<http://www.diva-portal.org>

Postprint

This is the accepted version of a paper published in *Journal of Micromechanics and Microengineering*. This paper has been peer-reviewed but does not include the final publisher proof-corrections or journal pagination.

Citation for the original published paper (version of record):

Karlsson, J., Haraldsson, T., Carlborg, C., Hansson, J., Russom, A. et al. (2012)

Fabrication and transfer of fragile 3D PDMS microstructures.

Journal of Micromechanics and Microengineering, 22(8): 1-9

<http://dx.doi.org/10.1088/0960-1317/22/8/085009>

Access to the published version may require subscription.

N.B. When citing this work, cite the original published paper.

Permanent link to this version:

<http://urn.kb.se/resolve?urn=urn:nbn:se:kth:diva-95357>

Fabrication and transfer of fragile 3D PDMS microstructures

J Mikael Karlsson¹, Tommy Haraldsson¹, Carl Fredrik Carlborg¹,
Jonas Hansson^{2,3}, Aman Russom^{2,3} and Wouter van der Wijngaart¹

¹ Microsystem Technology Laboratory, KTH Royal Institute of Technology, Osquldas väg 10, 10044 Stockholm, Sweden

² Division of Cell Physics, KTH Royal Institute of Technology, Albanova University Center, Roslagstullsbacken 21, 106 91 Stockholm, Sweden

³ Division of Nanobiotechnology, KTH Royal Institute of Technology, Albanova University Center, Roslagstullsbacken 21, 106 91 Stockholm, Sweden

E-mail: jmkarl@kth.se

Received 30 January 2012, in final form 9 April 2012

Published 4 July 2012

Online at stacks.iop.org/JMM/22/085009

Abstract

We present a method for PDMS microfabrication of fragile membranes and 3D fluidic networks, using a surface modified water-dissolvable release material, poly(vinyl alcohol), as a tool for handling, transfer and release of fragile polymer microstructures. The method is well suited for the fabrication of complex multilayer microfluidic devices, here shown for a PDMS device with a thin gas permeable membrane and closely spaced holes for vertical interlayer connections fabricated in a single layer. To the authors' knowledge, this constitutes the most advanced PDMS fabrication method for the combination of thin, fragile structures and 3D fluidics networks, and hence a considerable step in the direction of making PDMS fabrication of complex microfluidic devices a routine endeavour.

(Some figures may appear in colour only in the online journal)

1. Introduction

Microfluidic devices have long been used for miniaturization of common wet laboratory procedures in environmental monitoring, chemical synthesis and medical diagnostics [1]. In particular, microfluidic-based point-of-care devices with sample-in answer-out capabilities have been envisioned for use in primary care and personalized medicine [1]. However, the realization of such devices is difficult as complexity grows nonlinearly with each added on-chip function and as development of novel units for sample preparation and analysis is advancing, more complex integration methods are needed. Polydimethylsiloxane (PDMS) is the most commonly used material for rapid prototyping of microfluidic devices, mainly because of the ease of use and the elastomeric properties that enables valving and simple demoulding. While suffering from some problems with surface modifications and bonding [2], PDMS has unique properties difficult to obtain with other materials. An example is the exceptionally high gas permeability. In microfluidics, the superior gas transport properties of thin PDMS membranes have been used in a wide

range of applications: separation devices [3], perfusion of live cells [3], gas sensors [3], fuel cells [3], bubble removal [4, 5], liquid pumping and priming of microchannels [6].

Fabrication of PDMS devices is typically done using soft lithography, a process consisting of casting and curing of a prepolymer on a microstructured mould, after which the polymerized structure is demoulded, transferred and bonded to a destination substrate [7]. Some steps of the process are difficult to perform without manually handling PDMS layers, e.g. demoulding and vias fabrication. Manual handling of fragile structures, such as thin membranes, results in low device yield since even low levels of strain will rupture or deform the membranes. Several transfer processes [8–10] utilize a carrier to provide protection and control of spatial positions, which could be used to minimize handling of fragile structures and thus increase device yield. However, carrier transfer methods normally require a stepwise increase of bond strength between the polymer and mould, polymer and transfer carrier, and polymer and destination substrate [11]. This typically requires sequential formation and breaking of relatively strong bonds, which due to the high levels of induced

