Opportunities for SUEX dry laminate resist in microfluidic MEMS applications

Donald W Johnson[a], Jost Goettert[b], Varshni Singh[b] and Dawit Yemane[b]

[a]DJ DevCorp, 490 Boston Post Rd, Sudbury, MA 01776
[b]Louisiana State University, Center for Advanced Microstructures & Devices (CAMD), 6980 Jefferson Hwy, Baton Rouge, LA 70806

don@djdevcorp.com

Introduction

Microfluidics applications can benefit from a dry film approach in multiple ways – particularly in the preparation of multi-layer, multi-level fluidic channels and structures on patternable substrates and covers [1,2,3,4]. Of special benefit is the extreme simplicity in the use of the dry film sheets. A standard lamination approach allows one to apply the resist sheets as well as image them within minutes and, in addition, provides a coating with no edge bead and no solvent gradient through the film. With the added benefit of reduced baking times, and the use of polymer substrates, which eliminates the debonding of the final devices from a rigid silicon or glass base, the approach results in processing times of about 2 days for a 3-layer, all-polymer device. Furthermore, multi-level structures can be imaged at one time with extreme precision and sub-micrometer feature details when using x-ray exposures. Research examples from densely packed, high-aspect ratio post arrays [5], polymer microreactors [6], and biosensor devices [7] show outstanding structure fidelity as well as very high aspect ratios and structural details of sub-micron resolution. Recently, first results have been published [8,910] showing examples of structure quality and possible applications.

Experimental

A multi-layer fabrication process leading to a fully sealed device is illustrated in Fig. 1.

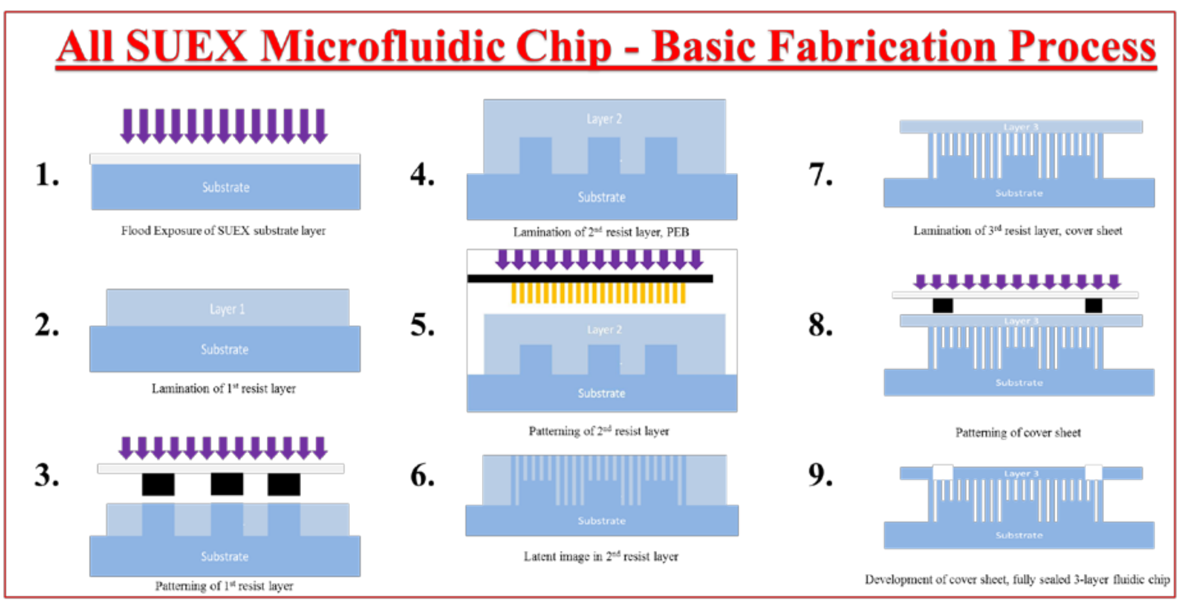
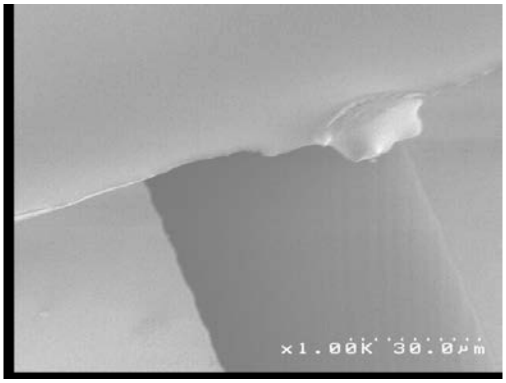


Fig. 1:Basic fabrication steps applied in making fully sealed microfluidic devices.

