

Adhesive bonding by SU-8 transfer for assembling microfluidic devices

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Received: 27 January 2012 / Accepted: 21 May 2012 / Published online: 23 June 2012
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Abstract SU-8 is largely used to make microfluidic molds or components, but mainly for producing high-precision and thermally stable structures. We present a versatile method that employs SU-8 as glue to perform an adhesive bonding between micro-patterned structures. More in general, this technique enables an easy assembly of microfluidic devices, which can also be made by different materials, where selective bonding is required. The adhesive bonding is achieved by transferring a thin layer of SU-8 5 (thickness $\leq 15 \mu\text{m}$) on a substrate by means of a polyimide foil. The method is described in detail and an example of its application is given. Finally, a shear test is carried out to prove sufficient adhesion strength for microfluidic applications.

Keywords SU-8 · Selective bonding · Microfluidics · Patterned substrates

1 Introduction

In the development of microfluidic devices, fabrication methods combine the use of various materials and the assembly of different structures (Abgrall and Gué 2007; Li and Psaltis 2008). The bonding process often employs epoxy and ultraviolet (UV) curable adhesives. Although the market offers biocompatible adhesives that are successfully used in many microfluidic applications, thermo-mechanical stress and chemical instability have been reported (Sarvar et al. 2002). In recent years, SU-8, a negative epoxy photoresist that allows easy fabrication of micro-architectures, has been largely adopted in hybrid microfluidic chips. SU-8 is well known for its excellent chemical, mechanical and thermal properties (Roach et al. 2007; Youn et al. 2008). Moreover, it is optical transparent and a high bonding strength can be achieved as stated in SU-8 shear analysis (MicroChem Corp. 2007). Therefore, it is logical to employ it for adhesive bonding.

A standard approach consists of creating complex micro-structures by bonding successive layers of SU-8. Monolithic chips can be fabricated by stacking multi-layers of SU-8 by successive coating steps (Moser et al. 2011) or by means of a sacrificial layer such as PDMS (poly(dimethyl siloxane)) and PET (poly(ethylene terephthalate)) (Aracil et al. 2010; Abgrall et al. 2006). In the latter case, the SU-8 is spin-coated on the sacrificial carrier and bonded to pre-existent SU-8 structures. After the removal of the carrier, which is usually peeled off, the SU-8 layers are eventually bonded together by ultraviolet (UV) exposure and subsequent baking steps. A similar technique, called flexible semisolid transfer (FST) by the authors (Song et al. 2004), employs a semi-solid layer of SU-8 to fabricate a microfluidic bioreactor. SU-8 microchannels are fabricated and bonded to PMMA (poly(methyl methacrylate)),

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stainless steel or PEEK (poly(ether ether ketone)) substrates. The bonding is achieved by controlling the exposure dose to lead SU-8 to a semi-solid phase where it maintains the properties of a cured resin and, at the same time, remains flexible. As in other methods, a sacrificial carrier, such as polyethylene, Teflon, polycarbonate and polyimide (PI) films, is used in the FST process. The carrier is peeled off after the complete curing of SU-8. A more hybrid device is fabricated by Ou et al. (2009) with three layers of PDMS, SU-8 and quartz, respectively. In this chip, the SU-8 is simply spin-coated and patterned on quartz, whereas the bonding with PDMS is carried out by plasma treating the surface.

At wafer level, SU-8 has been used to bond two wafers by applying a pressure depending on the bonding area and fragility of the involved materials (Svasek et al. 2004). Wafer-to-wafer bonding is also possible by contact imprinting (Yu et al. 2006). The wafer is put on a thin layer of SU-8 and, after the release of the wafer, the SU-8 is transferred on it. Finally, the wafer is aligned and bonded to a second wafer. However, this technique is not easy to apply because, to complete the transfer, the release of the wafer from SU-8 is performed on a vacuum hot plate at 150 °C by means of a blade. For bonding areas larger than 70 %, the authors suggest another technique that uses a Teflon cylinder to deposit SU-8 on the wafer. Nonetheless, we observed an insufficient SU-8 coating on a Teflon rod during our tests, which eventually led to unsatisfactory results.

