# SURFACE TREATMENTS OF SOFT MOLDS FOR HIGH ASPECT RATIO MOLDING OF POLY-PEGDA

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## ABSTRACT

This paper looks at surface treatments for Polydimethylsiloxane (PDMS) soft molds for cured Poly(ethylene) glycol diacrylate (Poly-PEGDA) to increase reliability of mold filling and decreasing mold permeability. Contact angle of uncured PEGDA on the different mold treatments (deep UV exposure, oxygen plasma and Parylene-C coating) was measured. Out of these, Parylene-C showed to have a good compromise between improved mold filling and durability. Our method allows for the creation of high aspect ratio structures (in excess of 10:1) as well as enclosed microfluidic channels.

## KEYWORDS

Poly-PEGDA, PDMS, Parylene C, High Aspect Ratio.

## INTRODUCTION

Interest in using polymers to fabricate microfluidic devices has increased in the past decade for their ease of fabrication, favorable mechanical and optical properties, low cost and biocompatibility, for applications such as chemical and biological assays [1][2].

Cured Poly(ethylene) glycol diacrylate (Poly-PEGDA) is a promising material as an alternative to the popular PDMS, and has been shown to have good optical properties, good water stability, and lower nonspecific adsorption than PDMS[3]. Another advantage of this material is its greater stiffness that contributes to micro channels with a better dynamic response due to a reduced hydraulic capacitance. Poly-PEGDA has also been shown to be biocompatible provided adequate fabrication procedures are followed [4].

Poly-PEGDA has not been widely explored as a structural material for microfluidic devices. Simple Poly-PEGDA straight microfluidic channels were demonstrated using rigid molds by Rogers et al.[3]. Previous work has also been done by Yue's group, demonstrating both high aspect ratio structured surfaces and soft mold surface treatment to improve mold durability[4][5]. They find the curing process leaves uncured residue, which can cause mold degradation, so they propose a hydrophobic surface treatment to minimize its effect; however, they did not provide measurements of the wettability of the PEGDA resin on the mold after surface treatment. We explored surface treatments that seek to increase the wetting properties of the mold to PEGDA and thus improve mold filling, while at the same time decreasing the permeability of the PDMS mold to improve durability. We also provide contact angle measurements of PEGDA on our treated molds.

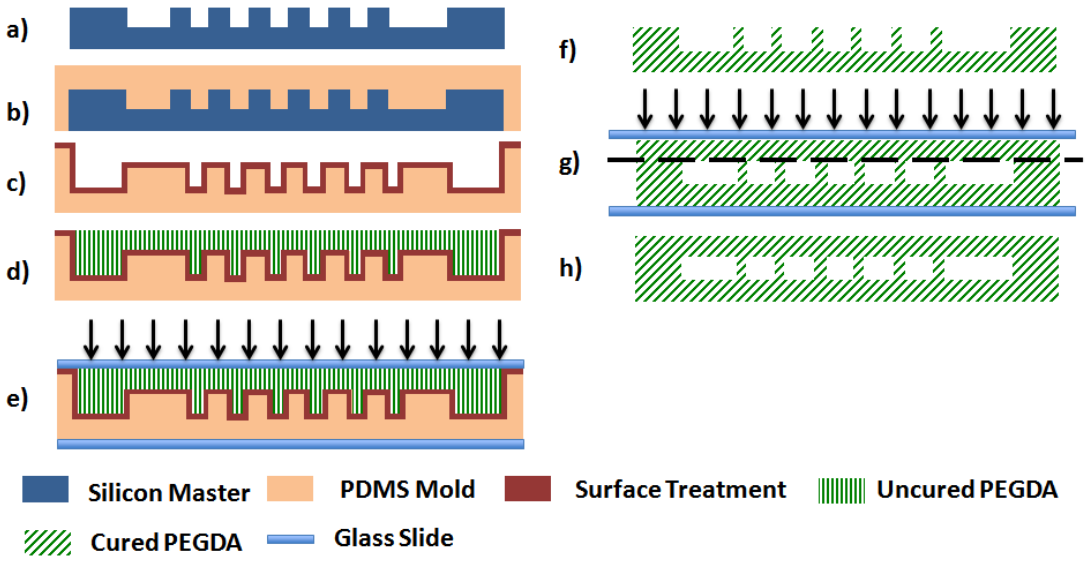


*Figure 1: Fabrication process. a) A Si master is etched using DRIE. b) PDMS soft mold is then cast over master. c) Surface treatment. d) Liquid uncured PEGDA is poured into mold. e) Compression between two glass slides and UV exposure. f) Partially cured PEGDA is then peeled off. g) A second layer of partially cured PEGDA (a-f) is brought into contact and compressed between two glass slides followed by a UV exposure step. h) Final microfluidic device.*

## FABRICATION

We follow the optimized formulation for Poly-PEGDA proposed by Rogers [3], comprised of 99.9% 258 PEGDA with 0.1% 2,2-dimethoxy-2-phenylacetophenone (DMPA) as photoinitiator. This formulation has good water stability, optical quality, and some mechanical flexibility.