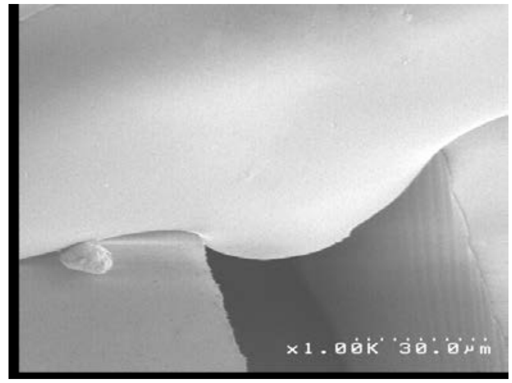
In the first step a SUEX layer functioning as a substrate is UV flood exposed to form a cross-linkable base. In this step one can also use an optical mask defining a pattern in the first layer, for example with fluidic inlets and outlets. Typical layer thickness is 100 to 250µm and exposure dose ranges from 500 to 1500mJ/cm^2. The backside of the SUEX substrate is covered with a PET sheet which can be used as a temporary carrier allowing one to pick off or peel individual parts after completion of the full process without cutting. Next, without heating (PEB'ing) the substrate, a first 100-250µm thick SUEX resist layer is laminated (roll laminator temperature set to 70˚C) onto the substrate (Step 2, Fig. 1) followed by UV- lithography of a base pattern (Step 3, Fig. 1). It is advantages to define alignment marks in this layer or also in the substrate to which subsequent layers can be aligned. After exposure of both the substrate layer and the base layer the stack is then post exposure baked to crosslink the exposed regions in both layers. It is optional to leave the top protective PET on during the PEB. We have used 85ºC for 1hr as our standard PEB condition starting at 50 ºC and ramping up to 85 ºC in 30min. For best flatness we have found it necessary to slowly cool the PEB'd substrate at 20ºC/hr to below 40ºC before cooling it down to room temperature. The exposure and PEB will darken the exposed areas of the resist noticeably making it readily visible for further alignment.

Typically the stack is not developed at this point in the process and a 2nd, normally thicker SUEX layer is next laminated onto the substrate (Step 4, Fig. 1) and patterned, in the case of HAR fluidic microstructures using X-ray lithography (Step 5, Fig. 1). When using SUEX or PET as the substrate, aligned exposures can be realized with moderate overlay accuracy by looking through the substrate and overlaying the dark latent image of the 1st layer pattern with the X-ray mask pattern. X-ray exposure is performed with a typical bottom dose of 180 J/cm^3 for the lowest (thickest) level and a top/bottom dose ratio not exceeding 5. For x-ray exposures it is necessary that the PET cover is left in place to prevent skin formation [1,8]. Despite multiple height levels x-ray photons will precisely pattern all thicknesses in one step. There is no problem observed so far with having a higher bottom dose at layers of less than maximum height. Subsequent PEB forms a latent image of the 2nd layer (Step 6, Fig. 1) which is now developed. Development is typically conducted face down with minimal agitation using standard propyleneglycol monoethyl ether acetate (PGMEA). Development times will vary with the thickness of the SUEX and via size. Smaller vias will require longer time to get proper movement of dissolved resist from the structures. It seems important to overdevelop and use much longer than usual development times, particularly when having HARM structures. For 100µm thick structures the development time can range from 20min for large open structures to over an hour for very fine vias. For 500µm tall structures development can require up to 4hrs depending upon smallest feature sizes and corresponding aspect ratios.

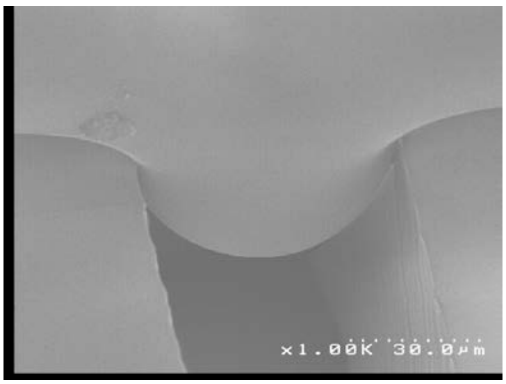
Lamination of the cover sheet, UV patterning of any desired features into the sheet, and curing completes the pattern definition (Steps 7&8, Fig. 1). Lamination of the SUEX coversheet over the extensive patterning of the structured layer requires adjustment of the temperature and pressure to the design, balancing proper bonding with adequate adhesion and minimum sagging across large gaps [11]. We have found that a lamination temperature within a few degrees of 50ºC generally gives satisfactory results. However, if there are residues or dirt particles on the surface a slightly higher temperature will eventually ensure full encapsulation and proper bonding of the structures at the price of some sagging (Figs. 2).



Lamination at 47˚C



Lamination at 55˚C



Lamination at 62˚C

Figure 2:

SUEX cover laminated across a 50µm wide channel of a test chip using the setup shown in Fig. 3. The temperature is the set temperature of the laminator with the effective temperature at the SUEX cover about 12˚C lower. With increasing temperature the material starts flowing into the channel ensuring good coverage but also reducing the effective height. The lowest temperature is not sufficient for proper sealing.