The literature clearly shows the adhesive properties of SU-8 as excellent adhesive layer, but the proposed methods presume that a SU-8 layer is completely transferred on the substrate surface and cured. When the fabrication of the microfluidic device requires the bonding of two patterned substrates, it is necessary to implement a different procedure.

In this paper, a new bonding technique is presented and tested. Unlike FST or common transfer approaches, a thin layer of SU-8 is transferred only on the patterned areas of one or both substrates by means of a sacrificial carrier, which is removed in a successive step. Subsequently, the bonding, and therefore the sealing of the entire structure, is accomplished by bringing the substrates in contact and performing a baking step. This technique is also an easy and fast alternative for fabricating hybrid microfluidic devices, where the choice of the appropriate glue can be critical due to the diversity of the used components. Furthermore, it is a suitable and relevant solution in those cases where it is not possible to spin SU-8 on a substrate surface that cannot withstand the development procedure or does not allow the SU-8 removal after bonding. The SU-8 transfer method and the optimization of processing conditions are described in detail as follows.

2 Materials and methods

2.1 Presentation of the SU-8 transfer method

The generic procedure is illustrated in Fig. 1. A thin SU-8 layer ($\leq 15 \mu\text{m}$) is spin-coated on a sacrificial substrate (Fig. 1a, b). In a next step, a substrate containing micro-patterns is brought in contact with the SU-8 (Fig. 1c). After separation, SU-8 is transferred on patterned areas of the substrate (Fig. 1d) and bonding with another microfluidic substrate can be achieved (Fig. 1e, f). Obviously, the separation is possible if the adhesion between the sacrificial substrate and SU-8 is weak enough to allow a sufficient transfer to the micro-patterned substrate. However, at the same time, during spin-coating, SU-8 has to be uniformly distributed and adhere to the sacrificial substrate. Several tests have been carried out to find the proper carrier (Table 1).

Materials such as PDMS and polyurethane have been proven unsuitable because of their intrinsic hydrophobicity. During our tests, this has led to the formation of SU-8 drops on the surface, even after plasma treatment which is known to render the surface hydrophilic (Bhattacharya et al. 2005). On the other end, PMMA, polycarbonate (PC), SiO_2 , Si_3N_4 , glass and ceramic have exhibited a strong adhesion with SU-8 and the separation has been found not feasible. In fact, either SU-8 fills the patterned areas or, in the worst case, the samples are damaged during the release. Copper, gold and brass are closer to the ideal material, but the separation is still a complex operation to perform for substrates larger than 1-in square. Finally, PI is chosen as carrier because it has proven to be the best compatible sacrificial layer since it does not suffer from the reported complications present in the other tested materials. The integration of PI leads to a change in the method described in Fig. 1.

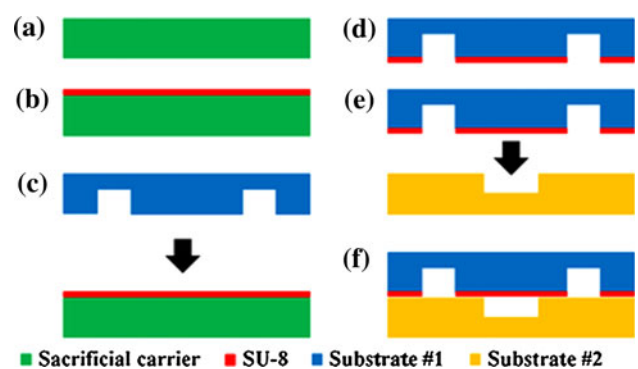


Fig. 1 Transfer method: **a** sacrificial carrier, **b** a thin layer of SU-8 is spin-coated on the carrier, **c** substrate 1 is brought in contact with SU-8, **d** Substrate 1 is released and SU-8 is transferred to the micro-patterns, **e** contact between substrates 1 and 2, **f** bonding of the two substrates