Figure 1. (a) Transfer process using PVA film as a carrier. Steps 1–3 are similar to traditional decal transfer methods. Instead of traditional mechanical removal by carrier, the described method involves dissolution of the carrier, as shown in step 4. (b) Principle of the inhibition process to produce through-hole vias in PDMS, as described by Carlborg *et al* [24].

strain and stress damages the transferred structure, thus making this approach unsuited for fragile membrane manufacturing. Also, the amounts of stress and strain induced in the membrane due to peeling during demoulding and transfer processes will strongly depend on the mechanical properties of the transfer carrier. Peeling of a flexible transfer carrier from a stiff substrate leads to a large peeling angle, which reduces the peeling force required and thus also the amount of stress induced during demoulding and transfer carrier release [12]. In contrast, the use of a stiff transfer carrier leads to smaller peeling angles and higher total adhesion force [13], which requires increased peeling forces to release, and thus increases the risk of structure damage.

A well-known approach to facilitate fabrication of fragile structures is to introduce release layers that dissolve upon contact with a suitable solvent [14]. However, swelling of PDMS in organic solvents has necessitated a substitution of materials involved, and in a particularly promising development, several water-soluble polymers were used as sacrificial materials in microfabrication [15]. An example of a suitable water-soluble polymer that has previously been used for release of microfabricated structures [16–23] is poly(vinyl alcohol) (PVA). We recently presented a method in which PVA for the first time was used as a transfer carrier material [22], where a flexible PVA film was applied on top of fragile PDMS structures, whereafter transfer was done in a pick-and-place manner, with water dissolution of the PVA allowing low-stress release of the PDMS structure in the final step, see figure 1(a). Another recently described fabrication method employed PVA as a mould release layer for fabrication and transfer of wafer-sized PDMS structures using floatation [23].

In multilayered devices, there is a need for vertical interconnects to join channels in different layers and to define inlet and outlet ports. Several alternatives to vias fabrication using traditional manual hole punching have been shown, though these are typically limited in practicality and yield, as

reviewed by Carlborg *et al* [24]. To alleviate yield problems, we recently developed a PDMS vias fabrication method using an amino silane to inhibit polymerization at desired sites [24], see figure 1(b). Briefly, the polymerization of PDMS relies on a freely diffusing catalyst, Pt, in the PDMS prepolymer. To prevent polymerization at the via sites, a top layer that contains amine groups that irreversibly bind and deactivate the Pt is contacted with the PDMS prepolymer. As a result, in thin regions, e.g. in a squeeze film over raised mould features, the number of Pt atoms is sufficiently small to be completely depleted. In addition, rediffusion of Pt from the bulk to the thin layer is obstructed by the mould feature. When the carrier is removed, the unpolymerized PDMS will follow the carrier, thus leaving behind through holes in the solid PDMS layer. While very efficient for fabrication of dense arrays of vias, the stiff glass plate coated with the Pt binding groups prevents high yield fabrication of thin membranes due to excessive stresses induced when removing the plate.

We here report on a refined method capable of *simultaneously* fabricating thin PDMS membranes and densely spaced vias. In this method we utilize three unique PVA properties: (1) the good resistance to solvents other than water [25]; (2) the high surface concentration of hydroxyl groups; and (3) the rapid dissolution of PVA in water. These unique properties enable surface functionalization with silanes to inhibit PDMS polymerization at desired sites and subsequent low stress release of fragile PDMS structures. The combination of PVA surface bound inhibition chemistry and the low stress release of structures via dissolution of PVA, allows for the facile fabrication of closely spaced through-hole arrays in PDMS. Furthermore, by careful manipulation of the density of PVA bound inhibitors, a high yield fabrication of very thin, large area membranes with closely spaced vias and valves in a single process is afforded.

Figure 2. Process flow most suited for the manufacturing of PDMS structures containing both thin membranes and densely spaced vias.