It is also important to precisely define the gap between the cover sheet and roller. As shown in Fig. 3 for the example of a GC chip released from a PET temporary substrate after process Step 6, Fig. 1, a frame, typically PC, PE or PMMA, was machined into which the chip was placed with a height about 100µm less than the combined thickness of chip and cover sheet including PET cover.

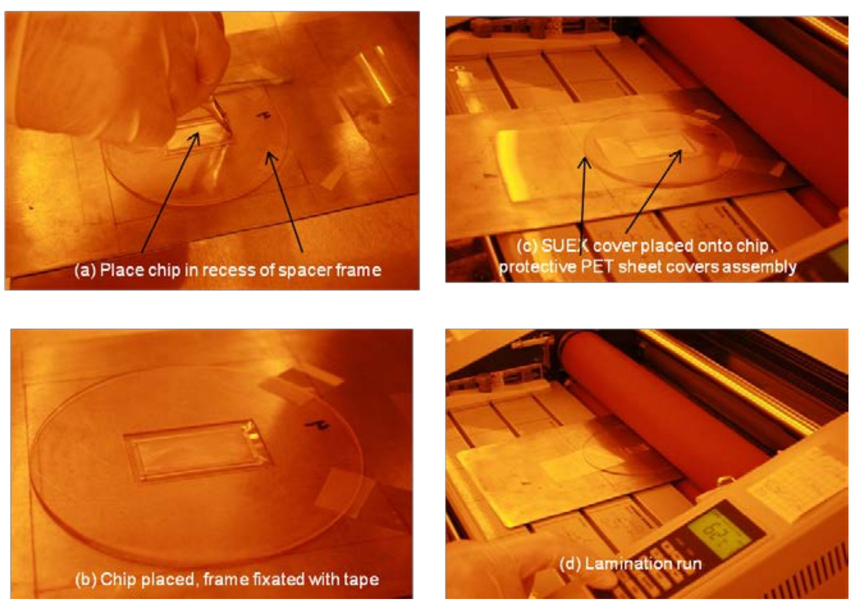


Fig. 3: Sealing of an individual GC chip with a SUEX cover sheet.

The frame ensures easy handling and no slipping of the GC chip while its height guarantees repeatable gap definition and temperature as the only critical parameter to control.

For final step the patterned cover sheet is developed providing for example inlet and outlet holes. If desired, a final optional hardbake step at temperatures ranging from 130-160°C for 1hr (plus 2hrs of ramp down time) completes the fabrication. The hard-baked chip is slightly darker brown than the regular PEB'd chip but still quite transparent. At hard-bake temperatures above 130ºC the SUEX material will become completely non-reactive with the highest Young's modulus achievable for the given dose and PEB temperature. The material will also become stiffer and less flexible.

Examples

A first example of a microfluidic chip consisting of three SUEX layers is shown in Fig. 4. In this example the chip used a PET sheet as temporary cover from which the individual chips were peeled off prior to the cover sheet lamination. The bottom layer was patterned with inlet and outlet holes while the cover sheet was flood exposed. Tubes were connected by gluing and colored water was pumped through the chip proving that there is no leakage. Fabrication of this chip took about 2 days with most of the time needed for the various bake steps (total exposure and lamination time was less than 2hrs). This is significantly faster than the ~3 week processing time needed in [12] where fabrication of a similar structure using liquid SU8 is described and much simpler than processes described in [13,14].