Table 1 Tested materials as sacrificial carrier for SU-8 transfer method

Material	Pass	Fail	Remarks
PDMS		X	Hydrophobic
Polyurethane		X	Hydrophobic
PMMA		X	Strong adhesion with SU-8
Polycarbonate		X	Strong adhesion with SU-8
SiO ₂		X	Strong adhesion with SU-8
Si ₃ N ₄		X	Strong adhesion with SU-8
Glass		X	Strong adhesion with SU-8
Ceramic		X	Strong adhesion with SU-8
Copper		X	Complex release from SU-8
Brass		X	Complex release from SU-8
Gold		X	Complex release from SU-8
Polyimide	✓		

2.2 Adhesive bonding by SU-8 transfer: procedure

The steps from Fig. 1a–e are modified as illustrated in Fig. 2. The process starts with a glass wafer that is chosen to guarantee an initial rigid and flat carrier. PDMS (Sylgard® 184 by Dow Corning) is spin-coated for 60 s at 3,000 rpm by SPIN150™ and cured at 100 °C for 2 h in a convection oven. The resulting PDMS thickness is approximately 20 μm (measured using an optical profiler WYKO NT3300). Polyimide foil (UPILEX®-25S by UBE Ind.) is manually laminated on the PDMS layer where it temporarily sticks. Care is taken so that no air is trapped in between the PI foil and the PDMS. Next, SU-8 5 (Micro-Chem Corp. 2007) is spin-coated on the PI foil. For a reliable transfer process, the SU-8 thickness (*t*) ranges from 7 to 15 μm, which corresponds to spin speeds of 2,000 and 1,000 rpm, respectively. For most applications, 10 μm is determined to be the optimal value. Before peeling the PI foil from the PDMS and performing the transfer (Fig. 2f, g), SU-8 overflow in the patterned substrate is minimized

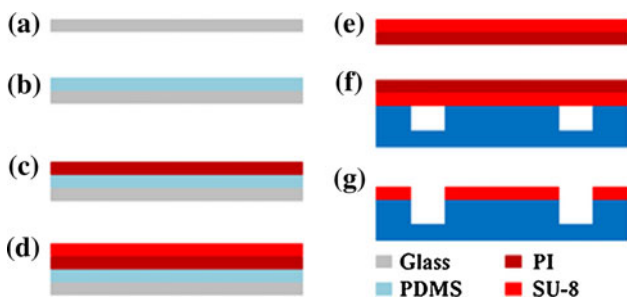


Fig. 2 Modified steps from Fig. 1a–e: **a** glass is chosen as rigid and flat carrier, **b** 20 μm PDMS layer is spin-coated on the glass carrier, **c** PI foil is laminated on PDMS, **d** a thin SU-8 layer is spin-coated on the PI foil, **e** the PI foil is peeled from the PDMS, **f** SU-8 is brought in contact with the micro-patterned substrate, **g** the PI foil is removed and SU-8 is transferred to the substrate

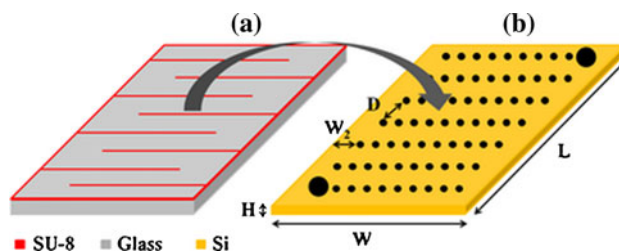


Fig. 3 Example of application of the SU-8 transfer method. A meandered SU-8 structure on a glass substrate shown in **a** is bonded to a Si chip (**b**). The chip is coated by 400 nm of SiO₂ and ten blocks (black dots lines) of one thousand pores each are present on the surface (pore diameter = 4 μm). In the external blocks, there are the inlet/outlet pores (diameter = 4 mm), which are drawn as larger black dots. *H* = 700 μm, *W* = 12 mm, *L* = 14 mm, *D* = 260 μm and *W*₂ = 1.3 mm

