

# SUEX for High Aspect Ratio Micro-Nanofluidic Applications

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## Abstract

SUEX epoxy Thick Dry Film Sheets (TDFS) are a promising material for micro- and nanofluidic applications [1]. They contain a cationically cured modified epoxy formulation utilizing an antimony-free photo acid generator (PAG). A highly controlled solvent-less process provides uniform resist coatings between two throw-away layers of protective polyester (PET) film in varying thicknesses ranging from 100 to 500 $\mu\text{m}$ . Patterning is possible with both UV and X-ray lithography as well as a combination of the two and enables the fabrication of multi-level, complex designs. This paper will demonstrate the utility of SUEX TDFS for all-polymer fluidic devices for gases and liquids, e.g. a GC separation column or a microfluidic filter by means of optical and x-ray lithography. Details of the fabrication process and discussion of results will illustrate the potential use of this material in micro- and nanofluidics. A fully sealed device can be patterned in about two days with high aspect ratio microstructures (HARMST).

**Keywords:** SUEX, dry film negative resist sheets, all-polymer microfluidics, high aspect ratio microstructures (HARMST)

## 1 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 [1-3]. Of special benefit is the extreme simplicity in the use of the dry film sheets. Standard lamination approach allows one to apply the resist sheets as well as imaging 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 with typical exposure times of 15-20min for a 500 $\mu\text{m}$  thick 4" substrate. This allows the rapid preparation of large reservoirs in the thick SUEX coatings while at the same

time providing thin fluidic channels optionally filled with densely packed post arrays [4] for separation and mixing. In addition, polymer microreactors and other microTAS devices benefit from both excellent material compatibility, even for aggressive chemicals [5], and outstanding structure fidelity as well as very high aspect ratios and structural details of sub-micron resolution. Recent results have been published [6, 7] illustrating examples of structure quality and possible applications.

## 2 EXPERIMENTAL

The microfluidic device is built layer by layer with a final design transferred into the combined but not yet developed resist sandwich. Figure 1 details a standard process flow for such microfluidic applications. First the SUEX substrate is UV flood exposed to form a cross-linkable base with no pattern. The substrate layer is typically 100 to 250 $\mu\text{m}$  thick and the exposure dose will range from 500 to 1500mJ/cm<sup>2</sup> (Figure 1, (a)). The 250 $\mu\text{m}$  thick sheet is more robust but the 100 $\mu\text{m}$  may be desired to reduce the overall form factor. Alternatively the substrate can be patterned with a UV mask where the bottom PET is left on. The PET acts as a temporary carrier allowing one to pick off or peel individual parts after completion of the process. It is also feasible to image in dicing lines when using a PET substrate. The top protective SUEX PET film can be removed either before or after UV exposure.

Next without heating the substrate a first 100-250 $\mu\text{m}$  thick SUEX layer is laminated (roll laminator temperature set to 70°C) onto the substrate followed by UV- lithography of a base pattern (Figure 1, (b,c)). In this case some kind of alignment marks need to be provided on the design in order to align subsequent layers to this base layer. 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 85°C for 1hr as our standard PEB condition starting at 50 °C and ramping up to 85 °C in 30min. For best flatness we have found it advantageous to slowly cool the PEB'd substrate at 20°C/hr to below 40°C before cooling to room temperature. To this point the process has taken approximately 3-4 hrs.

The exposure and PEB will darken the resist noticeably making it readily visible for further alignment. Typically the stack is not developed at this point in the process. However, if multiple layers or multiple levels are desired it may be necessary to develop now. Multiple layers can then

be individually applied and processed to provide regions with different functions. These multiple layers can also have multiple thicknesses for different needs.

Lamination of a 2<sup>nd</sup>, normally thicker SUEX layer which becomes the actual HARM structural layer is next applied (Figure 1, (d)). If the stack at this point has not been developed this is a simple lamination process. If the stack has been developed care must be taken to ensure that the areas of the resist to be exposed are planar and do not contain voids or air bubbles. If the area to be exposed is exclusively over the base pattern then a simple lamination process may be adequate. However if the areas to be exposed are over multiple thicknesses, the lamination may entrap air pockets or voids at the bottom corners of the features and may leave an uneven top resist surface. To overcome this will require vacuum lamination, where the lamination is conducted under a moderate vacuum utilizing special vacuum lamination equipment.