As purchased, the liquid polymer is mixed with 100 ppm of Monomethyl Ether of Hydroquinone (MEHQ) as a polymerization inhibitor for storage, which was removed by using commercially available inhibitor removing columns (Sigma Aldrich product number 306312).

The fabrication process of our samples is summarized in Figure 1. First, a master was fabricated on a silicon wafer using Deep Reactive Ion Etching (DRIE, $C_4F_8$ and $SF_6$ cycles @ 0.48µm/cycle up to 50 µm deep). A PDMS soft mold was formed out of the silicon master (with 10:1 ratio of base to curing agent) and then surface treated. Next, the mold was first filled with liquid PEGDA, carefully compressed between two glass slides to avoid trapping of air bubbles, and then exposed using a UV light box (7 mW/cm$^2$). To produce fully cured devices, a 30 minute exposure was sufficient. If bonding was required, samples were underexposed (5 minutes) leaving them purposely undercured. The samples were then removed from the mold and rinsed with isopropyl alcohol. For bonding, two undercured films were brought into contact, compressed by two glass slides and further cured.

The molds were subjected to three different surface treatments: oxygen plasma (70 W, 5 min), Parylene C coating (200 nm thick) and deep UV exposure (254 nm wavelength for 40 minutes). The contact angle of a 25 µl drop of liquid PEGDA on each of the surfaces was measured using a goniometer (KRÜSS DSA100), Figure 2. A decrease in the contact angle means an increase in wettability, which results in an easier filling of the mold cavity.

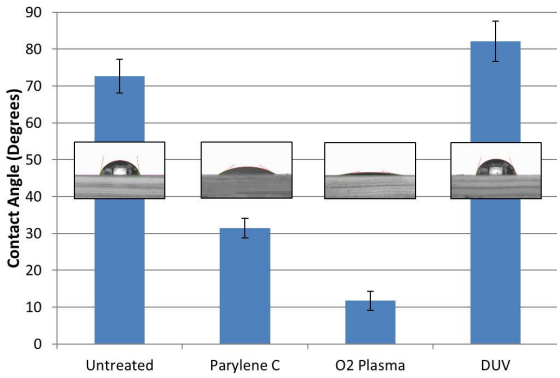


*Figure 2: Contact angle of 25 µl drop of un-cured PEGDA (mean with error bars showing standard deviation) on PDMS with different surface treatments, with inset goniometer photographs.*

## RESULTS AND DISCUSSION

Arrays of different sized circular columns and holes were successfully fabricated in Poly-PEGDA to test the quality of pattern transfer and ability to create high aspect ratio structures, with diameters ranging between 100 µm to 5 µm. Several patterns with different sized features were fabricated, small and high aspect ratio features were successfully reproduced, with excellent master pattern transfer, as shown in Figure 3a.

During the fabrication of the silicon master, the cylinders produced were not perfectly straight but instead had tapered sidewalls and ridges due to the DRIE Bosch process. Therefore our structures are of smaller diameter at the base than at the top end, which contributes to a higher aspect ratio (in excess of 10:1). These structures as well as the DRIE ridges are faithfully reproduced by the Poly-PEGDA replicate, which demonstrates its sub-micron precision.

Enclosed microfluidic channels were made using the fabrication method shown in Figure 1. A patterned bottom layer and a top unpatterned layer were cast separately, leaving them undercured (5 minutes), brought together in close contact, clamped and fully cured. Ports were attached to the finished device and connected to a syringe pump. An example of one of such devices is shown in Figure 3b.

One of the advantages of using soft molds when working with cured PEGDA can be noticed during the demolding step. It allows for a much easier removal of the replicate, which results in an increased yield, as the chances of damaging either the mold or the replicate are reduced. The flexibility of the mold also allows the creation and release of structures with negative sidewalls, as seen in Figure 3a. Another advantage is the fact that the original silicon master can be used to generate several molds, both allowing greater parallelization and protection of the master, which in our case was produced by more complex lithographic steps.

