Patterning of thick polymeric substrates for the fabrication of microfluidic devices

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Abstract. Plasma etching is investigated as an alternative technology to soft lithography for the fabrication of microfluidic devices. First, soft lithography was employed for the fabrication of microchannels in poly-dimethylsiloxane (PDMS), for use in a biosensor for protein and DNA detection. Then, a plasma etching process was investigated and optimized with respect to etch rate maximization for PDMS and PMMA substrates. Preliminary results for deep pattern transfer to these polymeric substrates with an Al etch mask are shown.

1. Introduction

There is an increasing need for fabrication of microfluidic devices, which currently attract the interest of both the scientific community and the industry, given the fact that lab-on-a chip systems allow fast and cost-efficient biological and/or chemical analysis [1]. Polymers such as poly-dimethyl siloxane (PDMS) and poly-methyl methacrylate (PMMA) present significant advantages as structural materials of microfluidic devices, including reduced cost, easy fabrication, and good physico-chemical properties, compared to Si or glass. For PDMS patterning, soft lithography,[2-4] realized by means of rapid prototyping and replica molding, is a widely spread fabrication process, since it is inexpensive and not demanding special equipment. Similarly to PDMS, PMMA is a material widely used as a plastic substrate where microfluidic channels are fabricated (usually by hot embossing) [5] as well as a lithographic material. However, patterning of PDMS and PMMA substrates based on alternative processes such as plasma etching [5-6] has been scarcely studied or used for fabrication of microfluidic devices.

In this paper, we aim at the exploration of high-density plasma etching processes for fabrication of microfluidic devices on plastic substrates as an alternative to soft-lithography or as a more advantageous on-chip production process.

2. Experimental

For the fabrication of the mold needed for soft lithography, the negative epoxy type photoresist SU-8 100 from MicroChem Corp. was used spin-coated on Si substrates with a Karl Suss RC8 spinner. The lithography was performed with a Karl – Suss MJ3 mask aligner (350-500 nm). Since for the lithography of SU-8 exposure to i-line (365nm) is recommended, a narrow band-pass filter with a FWHM of 55nm was initially used. However, the obtained lithographic structures of SU(8) suffered from T-topping. To avoid that, a home-made filter was used consisting of a 50µm film of SU-8 spin-coated on a glass slide, in order to cut off wavelengths lower than 365 nm, due to the high absorbance of SU(8) in this spectral region. The transmission spectra of the initial narrow-band pass filter, of the
‘homemade’ filter, and of their combination with a final FWHM of 20 nm are shown in ‘figure 1’ and ‘figure 2’.

The PDMS cast on photolithographically structured SU(8) molds was Sylgard 184, supplied from Dow Corning Corp. The base and the curing agent of the silicone elastomer were mixed in a volume ratio of 10:1 and placed in a desiccator for 45 min so as to remove bubbles created during mixing. The same type of PDMS, spin-coated on Si, was used in the form of blanket and patterned samples for investigating the plasma etching behavior of PDMS.

![Figure 1](image1.png)  
**Figure 1.** UV transmission spectra of the initial narrow band pass filter and the 50 nm spin-coated layer of SU-8 on glass.

![Figure 2](image2.png)  
**Figure 2.** The transmission spectrum resulting from the combination of the filters of ‘figure 1’ indicates a FWHM of 20 nm.

The dry etching of PDMS and PMMA was performed in a high density Micromachining Etch Tool (MET) from Alcatel and specifically an Inductively Coupled rf (13.56 MHz) Plasma (ICP) reactor, in which the plasma source and bias electrode source are independent, so that plasma density and ion energy can be independently adjusted. The etching gas was SF₆ for PDMS and O₂ for PMMA. The dependence of the etching rate on process parameters such as gas pressure, rf plasma power and bias voltage applied on the substrate was studied. In-situ laser interferometry at 650 nm and spectroscopic ellipsometry were used in order to measure the etching rates of PDMS and PMMA. Al deposited on PDMS and PMMA in a thermal evaporator was patterned by lithography and wet etching in order to be tested as an etch mask for these substrates.

