-8 is the use of adhesives to bond two SU-8 layers (i). To ensure the biocompatibility of the materials epoxy-based adhesive is most suitable [17]. Nevertheless, the process is quite sensitive concerning the thickness of the applied adhesive layer. If the layer is too thick, the adhesive will flow into the channel due to capillary forces and leads to blocking of the channels. For thin adhesive layers a fluidically sealed bond could not be obtained. Typically, the thicknesses of the adhesive layer have to be controlled in the micrometer range, which is a challenging task.

The second method is the use of sacrificial layers like in traditional surface micromachining technology (ii). The use of standard positive photoresists as sacrificial materials [18, 19] is quite challenging since the solvent of the SU-8 resists reacts with standard positive resists [21]. To improve the fabrication of defined microchannels further sacrificial materials are promoted such as sol–gel systems [20], or metals where chromium is well suited [22]. The challenge in using these materials is the fabrication of channel heights larger than 20 μm. This way, Psoma *et al* [22] promoted the use of polymers such as PMMA (Polymethylmethacrylate) as sacrificial material. However, using sacrificial layers extends the fabrication time drastically, since the material is removed by diffusion-based transport phenomena. Especially, for the fabrication of long channels, this method is not well suited. An alternative could be the use of heat-depolymerizable sacrificial materials, which escapes out of the embedded material by diffusion through the cover layer during a thermolysis step [21]. The use of this method is limited to a small channel height lower than 25 μm as well as thin cover layers. Additionally, this could cause sagging of the sealing layer by bonding channels structures with low aspect ratios (<1).

The third method (iii) employs structured metal layers direct on the first SU-8 layer as masks during UV-lithography. Following, further SU-8 layers could be applied. This way, the method allows the fabrication of 3D microstructures. Due to the metal-covered SU-8 layers the good optical properties of the resist are no longer accessible. Also, the uncured SU-8 has to be removed by diffusion during the development step which drastically increases the fabrication time and also prevents the implementation of long microchannels.

Another method (iv) for the fabrication of embedded microchannels in SU-8 is based on exposure dose illumination during lithography. Thus, only the top of the SU-8 layer is illuminated. Two variations have been presented so far. A UV-lithography-based fabrication of embedded microchannels [27] shows a high sensitivity of the channel quality to the complex process parameters. Improved channel features and high-aspect ratios could be realized by an expensive direct writing technology using proton beams to pattern SU-8 films [26]. Like the presented methods so far, the uncured SU-8 resist has to be removed by time-consuming diffusion as well.

An excellent alternative to fabricate complex 3D microstructures could be realized by low-temperature bonding. One possibility is the lamination of dry films [36]. To ensure homogeneous surface properties for a better control and handling of the fluids, the fabrication of all channel walls out of the same material is an important aim. Thus, various approaches for the fabrication of embedded microchannels in the SU-8 photoresist by low-temperature bonding have been presented. An uncured SU-8 film is spun on a pyrex wafer and positioned on top of a structured SU-8 layer. Afterwards, the uncured SU-8 layer is illuminated through the pyrex wafer and the bond is achieved during the post-exposure step by applying a defined temperature and pressure [13, 31, 32]. Drawbacks of this approach are a large proximity gap by illumination through the pyrex wafer which decreases the resolution and limits the accurate fabrication of the small channel. Furthermore, the handling and processing of the unexposed and thus uncrosslinked SU-8 is very complex since the resist flows into the mircochannels during the bonding process and leads to undefined channel geometries or even blockage of the channels. Another approach is to use structured SU-8 layer as an adhesive to bond silicon and/or glass wafers [16, 28, 33, 34, 35]. This approach allows the fabrication of defined channel geometries but requires complex post-processing steps to structure or remove the silicon or glass wafers, e.g. to generate fluidic interfaces. To improve this, two strategies have been presented [29, 30]. Abgrall *et al* [29] applies an uncured SU-8 film on a PET-foil and forms a bond by lamination of this film onto structured SU-8 layers. Afterwards, the uncured SU-8 is illuminated and developed. Due to the low adhesion of the SU-8 on the PET substrate the foil could easily be peeled off. This process allows the fabrication of 3D channels based on multiple layers of structured SU-8 layers. Nevertheless, the fabrication based on uncured resist layers as mentioned before is an extremely complex procedure. Agirregabiria *et al* [30] structures crosslinked SU-8 layers on top of a polyimide foil. The SU-8 layers are finally bonded together by applying a pressure and a temperature higher than the glass-transition temperature of the resist. After the bonding, the polyimide foil could also be peeled of from the SU-8 structures due to low adhesion forces. The presented process show good channel qualities as well as allows the fabrication of complex 3D structures. As mentioned in the publication, the limitation

