Optimization of Protocol for Fabrication of a Polymer Filter from a PDMS Mold

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Abstract:

Microfabricated structures have been demonstrated to filter specific types of cells out of blood samples when incorporated into a parylene filter. However, the process for creating these parylene filters can be time consuming, and requires expensive equipment. Also, the fabrication process utilizes harsh temperatures and specific techniques that are incompatible with softer polymers such as polydimethylsiloxane (PDMS), biodegradable polymers, or other implantable biomaterials [1]. The focus of this project was to optimize a protocol for an alternative micromolding fabrication process. We found the optimal combination of surfactant-based release agents to consistently create high quality molds with a high aspect ratio and features as small as 1 μm. We also successfully fabricated a PDMS filter using this micromolding process.

Introduction:

This project tested different methods of creating a polymer filter from a PDMS mold. The original filter, consisting of parylene, was used to create the polymer mold. This mold then served to fabricate more filters consisting of different polymers.

First, the mold material was optimized. Several types of PDMS as well as polyurethane were employed. The optimal protocol was found to be PDMS at a 5:1 base to crosslinking agent ratio, using a release agent of either a 0.1% SDS solution or 1% Triton X-100 solution with 95% ethanol as the solvent. Once the mold process was optimized, the next step was to then optimize a process of using the molds to stamp out filters out of PDMS which have been spun onto a silicon wafer. This project demonstrated in one instance that this process is feasible, but further optimization is necessary in order to make it more consistent and repeatable.

Experimental Procedure:

The first step was to determine which polymer would be most suitable for the mold. Polyurethane and a 1:1 ratio of 10:1 PDMS and Blue PDMS were tested against the standard, 5:1 PDMS. To fabricate the mold, the parylene filter was dipped in isopropyl alcohol (IPA) and laid flat on the bottom of a polystyrene container. This was then placed in a vacuum hood 15-20 minutes. Next, a pipette was used to place two drops of release agent onto the filter, making sure to completely cover the filter. The container with the filter was then placed back inside the vacuum hood for 30 minutes. Then, the mold polymer (either polyurethane, a 1:1 ratio of 10:1 PDMS and Blue PDMS, or 5:1 PDMS) was then poured over another container with a parylene filter in it. This was then left to cure overnight.

PDMS 5:1 was proven to be the best option in terms of feature resolution. Once this was determined, different release agents were tested at different concentrations to find the optimal combination: a 0.3 g Fairy dish soap solution, Triton X-100 at 0.1% and 1.0% concentrations by volume, and SDS solutions at 0.1% and 1.0% concentrations by mass were tested, utilizing 95% ethanol as the solvent. To test these release agents, The same process described above was used, and the 5:1 clear PDMS was used as the polymer for the mold. To cure, the container was placed on a hot plate at 80°C for one hour, and then cut out of the container with a size 22 mm circular punch tool. Tweezers were used to peel off the filter.

In order to fabricate filters from the molds, the general procedure that was followed was to take the molds that were fabricated in stage 1, and first apply release agent to them. Using a pipette, 50 μl of either Triton X-100 (1.0%) solution or SDS (0.1%) solution were applied to each mold. These molds were then placed in a vacuum hood for 30 minutes to dry the release agent. 10:1 PDMS was prepared, and 10 ml of the PDMS was used to spin a 10 μm layer of PDMS onto a silicon wafer (Single Polished 325 μm N-type 10-20). The recipe for spinning was 10 seconds at 500 rpm, and 5 minutes at 4000 rpm. The wafer was then removed from the spinner, the molds were placed on the PDMS-coated wafer and placed in a vacuum oven to be vacuum baked at 80°C overnight. The molds were then removed from the wafer using tweezers. The wafer then contained the filters stamped out on its surface.

Results and Conclusions:

Out of the three polymers tested, the 5:1 PDMS produced the best quality result, as shown in Figure 1. The 0.1% SDS solution and the 1% Triton X-100 solution proved to be the best release agents in terms of feature quality and amount of residue left, as seen in Table 2. In the second stage of the experiment, the only successful filter fabricated was in the first trial, with a single polished 325 μm N-type 10-20 wafer, a mold that was made using 1.0% Triton-X, and then treated with 0.1% SDS. This one filter was the only successful filter — even when the same conditions were repeated — and the reasons why are currently unknown. It is believed that there is an affinity issue between the PDMS and the release agent-treated mold. It could also be a cleanliness issue; there may have been small contaminations. HMDS treatment did clearly seem to be detrimental, and the application of pressure didn't help either. The affinity issue needs to be resolved in order to optimize this process. However, since it did work once, the process has been proven to be successful under the right conditions.