by reducing the solvent content in SU-8 by a baking step at 65 °C on a hot plate for 40 s for *t* = 7 μm or 90 s for *t* = 15 μm. Longer baking steps have to be avoided because an excessive reduction of the solvent leads to poor adhesive properties. Tests have shown that for *t* = 10 μm, after PI removal (that should not last more than 30 s to avoid excessive spreading of SU-8), the thickness of the SU-8 left on the PI foil is less than 2 μm (WYKO NT3300). In case both patterned substrates are not UV transparent, the SU-8 exposure step (100 or 150 mJ/cm² for *t* = 7 and 15 μm, respectively) can be carried out before bonding. Alternatively, if at least one of them is UV transparent, the entire structure is exposed to UV light after bonding.

SU-8 is eventually cured on a hot plate for 1 min at 65 °C, followed by 5 min at 95 °C, independently of the SU-8 thickness.

3 A microfluidic application

A practical example for a microfluidic application is shown in Fig. 3. A silicon sieve coated with 400 nm of SiO₂ (Fig. 3b; *W* = 12 mm, *L* = 14 mm, *H* = 700 μm, *W*₂ = 1.3 mm, *D* = 260 μm) has ten blocks of pores equally spaced (pore diameter = 4 μm). In each block there are one thousand pores except for the external blocks where an inlet/outlet (diameter = 4 mm) is present. Each pore has an integrated transistor to isolate, by impedance analyses, circulating tumor cells (CTCs) from blood or disseminated tumor cells (DTCs) from bone marrow. The SU-8 transfer method is used to bond the sieve to a meandered structure made by SU-8 100 (Fig. 3a; SU-8 (a) thickness = 100 μm, (b) width = *W*₂ for the rectangular border and (c) 100 μm for the internal walls) patterned on a glass substrate. The meandered structure is plasma treated (Pico by Diener Electronic) for 5 min to

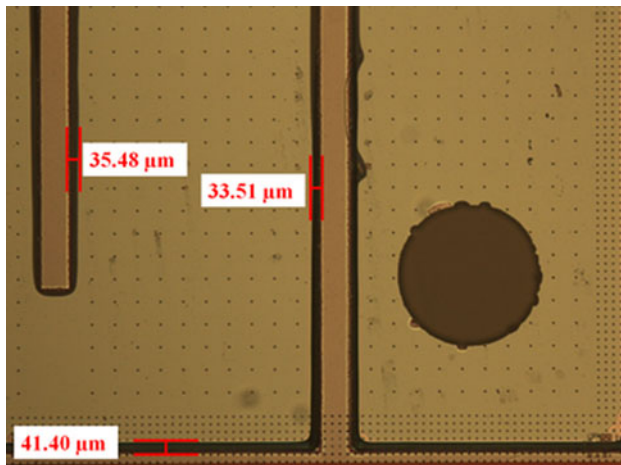


Fig. 4 Top view details of the SU-8 structure on glass substrate bonded to a Si chip with pores. The widths of the squeezed out SU-8 are acceptable since no occlusion of the pores is visible. The circular area on the right represents the chip inlet/outlet

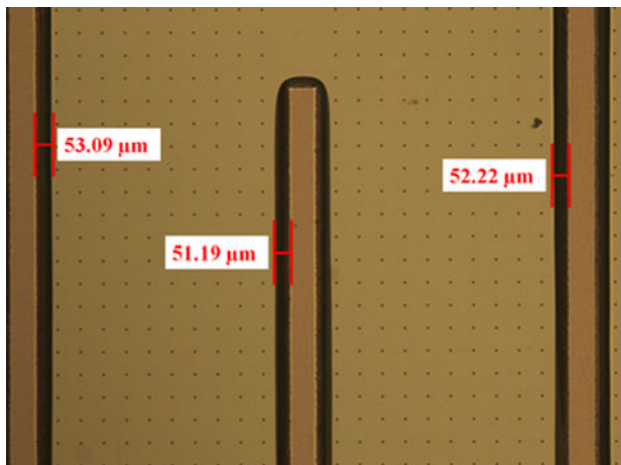


Fig. 5 The SU-8 overflow is found to be, on average for six samples, $42 \pm 10 \mu\text{m}$. The worst case is shown in this figure. The maximum width of the SU-8 surplus is $53.09 \mu\text{m}$