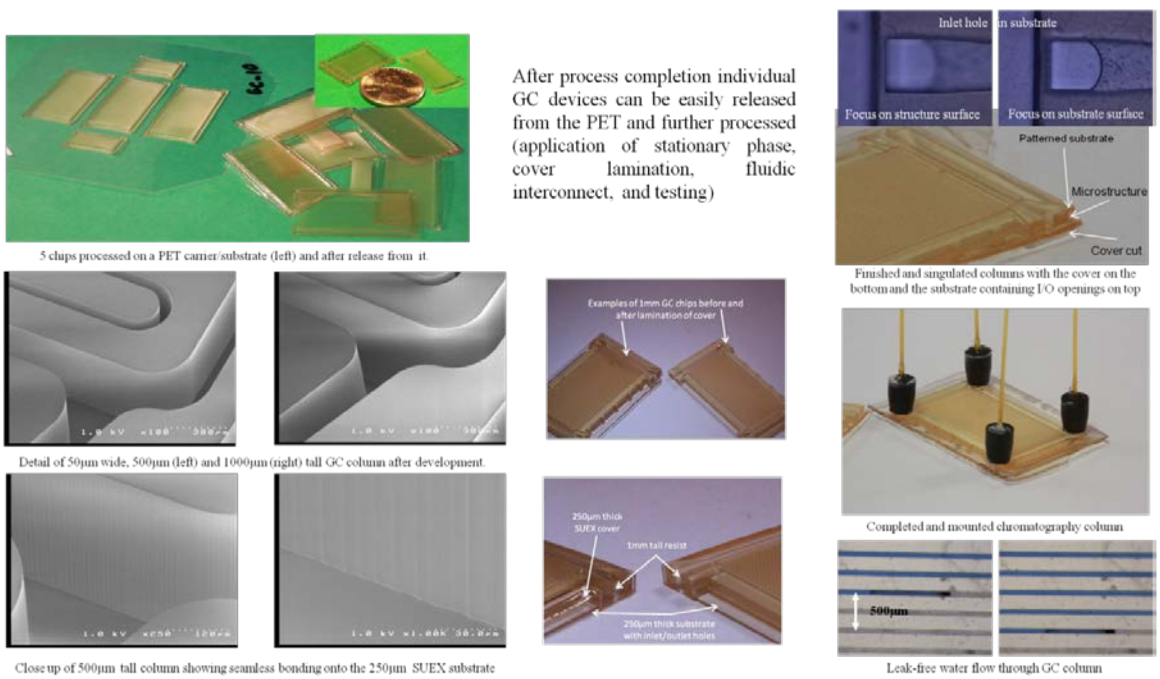


Fig. 4: Micro_GC chips made in SUEX with columns 50µm wide and up to 1mm tall. The chip is made from 3 SUEX layers and inlet-outlet holes are patterned for convenient fluidic interconnent.

A major advantage of processed SUEX structures that are not hard-baked is a fairly high flexibility as demonstrated in Figs. 5. In this case a single SUEX sheet, 250µm thick, was patterned with ~10µm wide holes in small strips of about 5mm width and 40mm length using x-rays. It should be emphasized that bending of the individual strips is fully reversible and no cracks or other signs of fatigue have been observed. It is also possible to deform patterned SUEX sheets and apply a hard-bake process in the deformed state freezing it permanently. This way patterned SUEX sheets can conform to non-planar surfaces as needed by the application.

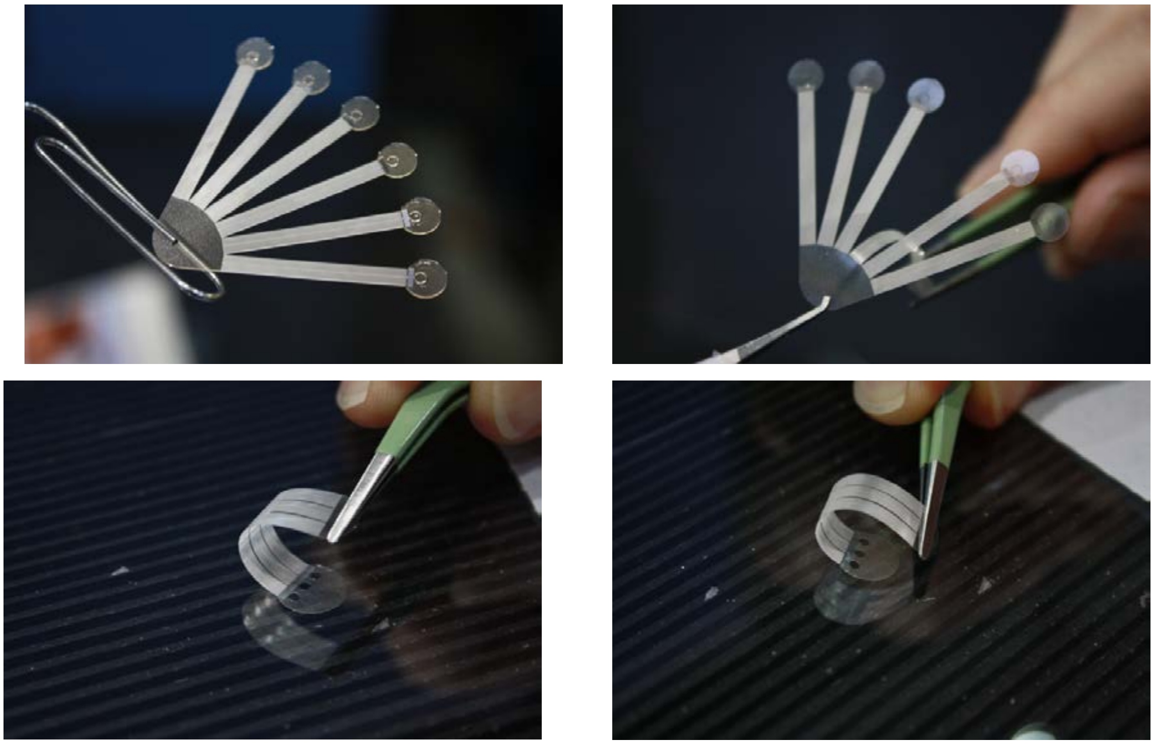


Fig. 5: Strips of fine flow filters, 250µ thick, with pores of ~10µm diameter. Reversible bending without breaking or crack formation of the stripes is possible.