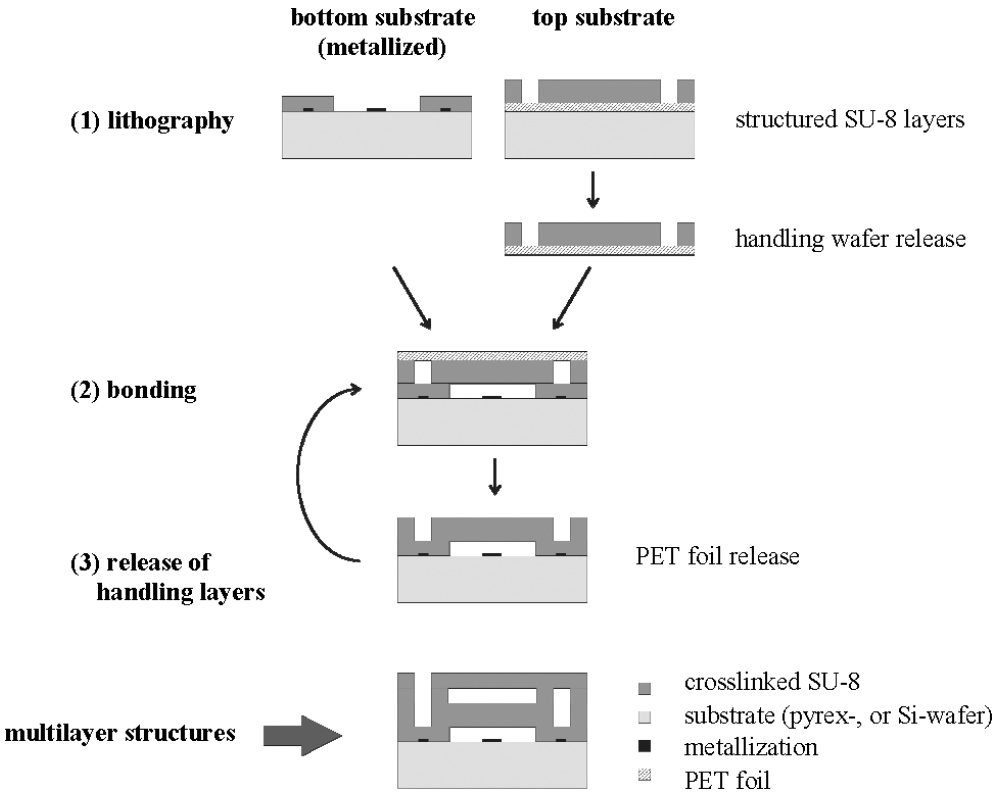

Figure 1. Fabrication process of SU-8 multilayers with embedded microfluidic channels. SU-8 layers are structured on a bottom substrate and on a PET layer on top of a handling wafer (1), than bonded together (2) and finally the PET foil could easily be peeled of the SU-8 structures (3). By repeating the process steps SU-8 multilayer featuring 3D microfluidic structures could be assembled.

is the realization of large freestanding areas. To also obtain a good yield of the bonding process, large anchor structures, a minimum of four times larger than the channel width, have to be implemented which leads to a large chip size.

