

# HYBRID FABRICATION OF MICROFLUIDIC CHIPS BASED ON COC, SILICON AND TMMF DRY RESIST

Kiril Kalkandjiev<sup>1</sup>, Roland Zengerle<sup>1</sup> and Peter Koltay<sup>1,2</sup>

<sup>1</sup>University of Freiburg - IMTEK, Germany

<sup>2</sup>BioFluidiX, Freiburg, Germany

## ABSTRACT

We describe the hybrid fabrication of silicon-plastic microfluidic chips based on machining of Cyclic Olefin Copolymer (COC), standard silicon processing and TMMF lithography. The combination of different processes enables an individual material selection leading to significant reduction of the manufacturing costs. We demonstrate the potential of the hybrid technology by manufacturing and testing a 24-channel TopSpot dispenser [1] which consists of an intermediate silicon layer, a COC interface and a TMMF sealing lid. Characterization studies show that TMMF lamination is ideally suited for the sealing of silicon microchannels showing numerous advantages over adhesive-based approaches, thermal and anodic bonding.

## INTRODUCTION

In contrast to sensor devices manufactured in batches of thousands, BioMEMS devices are often requested in smaller numbers, which makes low-cost fabrication challenging. While silicon processes go for higher densities and sub-micron structures, the size of a typical microfluidic device is given by the relatively large interface that requires thicker materials for operation with laboratory-scale equipments [2]. As a result, polymer techniques have been adapted for the requirements of BioMEMS. The main advantages of polymers – lower cost compared to silicon and glass, wide range of available materials and suitability for prototyping and high-volume production, have been frequently pointed out [3-5]. On the other hand, silicon components have the advantage of higher accuracy as a consequence of the established MEMS processes. Silicon is the material of choice when components with high-aspect ratio, sharply defined edges or high mechanical stiffness are required. It is furthermore advantageous when it comes to the integration of sensing or actuating elements [6].

Silicon micromachining is often combined with silicon-Pyrex anodic bonding to provide the chip with the necessary peripheral components. The machining of Pyrex, however, together with the anodic bonding and dicing of the stacked assembly is a time-consuming process that accounts for a very significant part of the manufacturing costs. At the same time, the properties of the Pyrex components and the high quality of the anodic bond are

not necessarily required for a typical lab-on-a-chip. Thus, silicon-plastic hybrid integration approaches should be considered as an open alternative for cost saving. A major aspect of the hybrid integration is the precise alignment and liquid-tight bonding of components possessing different chemical nature. Usually, bonding of dissimilar materials is based on the use of adhesives. In microfluidics, however, direct bonding is more popular [7] but it is mostly restricted to materials of the same type.

Few methods for direct silicon-plastic bonding such as localized heating with resistive heaters [8,9], plasma-assisted silicon-PDMS bonding [10], mechanical interlocking [11] or bonding of polymer to a black-silicon surface [12] have been reported to be suitable for the fabrication of hybrid liquid-tight assemblies.

In this paper, we describe an alternative fabrication technique using TMMF dry resist as a sealing lid for silicon microchannels and an adhesive tape (3M 9965) for bonding of the silicon core to a COC interface. Finally, we show the application of this technique to the manufacturing of 24-channel TopSpot printheads used for the formation of microarrays [1].

## FABRICATION

The printhead contains 24 reservoirs which can be filled with different samples using standard laboratory equipment. Each reservoir is connected to a corresponding nozzle via a capillary microchannel. The nozzles are arranged in a 6x4 grid with a pitch of 500  $\mu\text{m}$ . A one-to-one format conversion from the 4.5 mm pitched reservoirs to the microarray format is provided by the microchannels. A piezo-driven piston generates a pressure pulse by compressing an air chamber which causes the parallel ejection of a single droplet out of each nozzle (Fig. 1).

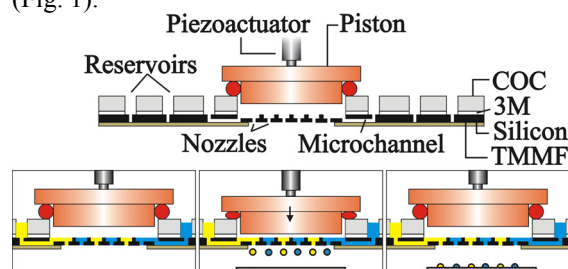


Figure 1: A schematic cross-section and operation principle of the hybrid TopSpot printheads.

The fabrication of the hybrid printheads is depicted in Fig. 2. The fabrication scheme involves: 1) Fabrication of the microfluidic network using standard silicon micromachining, 2) Selective sealing of the microchannels using wafer-level lamination of TMMF and photolithography and 3) Chip-level assembly of the microfluidic components onto the COC interface via laser-cut 3M tape.

