Effect of toluene treatment on PDMS molding into nanoholes

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Polydimethylsiloxane (PDMS) is the most popular and versatile material for soft lithography due to its flexibility and easy fabricating by molding process. However, for nanoscale patterns, it is challenging to fill uncured PDMS into the holes or trenches on the master mold that is coated with a silane anti-adhesion layer needed for demolding. Previous studies have shown that PDMS filling can be facilitated by diluting it with toluene or hexane, which was attributed to the great reduction of viscosity for diluted PDMS [1-2]. However, if viscosity is the limiting factor, the hole filling depth should be increased with the filling time, which is not the case according to our experiment. As a matter of fact, the hole filling time can be roughly estimated as $t=\eta z^2/\gamma R\cos\theta_c$ (η is viscosity, 3.9Pa·s for Sylgard 184 PDMS; z is filling depth, ~1000 nm; γ is surface tension and θ_c contact angle, $\gamma\cos\theta_c$~0.01 N/m; R is hole radius, ~100 nm), which gives 3.9 msec that is far less than typical molding time. Here we argue that it is the wetting properties (depends on interface energy between PDMS and mold) that determine whether PDMS can be filled into the holes; and the reason behind the improved filling for diluted PDMS is because the diluent toluene increases the surface energy of the silane-treated mold.

To validate our assumption, we casted undiluted PDMS onto silane-treated master mold with or without further toluene treatment, and compare their filling property. In the experiment, the master mold in silicon was fabricated by electron beam lithography and reactive ion etching first into an Al layer using the resist as mask, then into the silicon substrate using Al as mask. The pattern contains periodic hole arrays with the diameter from 1000 nm down to 50 nm and the depth close to 800 nm. Anti-adhesion treatment was carried out on the mold surface by using trichloro (1H,1H,2H,2H-perfluorooctyl)silane in a vacuum chamber at room temperature for overnight. Next, one master mold was dipped into toluene for 1 minute. Finally, molds with and without toluene treatment were casted with PDMS (undiluted, Sylgard 184, Dow Corning) and kept in vacuum for 3 hours for degassing, and subsequently cured at 110^{o}C for 3 hours on a hot plate.

As seen in Figure 1, PDMS pillars with diameter down to 273 nm can be duplicated from the master mold treated with toluene, as compared to 640 nm diameter for mold without toluene treatment. Therefore we showed here that toluene treatment greatly improves PDMS' wetting to the master mold. Water contact angle measurement of the master mold showed a slightly reduced contact angle upon toluene treatment.

[1] N. Koo, U. Plachetka, M. Otto, J. Bolten, J. Jeong, E. Lee and H. Kurz, Nanotechnology 19, 225304 (2008).
[2] N. Koo, M. Bender, U. Plachetka, A. Fuchs, T. Wahlbrink, J. Bolten and H. Kurz, Microelectronic Engineering, 84, 904 (2007).

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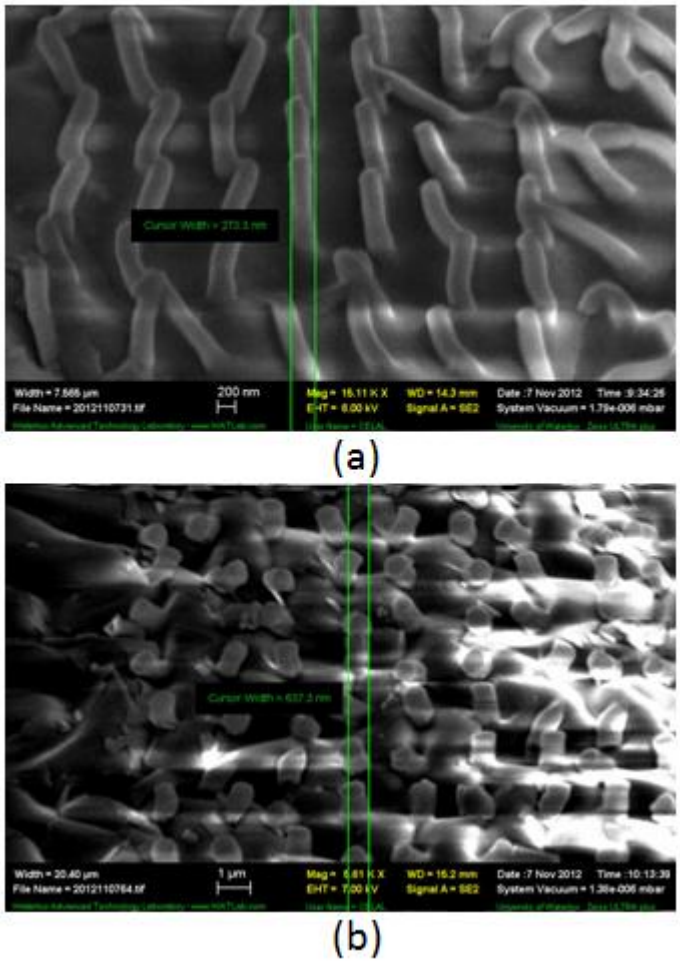

Figure 1 SEM image of PDMS pillar array molded from an anti-adhesion treated silicon master mold with (a) and without (b) toluene treatment. The pillar diameters are 273 nm and 640 nm, respectively. Pillar deformation and significant charging during SEM imaging are evident in both images.