Our approach presented in this paper is based on a low-temperature bond to fabricate complex microfluidic 3D structures containing embedded channels with precisely defined geometries in SU-8 photoresist. The fabrication process is based on the new SU-8 3000 series from Microchem Corporation [37]. We optimized the temperature and time of the soft-, and post-exposure bake to ensure both, mechanical stability of the resist for the fabrication of rectangular channels with well-defined geometries as well as a certain adhesiveness which is required for the low-temperature bond. The strategies of bonding SU-8 presented so far are based on the use of uncrosslinked SU-8 [29] or on completely cured SU-8 where a bond results from a heating over the glass transition temperature [30]. The fabricated microfluidic structures show low mechanical stress, allowing leakage free bonds of small structures with extremely high bond strengths up to 45 MPa. Typically bond strengths for the SU-8 bonds reported so far are in the range of 16–20 MPa [16, 34]. In comparison to the work presented so far our process does not require any additional adhesion layers even for the fabrication of large connected areas and features a high flexibility concerning various channel heights and contrary aspect ratios where only small anchoring structures (20 smaller than the channel width) are required. Furthermore, we present a technique for releasing of cured and structured SU-8 layers from a handling wafer to fabricate completely freestanding SU-8 structures. To demonstrate the high performance of our process we fabricated ink-jet dispensers which are known to be very sensitive to defined channel structures (e.g. nozzle) and leakage-free bond of small areas (e.g., flow resistance).

Fabrication process

Concept and materials

The presented full-wafer bond is based on the fabrication of SU-8 (3000 series) layers on a PET foil on top of a handling wafer (top substrate) and on a bottom substrate. The SU-8 layers are baked with an optimized parameter ensemble to ensure a viscosity high enough for the fabrication of defined channel geometries and small enough to ensure a tight bond. After exposure and development the PET foil with the structured SU-8 layer is easily peeled of the handling wafer. Finally, the SU-8 structures area aligned and bonded together using a low-temperature bond and the supporting PET foil is released. The procedure of this full-wafer bond is illustrated in figure 1.

The presented work is based on the new SU-8 3000 series from Microchem Corporation [37]. Two types of this photoresist have been used, SU-8 3025 and SU-8 3050. Compared to the older SU-8 series the mechanical stress which emerges during the baking steps is drastically reduced by an adapted thermal expansion coefficient and a decreased volume shrinking [38, 39]. This way, the adhesion force to the

substrates is drastically improved, up to the double depending on the used substrate materials and enables the fabrication of connected large-area SU-8 structures. Thus, additional adhesion layers such as those presented by Agirregabiria *et al* [30] or Calier *et al* [13] are no longer required.

For the mechanical release of the SU-8 microstructures from the handling wafer, a suitable process was selected. A lift-off technology presented by Bohl *et al* [40] relies on the high internal stress in SU-8 layers (SU-8 2000 series) which leads to a mechanical separation of the SU-8 layer and the supporting wafer during the development step. Due to the high internal stress and the large volume shrinkage, this fabrication process is not suited for subsequent bonding steps. Materials which feature low adhesive forces to SU-8 have been presented, e.g. Gadre *et al* [12] promotes the use of Teflon. Nevertheless, this process does not offer the possibility of aligning the structures before bonding since Teflon is not transparent. An intermediate layer based on a polyimide foil has been presented by Agirregabiria *et al* [30]. Due to the low adhesion force of polyimide to other materials, the foil is bonded via a sacrificial positive resist to a handling wafer. A more simple approach is the use of polyester (PET) which shows a low adhesion to SU-8 as well as a good adhesion to silicon [29].

Substrate preparation

As substrates we used both 4″ silicon and pyrex wafers. As handling layer a commercially available polyester (PET) foil (native, untreated) [41] with a thickness of 100 μm was used. First the wafers and the PET foil were cleaned with isopropanol, rinsed (DI-water) and dried. Since the PET foil is not mechanically stable, it is placed on a handling wafer i.e. a blank silicon wafer as a supporting substrate. To enhance the adhesion of the PET foil on the silicon substrate, a droplet of water is applied between the two materials. Due to the flat surfaces high meniscus forces emerge resulting in a sufficient adhesion of the PET foil up to 4000 rpm during spin coating. The following process describes the fabrication of the ink-jet dispenser. On a metallized (Ti/Au) bottom substrate a 25 μm thick layer of SU-8 3025 and on the PET-foil a 100 μm thick layer of SU-8 are spinned according to the data sheet [37]. The most critical step to achieve a highly reliable process is the precisely defined baking time. Both substrates, the bottom and the top substrates with the PET foil, are slowly heated up to 90 °C (4.5 K min^{-1}). Subsequently, the thin SU-8 layer on the bottom substrate is baked for 5 min and the thicker layer on the top substrate for 10 min. To obtain sticky SU-8 layers for building up 3D multilayer structures, the softbake period was reduced from the protocol mentioned in the data sheet (10 min at 95 °C for the 25 μm layer and 45 min at 95 °C for the 100 μm layer [37]). Finally, the layers are slowly cooled down to room temperature (1 K min^{-1}) to achieve mechanical stress-free resist layers. Using the SU-8 3000 series the influence of the temperature concerning the layer topography could be drastically decreased. Thus, flat surfaces with a roughness of <50 nm were fabricated using the mentioned lithography parameters. Furthermore, the thicknesses of the SU-8 layers could be realized with a reproducibility of CV = 3.5%, measured at nine different positions per wafer using a profilometer and a microscope. Since, the layer thickness of SU-8 drastically increases at the boundary of the wafer, the resist was stripped from the outer 3 mm. A further influence of the SU-8 layer quality on the bond quality was not observed. For SU-8 resists older than 1 year, the baking time have to be reduced by additional 2 min to ensure the required properties of the SU-8 layer.