2. Process design, materials and methods

The fabrication process of multilayer structures, containing both fragile membranes and densely spaced vertical interconnects, is divided into five parts; mould preparation, transfer carrier preparation, device layer preparation, transfer and release, and finally assembly of and additional PDMS layer via bonding. Figure 2 illustrates the process flow that was found to be most suited for the manufacturing of PDMS structures containing both thin membranes and densely spaced vias. Several of the process steps were studied in more detail and alternative methods were investigated, as illustrated in figures 3 and 4. All process steps are described in detail in this section.

2.1. Mould preparation

The mould structures were made using photolithographic patterning of spin-coated SU-8 2025 layers on 4" silicon wafer substrates (figures 2A(a)–(d)). Two types of mould structures were fabricated: single level moulds and dual level moulds. Single level moulds define a planar microfluidic channel pattern using a single SU8 layer and dual level moulds define a planar microfluidic channel pattern in a bottom SU8 layer, and vertical fluidic vias in a top SU8 layer. Also, single-level vias-forming pillar moulds were made for optimization of the via fabrication protocol. The moulds were passivated with a fluorocarbon film formed through C_4F_8 plasma deposition using a reactive ion etch (RIE) tool at 30 W for 60 s in order to facilitate demoulding of the PDMS. Both wafer-level and chip-level manufacturing methods were tested. For the latter, the moulds were diced into 14 × 30 mm single-chip moulds.

Figure 3. Illustration of the two methods for preparing PVA carriers. (a) Lamination of PVA film to a support plate. (b) Spin-deposition of PVA solution onto the carrier surface. (d) Submersion of the carrier into an amine silane bath with subsequent (e) rinsing and drying of the carrier, in order to obtain a PDMS polymerization inhibiting substrate.

Figure 4. Two alternative methods for transfer and release, using either a stack consisting of PVA film laminated to a support plate (top) or a PVA spin-coated carrier (bottom).

2.2. Carrier preparation

Figure 3 describes the preparation of the transfer carrier. Three substrates were investigated as PVA carriers: a 2 mm thick glass plate; a 2 mm thick polycarbonate (PC) sheet; and a 250 μm thick PC sheet. Two different PVA coatings were used for the transfer process; a solid PVA film and PVA solution spin-coated onto a carrier. In the first method (figure 3(a)), a 60 μm thick PVA film (The Fishing Bag Ltd, UK) was cut to fit the mould size and pressed onto one of the carrier materials at 70 °C, to create weak adhesion via hydrogen bonds between the PVA hydroxyl groups and either the hydroxyl groups on glass or carbonyl groups on PC. Two methods were used to laminate the film onto the carrier support plate: firstly by manually rolling out the film on the glass/PC plate using a metal cylinder at ca 20 kPa and a rolling rate at ca 1 cm s^{-1} on a hotplate set to 70 °C; and secondly by using a laminator machine (PouchMaster 12, GBC, USA) which pressed the film onto the plate at an elevated temperature. For the second transfer carrier preparation method (figures 3(b), (c)), each transfer carrier substrate was spin-coated at 800 rpm for 60 s with a PVA water solution (5% w/w PVA in water). To evaporate the water, the substrates were placed on a hotplate for 15 min at 70 °C. To achieve a stable transfer carrier, the thin

PC sheet was taped to a glass plate (not illustrated), whereas the glass and thick PC substrates were used without further mechanical support.

Two silanes with very different functions were used to ensure controlled vias formation: (1) the Pt inhibitor aminoethylaminopropyltrimethoxysilane (AEAPS, Z-6020, Dow Corning, USA); and (2) a silane without inhibitory function, 3-methacryloxypropyltrimethoxysilane (MEMO, Z-6030, Dow Corning, USA). The second silane, MEMO, is inert during the PDMS polymerization reaction and does not bind Pt atoms, and is included in the silane mixture in order to control the degree of inhibition. Different samples of each of the different carrier types were treated for 1 h with different surface activation mixtures (figure 3(d)), containing different silane proportions (% w/w) in methanol: 4% AEAPS; 2% AEAPS + 2% MEMO; 1% AEAPS + 3% MEMO; 0.5% AEAPS + 3.5% MEMO, respectively. The transfer carriers were thereafter rinsed with methanol to remove any unbound silanes and dried for 15 min at 70 °C, as seen in figure 3(e).