The core of the printhead is the silicon layer featuring microfluidic structures etched using a standard silicon DRIE process. Sealing of the microchannels was realized by wafer-level lamination of the epoxy-based TMMF dry resist (55  $\mu\text{m}$  thick, TOK, Japan). The lamination was performed on a DuPont laminator (Riston HRL) at a temperature of 60  $^{\circ}\text{C}$ , a pressure of approx. 1 bar and a roll speed of 1 m/min. These parameters have proved to be useful for sealing without distortion of the channel cross-section by sagging of the cover lid. Subsequently, cross-linking of the dry film is initiated using i-line with a dose of 150  $\text{mJ}/\text{cm}^2$ . A printed shadow mask was used to prevent cross-linking around the nozzles and enable selective removal of the dry film during the development. Post-exposure bake was performed on a hot-plate for 10 min. at 90  $^{\circ}\text{C}$ , followed by 45 min. at 150  $^{\circ}\text{C}$ . The development of the dry film was performed in two steps for complete removal of the non-exposed areas. First, TMMF was developed in PGMEA (propylene glycol methyl ether acetate) for 7 min. under ultrasonic actuation and rinsed with deionized water. Subsequently, a spin-dry process was carried out to expel the developed dry film out of the microchannels. The spinning, however, leads to a deposition of the developed TMMF on the nozzles. Thus, a second PGMEA treatment was necessary for complete removal of the non-exposed dry film. After dicing, the chips were assembled onto the COC interface via double-sided pressure sensitive adhesive tape (3M 9965). The used tape is initially inactive and allows alignment during contact of the two surfaces, followed by application of a slight pressure for bonding to take place. The tape was pre-structured by laser-cutting to open up access holes to the reservoirs.

Before use, the printheads should be surface-modified. Hydrophilic channels are required for capillary transport of the samples from the reservoirs to the corresponding nozzles. In contrast, the non-wetting properties of the nozzle array ensure homogenous droplet ejection. Since surface treatment degrades after several operations, a fast and easily applicable re-coating procedure is essential for reproducible performance. Here we present a method for selective surface modification which we applied for functionalization of the hybrid printheads.

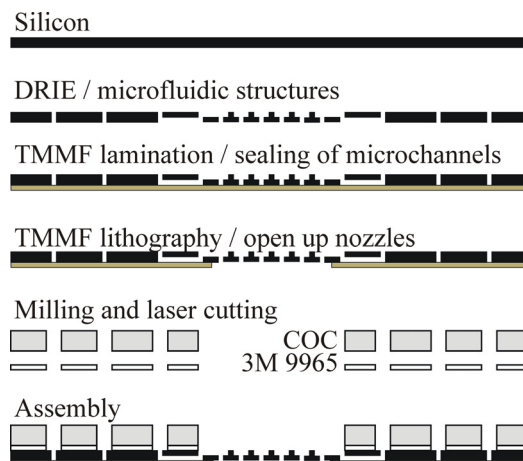


Figure 2: Hybrid fabrication based on COC, silicon and TMMF dry resist.

For hydrophilization, 2 percent water solution of the alkaline liquid Hellmanex II (Hellma, Germany) was injected through the channels for 1 min. at a pressure of 0.8 bar and a temperature of 80  $^{\circ}\text{C}$ . After the treatment with Hellmanex II, the nozzle area was incubated with Perfluorodecyltrichlorosilane (ABCR, Germany) for 1 min. During incubation, the hydrophilic channels were capillary filled with glycerol solution that prevents penetration of the silane into the channels.

## RESULTS AND DISCUSSION

### Sealing

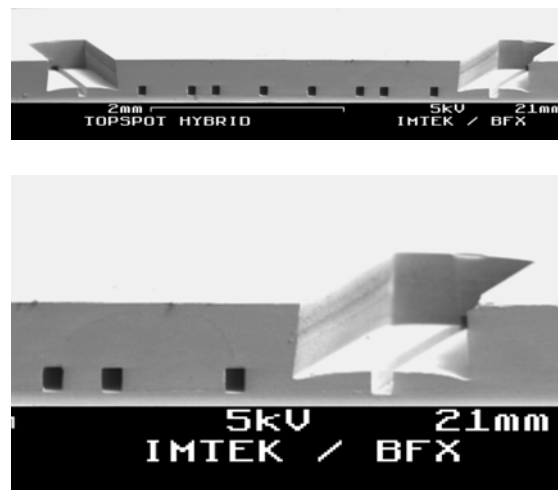


Figure 3: Cross-section of silicon microchannels sealed with TMMF dry resist. The used parameter set ( $t=60$   $^{\circ}\text{C}$ ,  $v=1$  m/min,  $p \sim 1$  bar) assured liquid-tight sealing without distortion of the channel cross-section.