An interesting application for Biosensors is the combination of SUEX fluidic structures with printed circuit board technology [15,16,17]. In our initial feasibility tests a wet-chemically etched copper sheet was conformably filled with SUEX using a vacuum hot embossing process and patterned with x-rays (Figs. 6-8) with masks of opposite tones forming holes and posts (hole mask is the same used for structures shown in Fig. 5). The hot embossing was done with the HEX 2 machine located at CAMD using a newly developed recipe. Close ups in Figs. 6 demonstrate several things: when using higher temperatures/forces the SUEX tends to overfill the structures (left); however there is hardly any lateral flow. When using thin sheets insufficient for complete filling of the copper pattern only smaller structures are fully filled while larger areas remain unfilled with a smooth transition between the areas (right). Further systematic studies of the flow behavior of SUEX into structured substrates are needed to fully understand the behavior and optimize the process.

SUEX filled copper substrates have been exposed with x-rays, post-baked, and developed using standard parameters and structures are shown in Figs. 7 and 8. The height is ranging from ~30 to 200µm. Exposures with a positive tone mask result in post arrays and free-standing structures conformably patterned on the copper substrate. Adhesion of smallest posts of ~10µm is overall promising although debonding is observed motivating additional surface preparation steps or optimization of process parameters for this application. The copper also acts as 'intermediate' mask preventing x-rays from exposing underlying SUEX which can easily be developed.

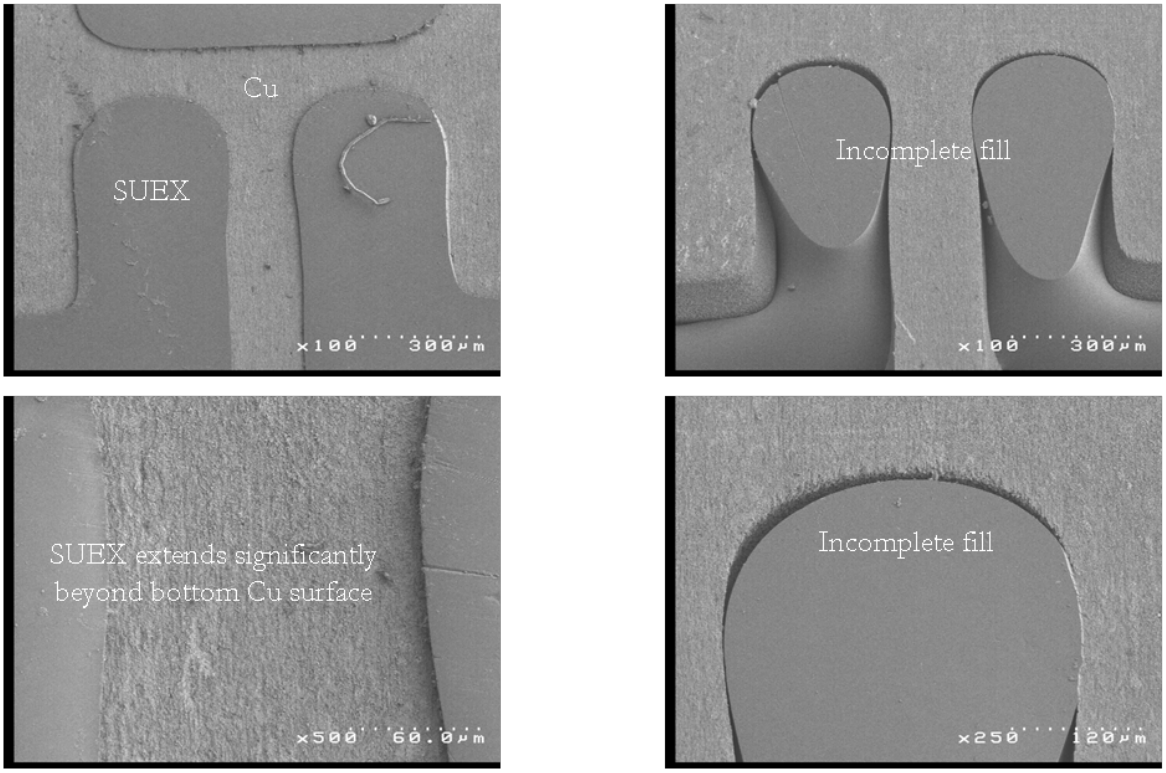


Fig. 6: Examples of SUEX filled copper substrates prepared by vacuum hot embossing.