The molds needed to be carefully handled when filling with the liquid PEGDA. At the step when the mold is compressed between two glass slides, it is important to make sure that no large air bubbles are trapped underneath the glass to ensure good results.

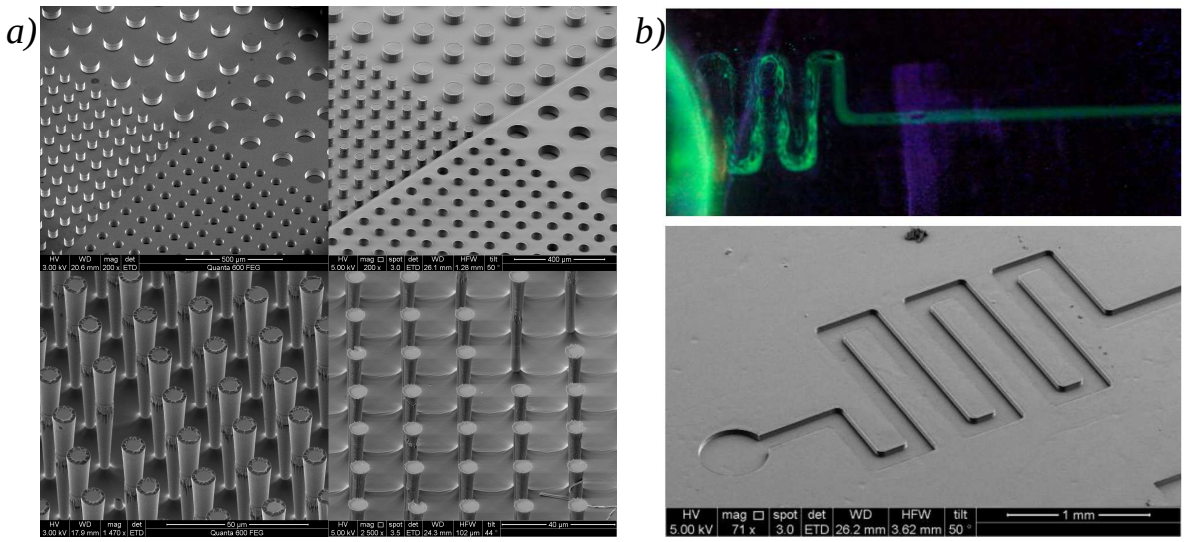


*Figure 3: a) SEM photographs showing comparison between Silicon master and Cross-linked PEGDA. Top Row: Low aspect ratio structures, 50 and 100 µm diameter, 50 µm height. Original silicon master (left), cured PEGDA replicate (right). Bottom Row: High aspect ratio structures, 8.5 µm diameter at tip, 3.5 µm diameter at base, 50 µm height. Original silicon master (left), cured PEGDA replicate (right). b) Enclosed microfluidic device, showing tracer fluorescent flow (top) and SEM photograph (bottom).*

Having small air bubbles trapped between the mold features is also a common problem during mold filling, which affects the pattern transfer negatively. Without surface treatment, a degassing step is necessary prior to the exposure to ensure complete filling, however a treatment that increases the wettability on PEGDA the mold facilitates filling and reduces the need for a degassing step.

## CONCLUSIONS

As shown by our cylindrical patterns, casting of Poly-PEGDA on soft PDMS molds can produce an excellent pattern transfer, faithfully reproducing sub-micron features, and is capable of high aspect ratio structures (in excess of 10:1).

The Parylene C coating proved to produce enough increased wettability to improve mold filling, while also providing a more robust and longer-term solution than oxygen plasma, with excellent step coverage. No degradation of the molds was observed on the large structures (100 - 20 µm diameter pillars, microfluidic structures) after several uses. However, because of the obvious fragility of the high aspect ratio pillars (5 - 10 µm diameter), degradation on these structures was apparent after a few uses.

This work showed the first process for creating both high aspect ratio structures and enclosed channels in Poly-PEGDA. The use of soft molds allowed for easy demolding, and a Parylene C coating was shown to improve the wettability of PEGDA on the mold and improve mold filling. Poly-PEGDA's low nonspecific adsorption and favorable optical qualities make it an interesting material to be used for microfluidic devices, and we have presented a fabrication process that achieves this.

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