Scanning Electron Microscopy on a LEO 440 instrument was used in order to examine the fabricated SU(8), PDMS and PMMA structures, and their profiles.

3. Results and discussion

i. Soft-lithography: First, the master was fabricated from SU(8) for use in replica molding of PDMS. After exposure of a 100 µm layer of SU-8 (spin-coated on a Si wafer) to UV light for 4.5 min, through a properly-designed photolithography mask and the combination of filters (see ‘figure 2’), and development, a 100µm-thick structure was patterned, shown in ‘figure 3’. This structure constitutes the mold for the final structure to be fabricated from PDMS. For this purpose, the Si wafer with the patterned SU(8) was placed at the bottom of a metallic cylindrical mold of 3-inch diameter and 1.5 mm height. The sidewalls of the metal mold were covered with a Teflon layer to facilitate peeling of PDMS from the metal mold. Then, PDMS pre-polymer was cast against the master covered with a transparency film slowly lowered on the PDMS surface, so as to obtain a smooth top surface and a uniform layer. After curing at 100°C for 1h, in order for the PDMS to be thermally cross-linked, the 1mm thick PDMS layer was successfully peeled off from the master. The final PDMS replica is shown in ‘figure 4’, where 100µm deep and 150 µm wide micro-channels are shown connecting the 800 µm circular inlet and outlet tubing plugs, used for fluid delivery to the microfluidic device. For the latter, the micro-channels will be sealed by pressure application on part of a biosensor, where bio-fluid delivery will be used for spotting of proteins on the biosensor [7].
ii. Patterning by plasma etching: Since PDMS is a Si-containing polymer, etching requires a fluorine-based chemistry [8]. This was experimentally verified by etch rate measurements in mixtures of SF$_6$/O$_2$, where the etching rate of PDMS was found to decrease with the concentration of O$_2$. Therefore, etching rates of PDMS in pure SF$_6$ plasma were measured and optimized for various process conditions. In ‘figure 5’, the increase of etching rate with plasma power is indicated, as expected at high plasma densities leading to higher densities of etching agents. The etching rate of PDMS increases as the pressure decreases, as it is shown in ‘figure 6’. At high pressures, due to collisions, F atom concentration is reduced and ions lose directionality, resulting to lower etching rates.

Similarly to PDMS, an etching process was developed and optimized for PMMA. The etching of PMMA requires O$_2$ plasma chemistry. Etching rates were measured in pure O$_2$, under various process conditions, as shown in ‘figure 7’ and ‘figure 8’.

Having obtained relatively high etch rates, the next step was the fabrication of channels. At the conditions where etching rates for PDMS and PMMA were maximized, pattern transfer was tested for both substrates through an Al mask. The latter was fabricated by thermal deposition of Al, followed by photolithography with a standard novolac resist (AZ5214) and wet etching of Al. Preliminary results are shown in ‘figure 9’ and ‘figure 10’.
Figure 7. Variation of etching rate of PMMA with plasma power. Plasma conditions: O\textsubscript{2} gas, 1.33 Pa pressure, 100 V bias voltage.

Figure 8. Variation of the etching rate of PMMA with pressure. Plasma conditions: O\textsubscript{2} gas, 1800 W plasma power, 100 V bias voltage.

Figure 9. SEM image of the etched PDMS microstructure through an Al mask (depth of structure 2 \(\mu\)m, after a 3 min etch)

Figure 10. SEM of a cross section of a PMMA channel 170\(\mu\)m wide etched successfully at a depth of 50\(\mu\)m, through an Al mask.

4. Conclusions
Fabrication of microfluidic channels was investigated and performed with soft lithography as a reference technology and with dry etching in SF\(_6\) and O\textsubscript{2} plasmas for PDMS and PMMA substrates, respectively. Further investigation is under progress for optimization of the etch rates and the pattern transfer process. Plasma etching shows a promise as a competitive technology for on-chip production of microfluidic devices.

5. References
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