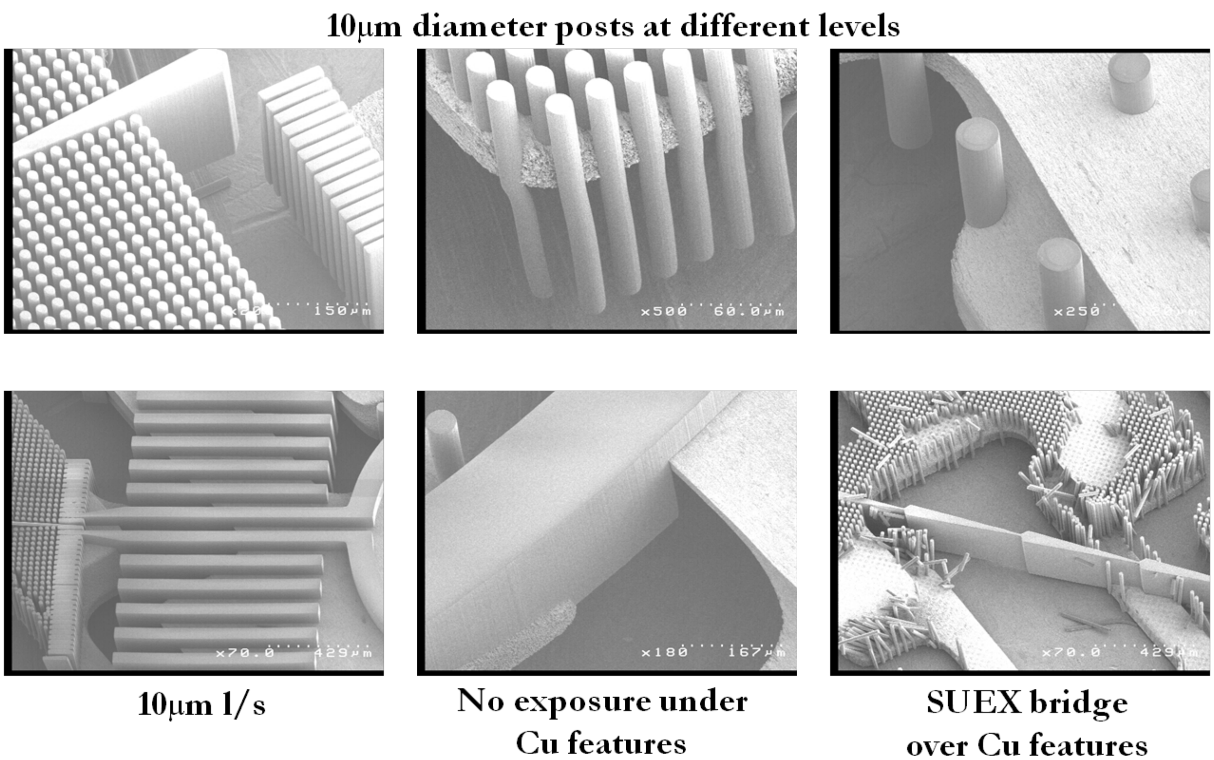


Fig. 7: SUEX post arrays patterned on a copper etched substrate using x-ray ltihography.

The negative tone mask defines tiny hole-arrays identical to the structures shown in Fig. 5. These fairly large, connected pattern also adhere well to the copper and show no signs of stress (cracks, debonding) suggesting that the process parameters are acceptable even for materials with different CTEs like copper and SUEX. Overall the results are very encouraging for building SUEX fluidic structures on printed circuit boards in an effort to make Bio- and environmental MEMS sensors and diagnostic chips.