2.3. Device layer fabrication

The device layer was fabricated according to figures 2C(a)–(c). First, PDMS (Sylgard 184, Dow Corning, US) was mixed at a ratio of 1:10 (curing agent:base), degassed, and poured onto the mould. The transfer carrier was pressed on top of the PDMS, bringing the PVA in contact with the PDMS prepolymer, and the stack was immediately put in an oven at 70 °C. PDMS curing times were 15, 25, 35, 120 min and 19 h, respectively. Figures 4(a), (b) describes two different demoulding processes. After curing, the support plate laminated to the PVA film was easily removed after PDMS curing (figure 4(a)), due to the weak adhesion between the plate and the PVA film. The PVA spin-coated carriers were peeled off from the mould, as illustrated in figure 4(b). The glass carrier support plate taped to the flexible PC sheet was removed prior the demoulding of the PC-PDMS. The flexible PVA-PDMS assembly was thereafter peeled off from the mould.

2.4. Transfer, release and bonding

After oxygen plasma treatment (15 s at 40 W, FEMTO A, Diener electronic GmbH), the PDMS structures were irreversibly bonded to a glass or silicon destination substrate (figures 4(c), (d)). In an additional series of experiments, non-plasma-treated PDMS was transferred to the silicon and glass substrates to elucidate the efficacy of the transfer protocol when reversible bonding between the destination substrate and the PDMS is desired. After transfer, the transfer carrier was removed from the PDMS through wet peeling, i.e. a small amount of water was applied at the PVA-PDMS interface to dissolve the PVA while the carrier was peeled off (figures 4(e), (f)), leaving the PDMS structure intact on the destination surface. When the carrier was removed, the unpolymerized PDMS parts on top of the via mould features adhered to the carrier, thus leaving open vias. In an alternative method, the whole structure was submerged in water until the PVA was dissolved. After drying, the PDMS structure remained transferred onto the destination glass surface.

A second PDMS layer was fabricated in a standard process without inhibition, in which inlet and outlet ports were made using hole punching. The two PDMS layers were treated with oxygen plasma, whereafter they were put in contact for bonding (figure 2E). Polythene tubes (Portex®, Smiths Medical International Ltd, UK) were thereafter inserted into the top layer ports.

3. Results and discussion

3.1. Release of fragile PDMS structures

For demoulding of the fragile PDMS structure (figures 4(a), (b) by peeling, it was found that stiff transfer carriers were difficult to remove from the mould and resulted in high levels of induced strain into the PDMS and thus membrane rupture. It was therefore crucial to use a flexible carrier, i.e. the PVA film or the spin-coated flexible PC sheet, which resulted in successful low-stress demoulding.

Dry peeling of the transfer carrier from the PDMS during the transfer carrier release step (figures 4(e), (f) always led to membrane rupture. Unlike dry peeling, the wet peeling approach utilizes dissolution of the PVA, resulting in minimal strain in the membrane, leaving the fragile structure intact. As observed for the demoulding step, the transfer carrier release step was not successful for stiff transfer carriers in combination with peeling, which left PVA dissolution by submersion of the stack in water as the only practical approach. In contrast, flexible transfer carriers, i.e. the soft PVA film and the thin PC sheet, provided successful release from PDMS structures using wet peeling, within one minute for chip-sized structures up to 10 min for wafer-sized structures.

In summary, non-disruptive peeling of fragile structures during demoulding and release of transfer carriers is ensured firstly using a flexible transfer carrier, to facilitate peeling at large angles, and removal of the bond between the carrier and PDMS via PVA dissolution.

3.2. Fabrication and transfer bonding of fragile PDMS membranes using PVA

The suitability of the above-described methods were evaluated by fabrication and transfer of PDMS structures containing fragile, large-area suspended PDMS membranes, with a thickness of 35 μm and an area of 1 cm^2 . A summary of the results using the different methods is found in table 1.