Photolithography process

The photolithography processes are carried out with chromium masks (Delta Mask V.O.F., NL) as well as photoplotter masks (KOENEN GmbH, D, 64 000 dpi) using the maskaligner MA6/BA6 from SUSS MicroTec AG, D. The 100 μm layer was illuminated for 30 s and respectively the 25 μm layer for 20 s via UV-exposure with a dose of 6 mW cm^{-2} using an I-line filter. A time-reduced post-exposure bake was carried out for 3 min at 90 °C and the wafers were again slowly cooled down to room temperature (1 K min^{-1}). Finally, the SU-8 layers were developed (mr-Dev-600, micro resist technology) for 20 min (100 μm layer) and 15 min (25 μm layer) followed by rinsing and drying. Subsequently, the PET foil supporting the SU-8 layer could easily be peeled off the handling wafer by using tweezers.

Bonding process

The SU-8 layers were aligned to each other using the MA6/BA6 bond aligner and fixed. First, the bottom substrate is loaded into the fixture of the bond aligner. Next, the PET foil supporting the SU-8 layer is placed on the chuck and fixed by applying a vacuum. After the precise alignment the two SU-8 layers were brought in contact and mechanically fixed between the clamps of the fixture. Finally, the fixture is released from the bond aligner and the substrates are ready for bonding. Due to the reduced bake times the SU-8 resist remain sticky. During a combined hard bake using the hot-embossing device (adapted hydraulic mould press from Schmidt Maschinentechnik, D) the low-temperature bond is carried out using an optimized parameter ensemble. A vacuum of 10 mbar is applied to avoid trapping of air bubbles between the SU-8 layers. Further, a bonding pressure of 70 N cm^{-2} was applied at 125 °C for 10 min. After bonding, the PET foil is peeled off the bonded SU-8 layers. By fabrication of further SU-8 layers and subsequent bonding to the so far process SU-8 stack, 3D multilayer structures were realized. To fabricate high quality SU-8 layers to achieve a leakage-free bond the precise control of the baking time and the temperature profile is the most crucial step. Furthermore, to achieve a tight bond the presented process has to be carried out within two days.

Results

Aspect ratio and quality of microfluidic structures

The most important issue concerning microfluidic structures is a leakage-free bond. This is ensured by realizing continuously

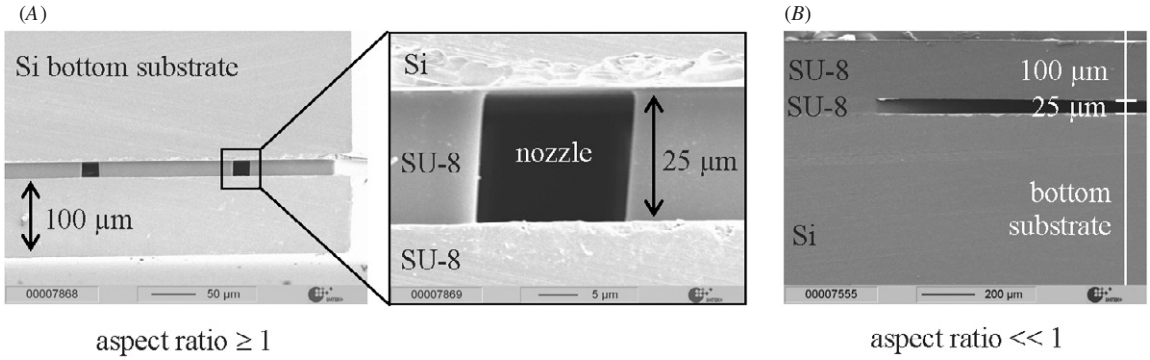

Figure 2. Simultaneously fabricated structures in SU-8 featuring with various aspect ratios. A pressure of $p = 70$ N cm^{-2} and a temperature of $T = 125$ °C were applied for 10 min. (*A*) Nozzle structure with aspect ratio $\geqslant 1$. (*B*) Bonding of large freestanding reservoir structures with extremely small aspect ratios $\ll 1$.