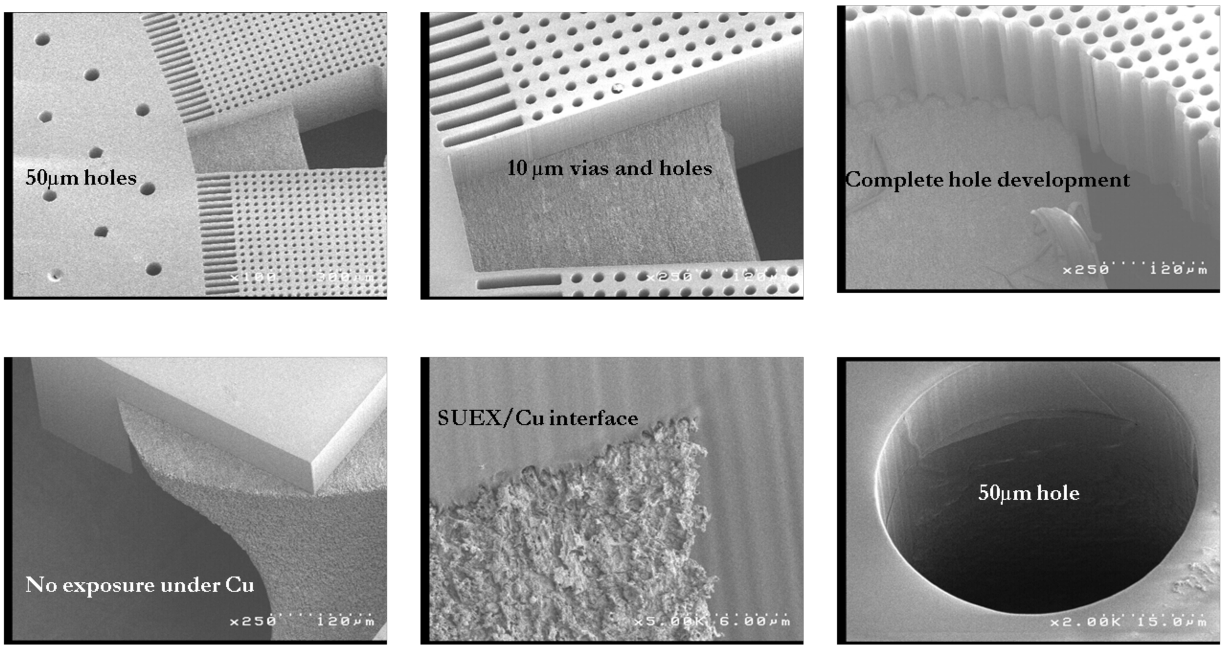


Fig. 8: SUEX hole arrays patterned on a copper etched substrate using x-ray ltihography.

Another microfluidics application is using the SUEX sheet as a patternable cover sheet on fluidic chips made, for example, by hot embossing or injection molding in PMMA. In Fig. 9 an example of a PMMA micromixer is shown which has been hot embossed and then covered with a 100µm thick SUEX sheet. The SUEX cover is patterned to match the inlet/outlet pattern from a micro-plumber connector making microfluidic interconnects and experiments a simple ‘plug&play’ process.

Conclusion

In conclusion the process outlined above to make 3-layer microfluidic MEMS devices is very attractive for rapid prototyping offering fast processing time, lithographic precision and process control along with smallest features below 1 micrometer. SUEX can be combined with a number of polymer and metal substrates and offers new opportunities for making microfluidic based MEMS devices. Future efforts will focus on combining SUEX fluidic chips with printed circuit board technology in an effort to make sensor chips for biomedical and environmental applications. Furthermore, the variable combination of processes (UV and X-ray lithography, molding) allows convenient patterning of complex, multi-level structures advantageous for microfluidic applications, for example micro-reactor and Micro Total Analysis Systems.