In the first set of experiments, the inhibition process was left out, in order to evaluate the process for fabrication and transfer of PDMS membranes. The lamination of PVA film to both glass and PC resulted in successful adhesion of the film to the transfer carrier support plate. Since the transfer carrier support plate was easily removed after PDMS curing, this approach provides a flexible carrier compatible with wet peeling, which was successful for transfer of membranes. Flexible PVA spin-coated PC sheets also resulted in successful membrane transfer using wet peeling release.

Submersion to obtain transfer carrier release from the PDMS structures was impractical and required long reaction times. Complete dissolution of the PVA film for PDMS structure release was time consuming and still after two hours there were PVA residues on the PDMS surface. For the PVA spin-coated carriers, a total dissolution time of 30–60 min for chip-sized substrates and up to 8 h for wafer-sized substrates was required.

Overall, the lamination method resulted in a much faster process as compared with the spin-on method due to the difference in carrier preparation time. As seen in figure 5, both chip-sized and wafer-sized structures containing membranes were successfully fabricated and transferred using the PVA film for transfer carriers. However, difficulties of manual handling of thin, 4" wafer-sized structures often lead to wrinkle formation in the contact between the PDMS substrate and the destination substrate surface, which reduced yield.

It was observed that transfer of PDMS structures when using PVA as a carrier material is insensitive to adhesion strength between the components and destination substrate; both low adhesion strength between untreated PDMS and

Table 1. Overview of the investigated materials and methods, indicating the combinations allowing successful fabrication of 35 μm thin, 1 cm^2 large area membranes (■) and densely packed vias (●). The optimal combination of material and methods to achieve both membranes and vias is highlighted with dotted boxes.

Surface activation mixture for vias	Transfer carrier material				
	1. PVA film laminated onto glass	2. PVA film laminated onto glass	3. Liquid PVA spun onto glass	4. Liquid PVA spun onto thick PC	5. Liquid PVA spun onto flexible PC
a. 4% AEAPS	⊗	⊗	●	●	●
b. 2% AEAPS + 2% MEMO	⊗	⊗	●	●	●
c. 1% AEAPS + 3% MEMO	⊗	⊗	●	●	■●
d. 0.5% AEAPS + 3.5% MEMO	⊗	⊗	⊗	⊗	■⊗
Demoulding					
e. Dry peeling	■⊗	■⊗	⊗●	⊗●	■●
Release from carrier					
f. Dry peeling of carrier	⊗	⊗	●	●	●
g. Wet peeling of carrier	■⊗	■⊗	⊗●	⊗●	■●
h. Submersion into water	■⊗	■⊗	(■●)	(■●)	(■●)
Substrate size					
i. Single chip	■⊗	■⊗	⊗●	⊗●	■●
j. Wafer	■⊗	■⊗	(■●)	(■●)	(■●)
<div> <div>■ = 30 μm membrane</div> <div>● = densely packed vias</div> <div>⊗ = membrane not achieved</div> <div>⊗ = vias not achieved</div> <div>■● = both membrane & vias achieved</div> <div>() = difficult / low yield</div> </div>					

destination surfaces as well as bonding strength, obtained via standard oxygen plasma treatment (figures 4(c), (d), gave successful results for release of PDMS structures onto both silicon and glass destination substrates.

The approach of using PVA as a transfer material has several distinct benefits over previous polymer transfer methods. Unlike dry mechanical peeling of the carrier substrate from the transferred components, the dissolution of the carrier substrate from the components prevents deformation of fragile structures. The methods that involve wet peeling or complete dissolution are therefore well suited for the transfer of mechanically fragile components. Moreover, the transfer bonding does neither require elevated temperatures nor organic solvents, both of which would limit the use of this method when combined with biofunctionalized surfaces.