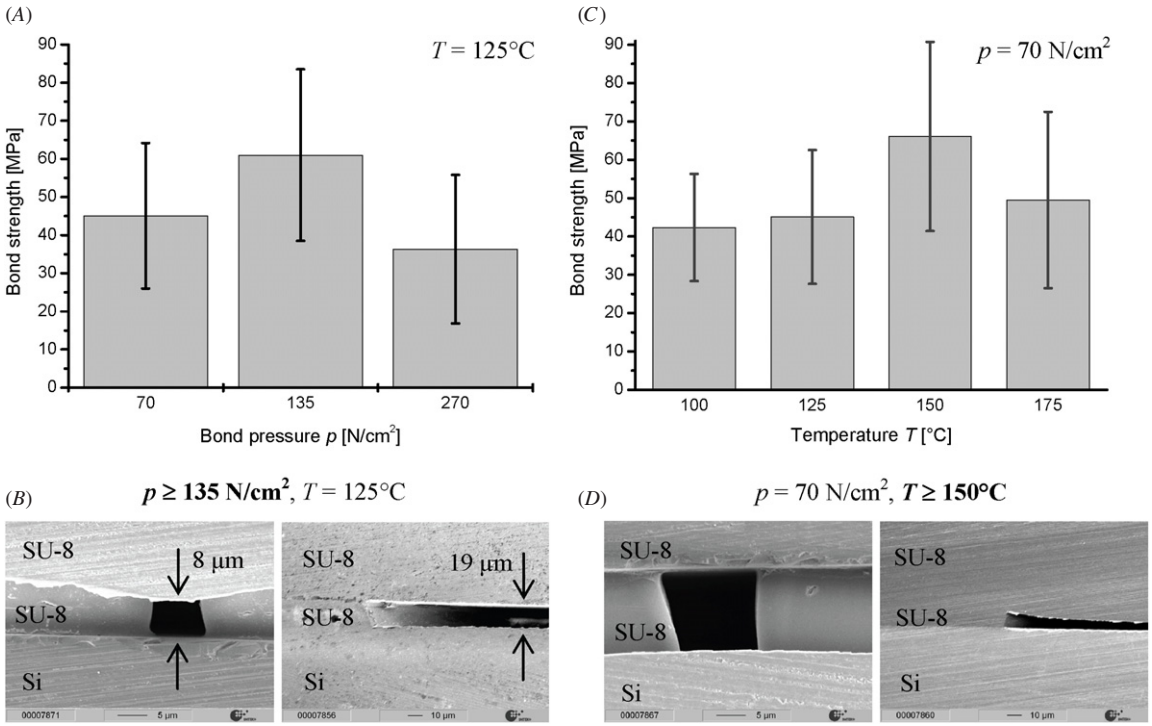

Figure 3. Realized bond strength depending on the bond pressure (*A*) and the bond temperature (*C*). With bond pressures too high the microfluidic structures are compressed (*B*). Too high bond temperatures lead to deformation of the channel geometry (*D*). The best combination of channel quality, yield and bond strength are realized by bonding with $p = 70$ N cm^{-2} at $T = 125$ °C.