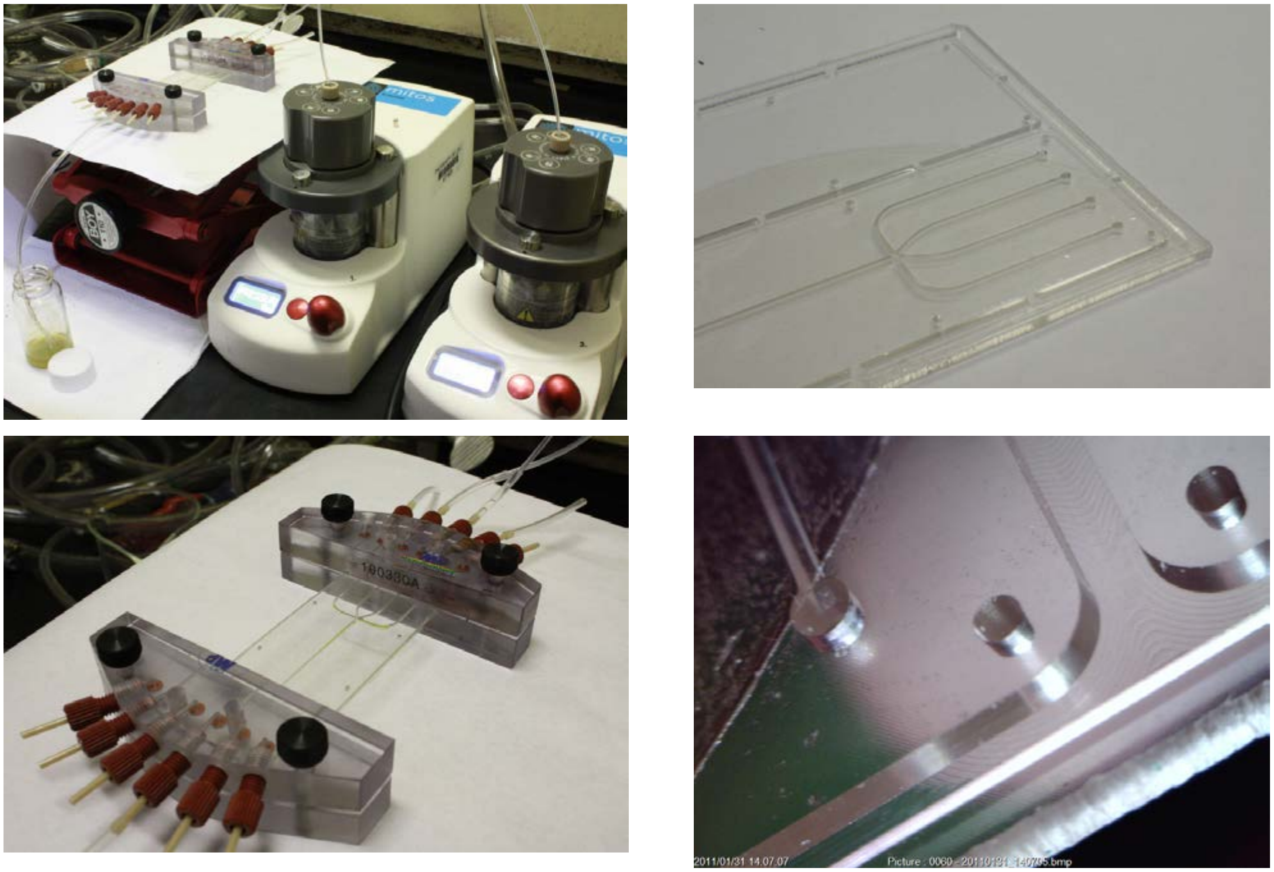


Fig. 9: Microreactor chip, molded in PMMA and covered with a 100µm SUEX sheet, passively mounted into a fluidic manifold (MicroPlumbers, LLC; http://microplumbers.com/) and operated with external micropumps (Dolomite Ltd., http://www.dolomite- microfluidics.com/). Courtesy: C. Kumar et al, LSU-ERFC.

References

[1] D. Johnson et al.; *SUEX Process Optimization for Ultra Thick High Aspect Ratio LIGA Imaging*; Proc. SPIE, Vol. 7872(2011), 79722U.

[2] D. W. Johnson, G. Ahrens, A. Vogt; *KM661 Thick Epoxy Sheets for MEMS and Packaging Applications*; Commercialization of Micro and Nano Systems 2009, Copenhagen, Sept 2, 2009.

[3] S. Mammitzsch et al.; *Free-standing HAR SU-8 Columns for Micro-fluidic Application*"; Proc. HARMST 2007, Besancon, June 2007:109-110.

[4] D.W. Johnson and J. Goettert; *Potential of SUEX Negative Laminate Resist for X-Ray Lithography and LIGA MEMS Applications;* COMS 2010, Albuquerque, NM, Aug 29-Sept 3, 2010.

[5] V. Singh et al.; *Fabrication and Characterization of HAR Microfluidic Device to Concentrate Microalgae*; Proc. Microtech 2012, Santa Clara, CA, June 2012, poster presentation # 1589.

[6] V. Reddy, D. Yemane, J. Goettert, Y. Li, K. Nanda Kumar , A. Sanampudi and C.S.S.R. Kumar; *Milli-fluidic reactor systems for gold nano cluster synthesis – Bridge between bulk and micro fluidic systems*; Proc. MRS Fall Meeting 2010 -Symposium TT5.5, Boston (2010).

[7] Z.-C. Peng et al.; *CMOS compatible integration of three dimensional microfluidic systems based on low temperature transfer of SU-8 films*; J MEMS Vol. 15 (2006): 708-716.

[8] J. Goettert et al.; *SUEX–Dry Laminate Resist for Ultra-tall X-ray Lithography Applications*; Proc. HARMST 2011, Hsinchu/Taiwan (2011): 29-30.

[9] D. Johnson et al.; *SUEX-Dry Laminate Resist-Opportunities for MEMS Applications*; Proc. HARMST 2011, Hsinchu/Taiwan (2011), 36-37.

[10] D. Johnson et. al; *Processing SUEX Dry Film for Microfluidic Applications*; Proc. MNE 2011, Berlin, Sept 2011.

[11] N. Wangler et al.; *High-resolution permanent photoresist laminate TMMF for sealed micro-fluidic structures in biological applications*; J. Micromech. Microeng. **21** (2011) 095009 (9pp).

[12] S. Mammitzsch; *Development of a micro gas chromatography column in SU-8*;Master thesis Fachhochschule Gelsenkirchen, Gelsenkirchen, Germany (2005).

[13] Y. Song et al.; *Fabrication of an SU-8 based microfluidic reactor on a PEEK substrate sealed by a 'flexible semi-solid transfer' (FST) process*; J. Micromech. Microeng 14 (2004): 932-940.

[14] F.J. Blanco et al.; *Novel three-dimensional embedded SU-8 microchannels fabricated using a low temperature full wafer adhesive bonding*; J. Micromech. Microeng 14 (2004): 1047-1056.

[15] A. Wego, S. Richter, L.Pagel; *Fluidic microsystems based on printed circuit board technology*; J. Micromech. Microeng. **11** (2001) 528-531.

[16] D. Fries et. al.; *PCBMEMS for Environmental Sensing Systems*; Proc. IECON 2005 (2005): 2352-2356.

[17] L. L. Wu, et. al.; *A printed circuit board based microfluidic system for point-of-care diagnostics applications;* Proc. µTAS 2011, Seattle (2011): 1819-1821.