References:

[1] S. Y. Zheng, H. K. Lin, B. Lu, A. Williams, R. Datar, R. J. Cote and Y. C. Tai, 3D microfilter device for viable CTC enrichment from blood, Biomed. Microdevices, 2011, 13, 203–213.

Polymer	Release Agent	Results
5:1 Clear PDMS	Fairy 0.3g	Worked well, small and large features visible. Clean, except for a little residue. The filter came off of the mold easily
Polyurethane	Fairy 0.3g	Some features visible. Not the small ones. Not very clean, poor quality, many bubbles, although not on area of filter mold.
1:1 Blue and Clear PDMS	Fairy 0.3g	Not as clean or uniform as the 5:1 clear PDMS

Figure 1: Results of mold material optimization.

Release Agent	Mold Polymer	Results
Fairy 0.3g	5:1 PDMS	Worked well, small and large features visible. Still showing a little residue, consistently throughout. The filter came off of the mold easily
0.1% TritonX (70% Et)	5:1 PDMS	Filter completely stuck to PDMS, small part did come off, and mold underneath was not clean, bad resolution, features not complete
0.1% TritonX (95% Et)	5:1 PDMS	Filter was slightly difficult to remove, but it did come off, in pieces. Features, however, came out very clean with good resolution. Small and large features visible.
1.0% TritonX (95% Et)	5:1 PDMS	Filter came off easily, slight residue but not much. Features very clean, almost perfect.
0.1% SDS (95% Et)	5:1 PDMS	Filter came off easily. Some spots of residue, but features were almost perfect, large and small.
1.0% SDS (95% Et)	5:1 PDMS	Filter came off easily, but there was substantial residue. Features came out well but very high amount of residue left from the release agent.

Figure 2: Results of mold release agent optimization.

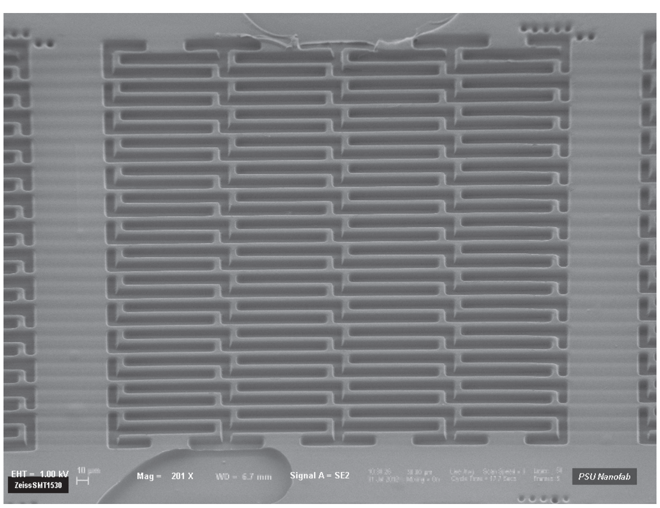

Figure 3: PDMS filter fabricated using molding process.

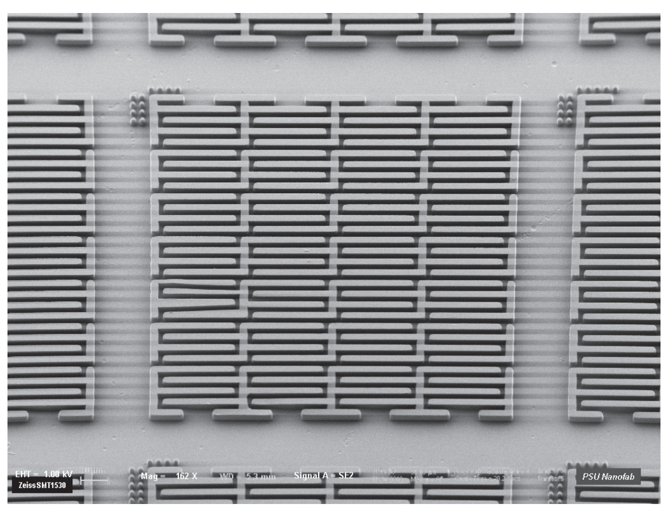

Figure 4: PDMS molds fabricated using molding process.