bonded structures with an extremely high bond strength of 45 ± 15 MPa. Furthermore, various contrary structures featuring a wide range of aspect ratios were bonded leakage-free. Concerning the aspect ratios of microfluidic structures typical values in most applications are $\leqslant 2$. With our process we achieved embedded structures with a maximum aspect ratio of 2.5 realized on small anchoring structures (flow resistance with 10 × 14 μm^2) featuring a very small channel size (width 10 μm, height 25 μm) as well as large freestanding structures (reservoirs) with an aspect ratio <0.02 (width 2000 μm, height 25 μm) bonded on 100 μm width columns without any sagging of the sealing layer. In this case, the anchoring structures were 20 times smaller than the channel width. This way, an extremely flexible process was developed to fabricate precise channel structures within a transparent SU-8 resist covering a wide range of microfluidic geometries. Figure 2 presents the realized structures of the well-defined nozzle with a cross-sectional area of 25 × 25 μm^2 (figure 2(*A*)) and large reservoir structures featuring aspect ratios <0.02 (figure 2(*B*)) bonded with $p = 70$ N cm^{-2} at $T = 125$ °C (below mentioned as 'standard parameter set'). Using this parameter set, complementary aspect ratios with various embedded microchannels featuring rectangular cross-sectional areas are successfully realized.

Bond strength

During process development the main focus was put on reliable geometries with high bond strengths at the same time. This was achieved with the above-mentioned standard

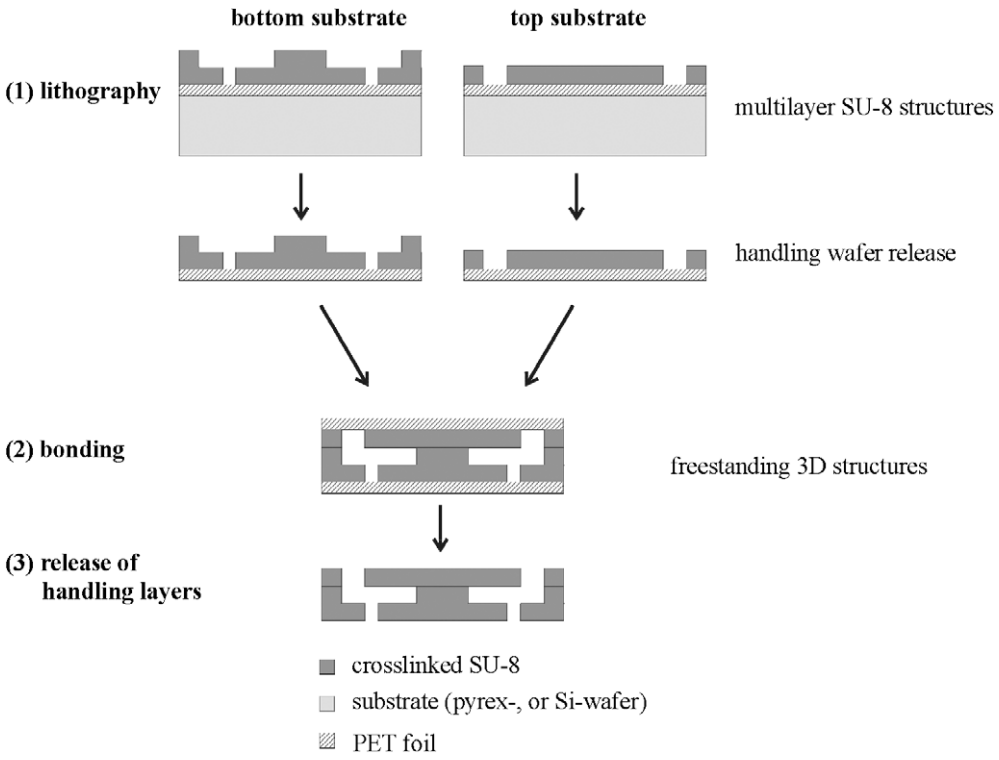

Figure 4. Fabrication process for completely freestanding SU-8 structures with embedded 3D microchannels. Multiple SU-8 layers are structured on PET release layers (1). After alignment and bonding (2), the PET foil is removed easily (3).