3.3. Fabrication of inter-layer via connections using PVA

During the silane treatment of the PVA film on the carrier support plate (figure 3(d)), it was difficult to keep the PVA

film from releasing from the carrier support plate. Therefore, the inhibition experiments were performed using the PVA spin-coated transfer carriers. The PDMS was still uncured after 15 min, though a polymerized structure with vias was observed after 25, 35, 120 min and also after the 19 h overnight test. As long as the PDMS had time to polymerize, the vias were formed irrespectively of the baking time, which indicates that there is a sufficient amount of amines to deactivate all Pt-atoms in the thin squeeze film. The pre-baking time, i.e. the time from application of cover plate until exposure to polymerization temperature, is an important factor, as shown by Carlborg *et al* [24] since the Pt atoms are more mobile in a liquid environment and therefore bind to the inhibition plate at a higher rate before the prepolymer starts to gelify, which will ultimately lead to a situation where Pt atoms are depleted from the membrane regions of the mould, preventing membrane polymerization. The minimization of the pre-baking time in these tests was therefore an important step towards achieving conditions in which both a fully polymerized membrane and open vias are obtained.

Figure 5. Transfer processes and results of chip sized structures (top) and wafer-sized structures (bottom) using PVA film as a carrier. (a), (b) PDMS clamped between a SU-8/Si mould and a PVA film-covered glass plate. (c), (d) The PVA-PDMS assemblies after demoulding. (e), (f) Assemblies were transferred to silicon substrates, onto which release was done. The arrows implicate the direction of peeling of the film. (g), (h) The transferred and released PDMS structures resting on silicon surfaces. Photos (c), (d) have been treated with contrast enhancement for clarity.

For simultaneous fabrication of membranes and vias, the polymerization inhibition has to be sufficient to prevent squeeze-film polymerization above the raised vias mould features, while still allowing thin membrane structures to polymerize. Using PVA instead of the smooth glass surface, used by Carlborg *et al* [24], results in a surface area enlargement in comparison to glass due to the PVA surface roughness, which in turn gives an increased amount of binding sites per projected area and thus an enhanced inhibition effect. Therefore, it was crucial to use the inert silane, MEMO, which does not bind Pt atoms, in order to reduce the surface concentration of the inhibitory silane. In this manner, sites occupied by the inert silane are unavailable for the inhibitory silane, leading to a lower surface concentration, hence less capacity for binding Pt. A 100% yield of open via formation using several types of pillar array moulds were observed for the composition of 4% AEAPS dissolved in methanol, as seen in figure 6. A surface activation mixture of 0.5% AEAPS + 3.5% MEMO resulted in no via formation, due to the low surface concentration of AEAPS. Both glass and PC carriers successfully resulted in open through-hole vias.

3.4. Fabrication of PDMS membranes and through-hole vias using PVA

Structures containing both through-hole vias and a membrane were successfully fabricated and transferred using the inhibition method in combination with PVA-based transfer. We fabricated a demonstration lab-on-chip device consisting of a PDMS structure with a 35 μm thin, 1 cm^2 large area suspended membrane as well as nine vias, as seen in figure 7. Fabrication of this structure required dilution of the inhibitory silane surface concentration; successful results with the two level mould were obtained when using a surface activation

Figure 6. Inhibition results using pillar moulds and a silanized PVA-PC cover plate, showing perfect yield of the vias fabrication method in four different regions of the PDMS structure. The moulds consisted of 50 μm high pillars of various sizes.

mixture of 1% AEAPS + 3% MEMO on a flexible PC sheet with spun-on PVA. Mixtures with larger amount of AEAPS led to incomplete membrane polymerization, whereas a lower amount of AEAPS led to blocked vias. As described in section 3.3, the pre-baking time was important for optimization of the inhibition protocol. To keep stringent control of the degree of inhibition, i.e. to minimize the loss of Pt atoms in the membrane structure, the pre-baking time was kept to <1 min, which resulted in an intact membrane while simultaneously resulting in vias formation.

Attempts to fabricate wafer-sized structures with membranes and vias were successful using PVA spun onto transfer carriers, though unpractical as the transfer carrier

Figure 7. Transfer and inhibition using a silanized PVA-PC cover plate, showing (a) the result after tuning the degree of inhibition to allow simultaneous fabrication of (b) through-hole vias and (c) a 35 μm thin, 1 cm^2 large area membrane. This structure was assembled with a second layer, containing structures for pneumatic valving, shown in (d). (e) Microfluidic channels were visualized using dyed water introduced via the tubes. (f), (g) The functionality of the fluidic connections between the layers was investigated using the integrated pneumatic valves.

release step was performed using time-demanding submersion into a water bath. Vias were difficult to obtain evenly over the whole substrate, possibly due to uneven distribution of PVA and AEAPS on the transfer carrier surface, or through uneven contact of the mould and the inhibitory carrier surface.