This structural layer is then exposed with a microfluidic pattern typically using UV lithography. In the case of HARM structures this layer will be patterned using X-ray lithography [8]. When using SUEX or PET as the substrate, aligned exposures can be easily realized with moderate overlay accuracy by looking through the substrate and overlaying 1<sup>st</sup> layer pattern which are dark after the PEB with the mask pattern. UV exposure of the combined stack will depend on the thickness of the resist layer to be exposed and can range from about 500mJ/cm<sup>2</sup> for a 100µm thick film to 1500mJ/cm<sup>2</sup> for a 250µm thick film up to about 2500mJ/cm<sup>2</sup> for a 500µm SUEX thickness. Films thicker than about 350µm will typically require x-ray exposure. X-ray exposure is performed with a typical bottom dose of 180 J/cm<sup>3</sup> for the lowest (thickest) level and a top/bottom dose ratio not exceeding 5 (Figure 1, (e)). For x-ray exposures it is necessary that there is a PET foil left in place covering the top of layer 2 which will prevent skin formation [9]. For a substrate with multiple height levels x-ray photons can be used and the HARM layer can be precisely patterned over all of the various thicknesses in one step. There is no problem observed so far with having a higher bottom dose at layers of less than maximum height. Note that most exposures will generally be over only previously exposed areas in the substrate or 1<sup>st</sup> layer as any open (unexposed) regions will be exposed at this point.

During PEB a latent image is formed which is then developed (Figure 1, (f)). Development is typically conducted face down with minimal agitation using standard propyleneglycol methoxyethyl ether (PGMEA). Development times will vary with the thickness of the SUEX and the 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 dedevelopment 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 a 500µm structure develop times range from 1hr to over 2.5hr.

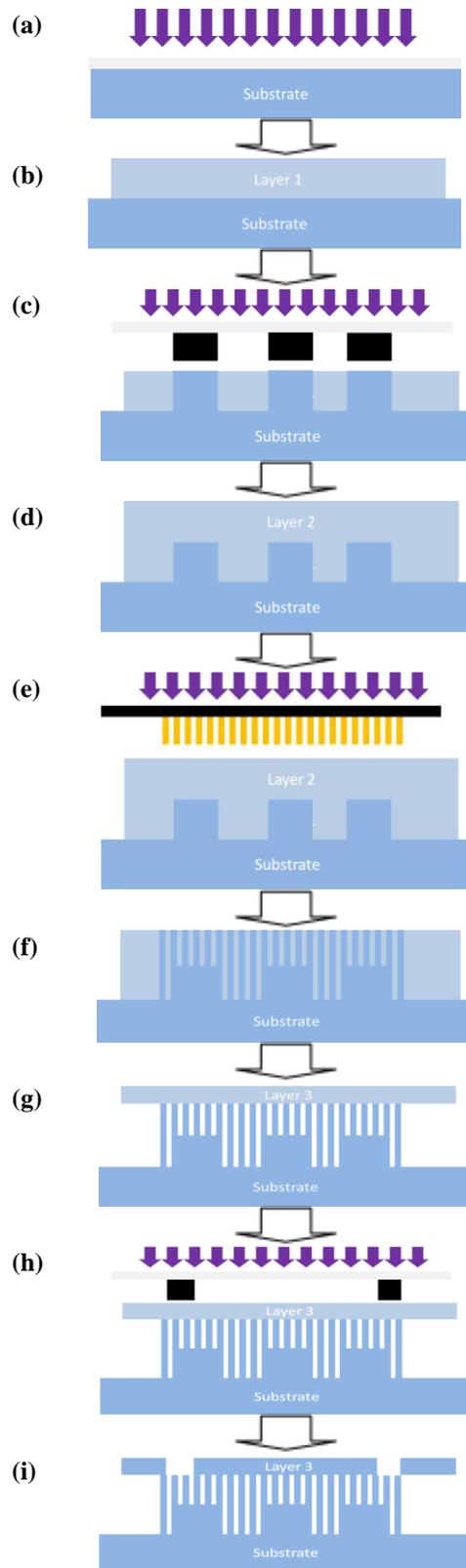


Figure 1: Process flow fabricating a 3-level all-polymer microfluidic chip with two patterned layers.