Figure 3 shows a cross-section of the chip prior to the assembly. The silicon microchannels were

sealed without sagging of the cover lid, free of leakage and delamination within the tested pressure range of up to 8 bar. A mechanical removal of the cross-linked dry film from the substrate was not possible. Thus, the TMMF-based sealing can be considered as irreversible.

The printheads were tested for liquid-tight sealing by filling the reservoirs in a checkerboard pattern with a fluorescence dye (Rhodamin 6G) and deionized water. The printheads showed neither obvious leakages nor any cross-talk between adjacent microchannels (Fig. 4).

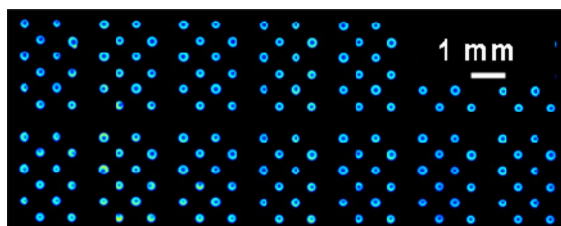


Figure 4: Spotted checkerboard pattern for verification of the leak tightness.

#### Removal of the non-exposed TMMF

A big advantage of TMMF dry resist as a sealing material is the possibility for accurate and selective removal (“reopening”) of the lid with a single photolithographic step using only a conventional aligner. Since the openings are patterned subsequently, there are no alignment requirements for the lamination process. We use this possibility for the simultaneous reopening of all nozzle arrays. The two-step developing process assured the existence of nozzles without any TMMF residuals (Fig. 5).

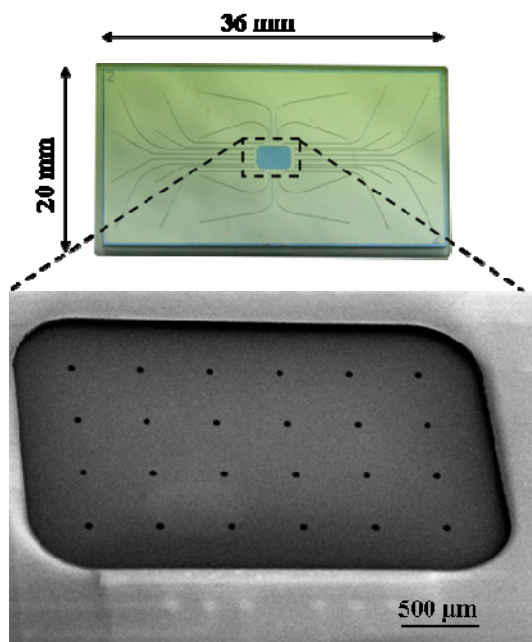


Figure 5: Bottom side of the hybrid printhead with detailed view on the nozzle area.

#### Surface modification and test

The technique for selective surface modification was experimentally investigated using ten hybrid printheads in a total of 100 coating experiments. Every coating experiment was followed by a filling and dispensing test to prove the capillarity of the microchannels and the quality of the hydrophobic coating. A stroboscopic camera was used to measure the homogeneity of the droplet diameter and speed during flight and an E-Vision microarrayer [13] for spot observation immediately after printing. Non-ideal surface properties would lead to one or more of the following failures: 1) satellite droplets, 2) missing droplets, 3) wetting of the nozzle area, 4) improper deviation of the droplet velocity, 5) improper spot offset on the slide ( $> 50 \mu\text{m}$ ) and 6) no capillary filling.

The surface modification showed a yield of 90 % using water as dispensing medium. 4 % of the tests fail at the capillary filling, 3 % at improper velocity deviation and 3 % at improper spot offset on the slide. Figure 6 shows an instantaneous image of optimal dispensing after successful surface treatment.

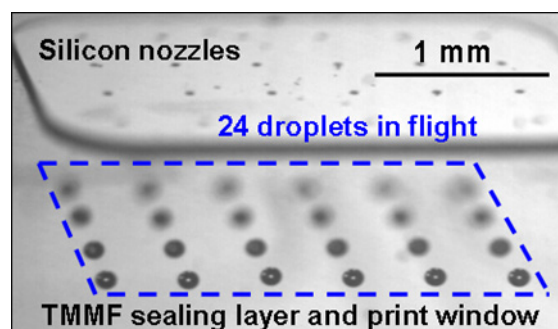


Figure 6: Stroboscopic image of the droplets in flight. Satellite-free dispensing of a complete 6x4 array, no wetting of the nozzle area and no velocity deviation.