It was previously reported that the inhibition typically results in a PDMS structure with a sticky surface [24]. Although this lead to tape-like adhesion, the ability to achieve covalent bonding to a second PDMS layer relies on reinitiation via Pt-addition to unpolymersed regions [24]. For surface activation mixtures containing 4% AEAPS, the inhibited surface was sticky. However, it was observed that the top surface was not sticky after using a surface activation mixture of 1% AEAPS + 3% MEMO, which may be a result of the optimization of the vias protocol to prevent over-inhibition of the membrane. The top surface was also investigated with a contact angle measurement in order to elucidate surface wettability. No change of contact angle was seen for PDMS structures released by peeling from PVA film or PVA spin-coated transfer carriers as compared to PDMS surfaces not exposed to PVA or inhibition. In contrast, complete dissolution of the PVA film using submersion into a water bath left a PDMS surface which was difficult to fully cleanse from PVA residues, thus resulting in a surface with reduced contact angle as compared to native PDMS.

We thereafter used oxygen plasma assisted bonding of the inhibited surface to achieve a covalent bond between a previously transferred PDMS layer to a second PDMS layer, forming a two-layered lab-on-chip device (figure 7). To test the bond quality, the PDMS top layer was peeled from the bottom layer, and the site of device rupture was investigated. In all cases studied, the top layer broke in the PDMS bulk, which implies that neither the inhibition nor the PVA modification prevented good interlayer bonding. The assembled PDMS structures formed a device, containing channels in two layers and pneumatic valves [26]. Also, a second chamber was

situated on top of the membrane, thus totally encasing an integrated suspended membrane into the chip.

4. Conclusions

A novel method for low-stress transfer of fragile PDMS layers was successfully demonstrated, using only mild conditions well tolerated by PDMS. This was achieved using PVA, a water dissolvable transfer carrier material. Fragile PDMS structures of single-chip and wafer sizes are protected during transfer steps such as demoulding, handling and release in this high-yield process. Low-stress demoulding is achieved by the use of a flexible PVA carrier, which after transfer is released under low-stress conditions from the structure by dissolution. Also, the method is used for producing interlayer through-hole connections, resulting in a method for simultaneous fabrication of suspended PDMS membranes and 3D fluidic networks. This was demonstrated through the successful fabrication of a lab-on-chip structure, containing nine interlayer via connections and a fragile, suspended membrane with a thickness of 35 μm and an area of 1 cm^2 . To the authors' knowledge, this method constitutes the most advanced PDMS fabrication of thin structures combined with 3D fluidics networks, and constitutes a large step in the direction of making PDMS fabrication of complex microfluidic devices a routine pursuit.

Acknowledgment

This work has been financially supported by the European Union through the seventh framework programme project InTopSens (www.intopsens.eu).

References

- [1] Yeo L Y, Chang H-C, Chan P P Y and Friend J R 2011 Microfluidic devices for bioapplications *Small* **7** 12–48