It is also highly advantageous to use a two bath system where the bulk of the development is done in a used bath until the bulk of the resist is removed and the final developing done in a clean bath. The final resist development can take as much a twice the time as the bulk removal with HARM structures. After development the structures are washed with developer and then IPA. If a white precipitate is noted the part is returned to the 2<sup>nd</sup> developer bath. If no precipitate is noted the part is rinsed in an IPA bath for 15-60min. Optionally the part can next be placed in an IPA/water bath for the same time, removed and dried. Vacuum drying of HARM structures is strongly recommended.

Lamination of the cover sheet, UV patterning of any desired features, and curing completes the process (Figure 1, (g-i)). 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 [10]. We have found that a lamination temperature within a few degrees of 50°C generally gives satisfactory results. A second pass through the laminator can also be helpful to get good bonding at all of the surfaces. Also we prefer to avoid O<sub>2</sub> plasma for cleaning purposes as we have noticed lamination problems after plasma treatments.

Either flood or patterned UV exposure of layer 3 is needed to cure the coversheet. In this step inlet/outlet structures or other optional features are lithographically defined by not exposing the areas to be left open. However, there are underlying structural differences with some areas bonding to the 2<sup>nd</sup> layer SUEX, some areas over open regions and others around the tops of features where they have been embedded in the coversheet. All of these lead to differences in necessary dose, reflections and diffraction patterns. These can generate very unexpected, unwanted patterns in the cover. We have found that it is preferable to minimize the dose to minimize the optical effects but not so much that you cannot cure the cover over openings.

At our standard 85°C PEB conditions the cover will flow down into the fluidic channels faster than it will cure; thus an initial gentle PEB at 50°C was found to be required. This temperature is low enough that it generally prevents material flow into the structured layer 2 yet high enough to slowly crosslink the SUEX cover. We typically PEB at 50°C overnight. We also find that having the coversheet side down on top of a piece of PET is advantageous to minimize flow. Development after a patterned exposure is challenging as dissolved material may be carried into fluidic structures and affect later performance. We have found it necessary to run the development within a vacuum in order to remove the air and allow the developer to completely wet these surfaces. Cycling vacuum conditions will also generate sufficient flow and clean closed areas from any residues. A final IPA rinse, eventually vacuum supported, and vacuum drying for 1hr completes the process. A final PEB at normal conditions (85°C) or an optional hardbake at

160°C for 1hr (plus 2hrs of ramp down time) completes the fabrication. The hardbaked chip is slightly dark but still quite transparent with improved material properties. The total processing time is approx. 2 days mainly determined by the PEB and hardbake step.

### 3 RESULTS

We have demonstrated the utility of this fabrication process by producing two microfluidic devices, an all polymer gas chromatography column and a microfluidic separation column. Both structures are complete examples of fluidic chips during the fabrication process showing high aspect ratio features, multi-level patterning, cover sheet application and issues, and packaged device. Figure 2 shows a picture of three layer GC columns fabricated on a SUEX substrate which were fabricated in under 2 days. Figure 3 shows SEM pictures of the 50µm wide column structure and demonstrating straight vertical sidewalls. Close up of interface between the SUEX substrate and the SUEX resist demonstrates proper bonding and clean development down to the substrate surface.

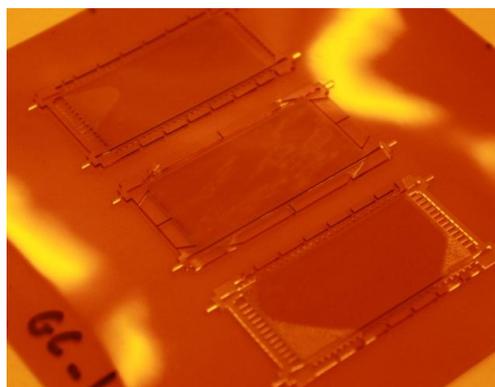


Figure 2. All polymer GC columns on a common SUEX substrate.