improve the adhesion between SU-8 5 and 100. Figure 4 (taken by optical microscope Nikon Optiphot 200) shows a detail of the sieve bonded to SU-8 walls. Due to the weight of the glass substrate, there is a compressive force that causes a partial squeezing out of SU-8. Nevertheless, since no occlusion of the pores is visible, this phenomenon is acceptable. After tests performed on six samples, the average width of SU-8 overflow is found out to be $42 \pm 10 \mu\text{m}$, approximately. The worst overflow case is shown in Fig. 5. Hence, at least for this application, the minimum channel width, i.e., the minimum distance between two SU-8 walls, should not be less than $100 \mu\text{m}$.

The chip has been tested with a buffer solution ($1 \times \text{PBS}/0.1\% \text{BSA}$) at a flow rate of $200 \mu\text{l}/\text{min}$. The

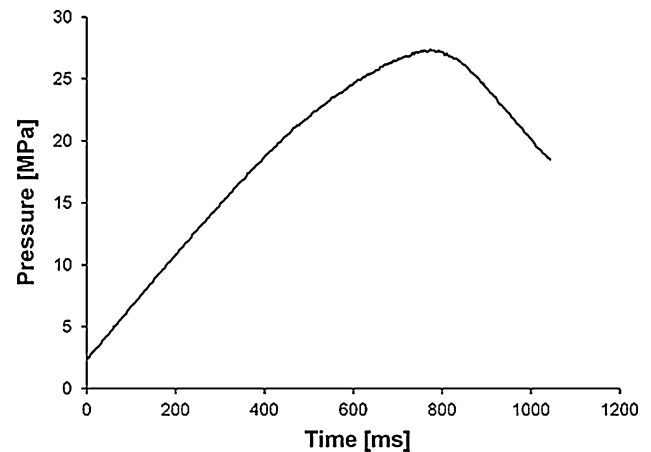


Fig. 6 Example of a shear test result that shows the pressure variation versus time. The detachment between the sieve and the SU-8 meandered structure is visible at approximately 27 MPa after 0.8 s

meanders are fully filled at a pressure of 4 mbar (manometer GDH 200 by Greisinger), whereas the flow through the pores occurs at 100 mbar, roughly.

To determine the adhesion strength, a shear test is carried out on three samples. To this end, a Dage series 4000 system (Nordson) is used. Test speed is $500 \mu\text{m}/\text{s}$ and the force is applied by the tester tip on the short side of the chip. Results show that the average debonding pressure is approximately $27 \pm 3 \text{ MPa}$ (Fig. 6), while 10 MPa is needed for a shear movement of $100 \mu\text{m}$. These values are definitely large enough to allow the use of the presented method in microfluidic devices for gluing together patterned substrates.

4 Conclusions

In this paper, a novel method is presented to perform selective adhesive bonding that is believed to facilitate the development of microfluidic devices. An application to a practical example has been illustrated and results have proved the applicability of the SU-8 adhesive bonding. This technique is applicable for assembling patterned and, more in general, hybrid components. This approach can be extended to large series of materials that show a good adhesion with SU-8, e.g., Cu, SiO_2 , SiN, Au, glass and quartz. Adhesion for other materials of common use in microfluidic devices such as PMMA, PC and SU-8 itself can be improved by plasma treating their surface. Moreover, since SU-8 is broadly used to make microfluidic channels, the use of the same type of photoresist as glue instead of other adhesives is of great advantage. In fact, uniform thermal, chemical and electrical properties can maximize the mechanical stability by reducing the stress due to the mismatch of coefficients of thermal expansion,

improve the compatibility between the chip and the analytes of interest, and facilitate the electro-osmotic flow inside the device.

Acknowledgments This work was supported by the European Commission under the Miracle project within the 7th Framework Programme (FP7-ICT-2009.3.9).

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