- [2] Zhou J, Ellis A V and Voelcker N H 2010 Recent developments in PDMS surface modification for microfluidic devices *Electrophoresis* **31** 2–16
- [3] De Jong J, Lammertink R G H and Wessling M 2006 Membranes and microfluidics: a review *Lab Chip* **6** 1125–39
- [4] Karlsson J M, Haraldsson T, Laakso S, Virtanen A, Maki M, Ronan G and van der Wijngaart W 2011 PCR on a PDMS-based microchip with integrated bubble removal *Proc. 16th Int. Conf. on Solid-State Sensors, Actuators and Microsystems (Beijing, China)* pp 2215–18
- [5] Xu J, Vaillant R and Attinger D 2010 Use of a porous membrane for gas bubble removal in microfluidic channels: physical mechanisms and design criteria *Microfluidics Nanofluidics* **9** 765–72
- [6] Eddings M A and Gale B K 2006 A PDMS-based gas permeation pump for on-chip fluid handling in microfluidic devices *J. Micromech. Microeng.* **16** 2396–402
- [7] Xia Y and Whitesides G M 1998 Soft lithography *Angew. Chem. Int. Ed.* **37** 550–75
- [8] Childs W R and Nuzzo R G 2002 Decal transfer microlithography: a new soft-lithographic patterning method *J. Am. Chem. Soc.* **124** 13583–96
- [9] Meitl M A, Zhu Z-T, Kumar V, Lee K J, Feng X, Huang Y Y, Adesida I, Nuzzo R G and Rogers J A 2006 Transfer printing by kinetic control of adhesion to an elastomeric stamp *Nat. Mater.* **5** 33–8
- [10] Onoe H, Iwase E, Matsumoto K and Shimoyama I 2007 3D integration of heterogeneous MEMS structures by stamping transfer *Proc. IEEE 20th Conf. MEMS* pp 175–78
- [11] Chen L, Degenaar P and Bradley D D C 2008 Polymer transfer printing: application to layer coating, pattern definition, and diode dark current blocking *Adv. Mater.* **20** 1679–83
- [12] Kendall K 1975 Thin-film peeling-the elastic term *J. Phys. D: Appl. Phys.* **8** 1449–52
- [13] Kendall K 1975 Crack propagation in lap shear joints *J. Phys. D: Appl. Phys.* **8** 512–22
- [14] Madou M J 2002 *Fundamentals of Microfabrication* 2nd edn (Boca Raton: CRC Press)
- [15] Linder V, Gates B D, Ryan D, Parviz B A and Whitesides G M 2005 Water-soluble sacrificial layers for surface micromachining *Small* **1** 730–36
- [16] Schaper C D 2004 Water-soluble polymer templates for high-resolution pattern formation and materials transfer printing *J. Microlith. Microfab. Microsyst.* **3** 174–85
- [17] Guan J, He H, Hansford D J and Lee L J 2005 Self-folding of three-dimensional hydrogel microstructures *J. Phys. Chem. B* **109** 23134–37
- [18] Ferrell N, Woodard J and Hansford D 2007 Fabrication of polymer microstructures for MEMS: Sacrificial layer micromolding and patterned substrate micromolding *Biomed. Microdevices* **9** 815–21
- [19] Addae-Mensah K A, Reiserer R S and Wikswo J P 2007 Poly(vinyl alcohol) as a structure release layer for the microfabrication of polymer composite structures *J. Micromech. Microeng.* **17** N41–46
- [20] Higueta N, Dai Z, Kaletunç G and Hansford D J 2008 Fabrication of pH-sensitive microparticles for drug delivery applications using soft lithography techniques *Mater. Res. Soc. Symp. Proc.* **1095** 7–12
- [21] Wang G-J, Ho K-H and Hsueh C-C 2008 Biodegradable polylactic acid microstructures for scaffold applications *Microsyst. Technol.* **14** 989–93
- [22] Karlsson J M, Haraldsson T, Carlborg C F, Stemme G and van der Wijngaart W 2010 Transfer bonding of microstructures and fabrication of fragile PDMS membranes using water dissolvable film *The 14th Int. Conf. on Miniaturized Systems for Chemistry and Life Sciences (MicroTAS 2010) (Groningen, The Netherlands)* pp 1202–04
- [23] Karlsson J M, Haraldsson T, Carlborg C F and van der Wijngaart W 2011 Low-stress wafer-level transfer bonding of polymer layers using floatation *Proc. 16th Int. Conf. on Solid-State Sensors, Actuators and Microsystems (Beijing, China)* pp 418–21
- [24] Carlborg C F, Haraldsson T, Cornaglia M, Stemme G and van der Wijngaart W 2010 A high-yield process for 3-d large-scale integrated microfluidic networks in PDMS *J. Microelectromech. Syst.* **19** 1050–57
- [25] Amjad Z 1998 *Water Soluble Polymers: Solution Properties and Applications* (New York: Plenum Press)
- [26] Unger M A, Chou H-P, Thorsen T, Scherer A and Quake S R 2000 Monolithic microfabricated valves and pumps by multilayer soft lithography *Science* **288** 113–16