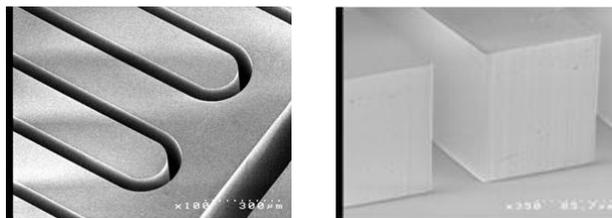


Figure 3. SEM overview and cross-section of GC column showing straight vertical sidewalls in ~500µm thick SUEX prior to cover sheet lamination.

The final device features of the separation filter along with pattern detail and other structures are shown in Figures 4 and 5. Test structures of the densely packed post arrays with 25µm wide posts and a 10µm minimum distance between them which are used as flow filters within microfluidic channels are shown in Figure 4. The structures were patterned by x-ray lithography at the LSU CAMD synchrotron facility in Baton Rouge, Louisiana [11].

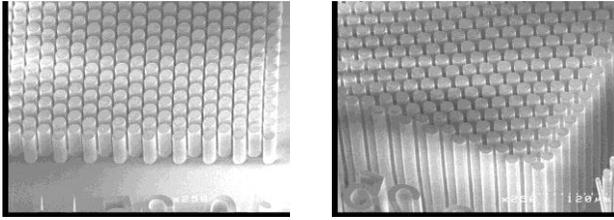


Figure 4. 25 $\mu$ m posts in flow filter region of separation filter with a height of 100 $\mu$ m (left) and 350 $\mu$ m (right).

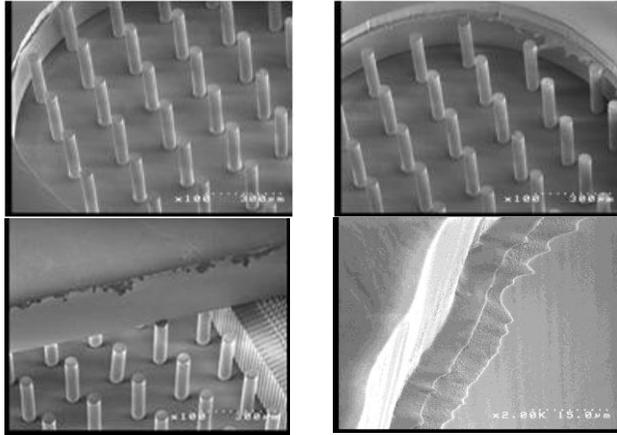


Figure 5. Separation filter after application and structuring of coversheet showing 50 $\mu$ m posts in inlet/outlet regions, structure height is 250 $\mu$ m with smallest features of 10 $\mu$ m.

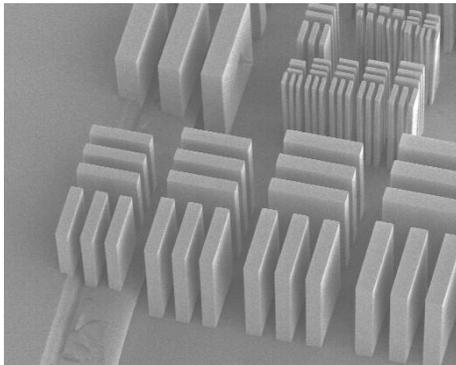


Figure 6. Example of a 3- level test structure with heights varying from 150-350 $\mu$ m; smallest structures are about 10 $\mu$ m wide and 350 $\mu$ m tall.

Figure 5 shows the cover sheet above two different sides of the outlet area and close ups of the cover and underlying 50 $\mu$ m posts (more details are discussed in [4]). The last SEM demonstrates some of the imaging issues over structured features which we have encountered. The SEM shows minimal finger structures which will have no major impact on the performance of the device. The ‘poor optical appearance’ is primarily caused by thin surface layers which should not block the flow but may influence the wetting behavior.

In Figure 6 we show features patterned over a base level with different heights. All structures are patterned by x-ray

lithography in one step taking advantage of the high resist contrast and wide dose range. The two different height levels have been defined by SUEX layers cut to size, laminated into the substrate, and flood exposed and baked.

## 4 CONCLUSIONS

SUEX TDFS have been successfully used to make three layer MEMS devices with HARM features. Fully sealed, high aspect ratio, multi-level microfluidic chips on a free standing substrate or as individual free standing parts can be processed in approx. 2 days and will offer many opportunities for microfluidic MEMS and NEMS applications including rapid prototyping.

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