Since the nozzles are fabricated in silicon and only the peripheral components (reservoirs, sealing lid) are replaced by polymer materials, the dispensing performance of the hybrid printheads remains unchanged compared to the commercially available silicon-glass printheads (droplet volume about 1 nL, CVs for droplet diameter lower than 1 % per nozzle and lower than 1.5 % over all 24 nozzles of a printhead for printing of oligonucleotide and protein solutions).

#### CONCLUSIONS

A fabrication method for hybrid microfluidic devices was presented. Advantages of silicon, COC and the epoxy-based dry photoresist TMMF were combined successfully to enable a proper material selection for the individual chip components with

regard to reduction of the manufacturing costs. We showed the capability of the hybrid fabrication by manufacturing a small batch of TopSpot hybrid printheads consisting of COC reservoirs, a silicon microfluidic layer and a TMMF sealing lid. Characterization of TMMF showed that it is ideally suited for selective, irreversible sealing of silicon microchannels. All microchannels were liquid-tight sealed in a wafer-level process and all nozzles were simultaneously "reopened" without leaving any residuals from the dry film. The combination of silicon with polymer materials instead of the established silicon-Pyrex anodic bonding results in a cost reduction of more than 50 % for a standard 24-channel TopSpot printhead. The fabrication scheme presented here can be easily adapted to hybrid systems dedicated to specific microfluidic applications. Based on it, there is a variety of possibilities for silicon-plastic integration that opens new opportunities for cost-efficient prototyping and manufacturing.

#### ACKNOWLEDGEMENT

This work was supported by the German Ministry of Science and Education (BMBF) and MicroMountains Applications AG.

#### REFERENCES

- [1] O. Gutmann, R. Kuehlewein, S. Reinbold, R. Niekrawietz, C. P. Steinert, B. de Heij, R. Zengerle, M. Daub: "A highly parallel nanoliter dispenser for microarray fabrication"; *Biomed. Microdevices*; vol. 6, pp. 131-137; 2004.
- [2] C. K. Fredrickson, Z. H. Fan: "Macro-to-micro interfaces for microfluidic devices"; *Lab Chip*; vol. 4, pp. 526-533; 2004.
- [3] D. C. Duffy, J. C. McDonald, O. J. A. Schueller, G. M. Whitesides: "Rapid prototyping of microfluidic systems in poly(dimethylsiloxane)"; *Anal. Chem.*; vol. 70, pp. 4974-4984; 1998.
- [4] P. Abgrall, V. Conedera, H. Camon, A. M. Gue, N. T. Nguyen: "SU-8 as a structural material for labs-on-chips and microelectromechanical systems"; *Electrophoresis*; pp. 4539-4551; 2007.
- [5] H. Becker, C. Gartner: "Polymer microfabrication technologies for microfluidic systems"; *Anal Bioanal Chem*; vol. 390, pp. 89-111; 2007.
- [6] P. Abgrall, A. M. Gue: "Lab-on-chip technologies: making a microfluidic network and coupling it into a complete microsystem - a review"; *J. Micro-mech. Microeng.*; vol. 17, pp. R15-R49; 2007.
- [7] C. W. Tsao, D. L. Devoe: "Bonding of thermoplastic polymer microfluidics"; *Microfluid. Nanofluid.*; vol. 6, pp. 1-16; 2009.
- [8] K. Wulff: "Hybride Drucksensoren aus Kunststoff und Glas nach dem AMANDA-Verfahren"; 2001.
- [9] Y. C. Su, L. W. Lin: "Localized bonding processes for assembly and packaging of polymeric MEMS"; *Ieee Transactions on Advanced Packaging*; vol. 28, pp. 635-642; 2005.
- [10] S. Bhattacharya, A. Datta, J. M. Berg, S. Gangopadhyay: "Studies on surface wettability of poly(dimethyl) siloxane (PDMS) and glass under oxygen-plasma treatment and correlation with bond strength"; *J. Microelectrochem. S.*; vol. 14, pp. 590-597; 2005.
- [11] M P Larsson, R R A Syms, A G Wojcik, "Improved adhesion in hybrid Si-polymer MEMS via micromechanical interlocking"; *J. Micromech. Microeng.*; pp. 2074-2082; 2005.
- [12] M. Stubenrauch, M. Fischer, C. Kremin, M. Hoffmann, J. Mueller: "Bonding of silicon with filled and unfilled polymers"; *Micro & Nano Letters*; vol. 2, pp. 6-8; 2007.
- [13] BioFluidiX;  
[http://www.biofluidix.com/uploads/media/TopSpot\\_E-Vision\\_02.pdf](http://www.biofluidix.com/uploads/media/TopSpot_E-Vision_02.pdf) (November 15, 2009)