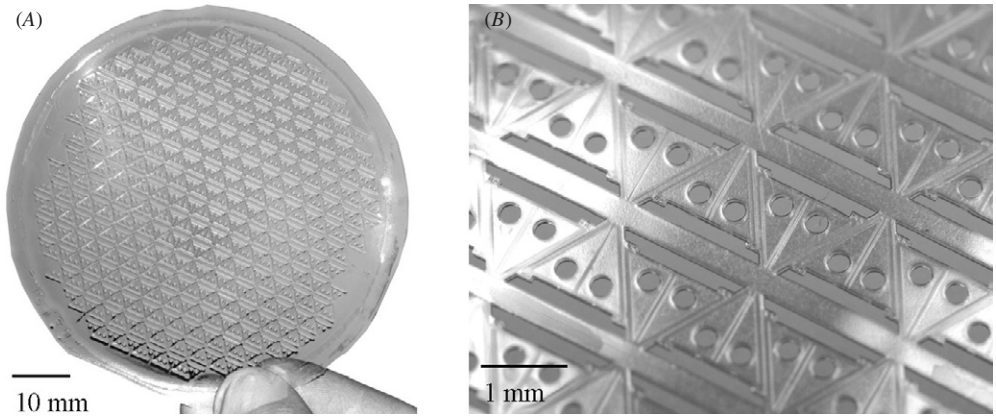

Figure 5. (*A*) Realization of a completely freestanding SU-8 foil in 4″ format with a thickness of 225 μm featuring a 3D embedded microchannel. (*B*) Detailed picture of the SU-8 foil featuring vias and microfluidic structures.

parameter set. The bond strength was investigated via pull tests using the *Z*010 from Zwick GmbH & Co. KG. We achieved extremely high bond strengths of 45 ± 15 MPa using the above-mentioned bond parameters for various substrate materials (silicon, silicon nitride, pyrex) with a very good yield of more than 80% of bonded chips. Applying higher bond pressures could improve the bond strength (60 MPa bonded with 135 N cm^{-2}) (figure 3(*A*)), but the microfluidic structures are drastically deformed (figure 3(*B*)). To achieve a bond, a minimal temperature of 100 °C has to be applied resulting in a low yield (<50%). Temperatures larger than 125 °C increase the bond strength (up to 66 MPa at 150 °C) (figure 3(*C*)) but result in tapered sidewalls (figure 3(*D*)). Concerning larger bond periods than 10 min, no influence was observed.

Freestanding 3D multilayer structures

Completely freestanding 3D microfluidic structures out of SU-8 are realized by slight modification of the fabrication process. Multiple SU-8 layers were each structured on two PET foils supported by handling wafers and were bonded together using the above-mentioned process with the standard parameter set (figure 4). Afterwards, the PET-foil is peeled of from the top- and bottom side and completely freestanding SU-8 structures are realized. Figure 5 presents a transparent SU-8 foil with a thickness of 225 μm featuring embedded 3D

microchannels. This opens new, fast and simple fabrication prospects for versatile microfluidic application, e.g. for small-size microneedle arrays for *in vivo* drug release.

Microfluidic characterization

In this context the developed versatile fabrication process was used to realize thermal ink-jet (bubble-jet) dispensers. The robust functionality and a good reproducibility of bubble-jet dispenser depend mainly on three parameters. First, the quality of the metallization and especially the heater structure. Second, the precise definition of the microfluidic resistance by defined channel geometries to achieve a reproducible fluid ejection. Third, the quality of the dispenser nozzle to realize a reproducible droplet formation. We successfully fabricated bubble jets, dispensing a minimal droplet volume of 6 pL and operating with a maximum actuation frequency of 11 kHz. The functionality of the bubble-jet dispenser was simulated using computational fluid dynamics and the results predicted a dispensed volume of 5.9 pL and a maximum actuation frequency of 11.2 kHz. The very good correlation between the simulation and the experimental results arises from the precise fabrication.

Conclusion

We successfully presented a new, versatile fabrication process for the realization of embedded microchannels in SU-8. In contrast to the approaches presented so far, we have combined extremely high bond strengths up to 45 MPa, have simultaneous fabricated of channel geometries featuring a wide range of contrary aspect ratios (0.02–2.5) and have gained the possibility of microelectronics integration. Furthermore, this low-temperature bond approach allows the cost-efficient fabrication of microfluidic structures on a wafer level with a good yield (>80%) whereas small anchor structures (down to 20 times smaller than the sealed channel width) are required for a leakage-free bond. We demonstrate the flexibility of the developed process by the fabrication of high performance ink-jet dispensers as well as a completely freestanding SU-8 foil with integrated 3D microfluidic structures. Possible applications could be the fabrication of small size microneedle arrays for *in vivo* investigation with integrated microchannels for chemical stimulation and microelectronics for electrical recording. Also lab-on-a-chip systems with the need of microchannels featuring a wide range of channel geometries and aspect ratios will benefit from the presented process. This way, the developed process opens new fabrication possibilities for realizing microfluidic structures in various applications.

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