Improved anti-adhesion FOTS coating for imprint mold

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In nanoimprint lithography (NIL), one major challenge is the peeling off of the imprinted polymer resist onto the mold structures. The obvious solution is to coat the mold with a low surface energy self assembled monolayer (SAM). Among all the SAM coatings, perfluorooctyltrichlorosilane (FOTS) is undoubtedly the most popular one that works best with silicon or silicon dioxide mold terminated with hydroxyl group. Both liquid phase (dissolve FOTS in a solvent such as heptane) and vapor phase surface treatment process have been developed, with the latter being more controllable and cost-efficient as it needs only tiny (μL) amount of silane[1]. However, it has been reported that both methods do not provide complete surface coverage. In fact, even using a CVD system with precise control of H_2O and FOTS vapor pressure/flow, the SAM coverage is only 70-90% of its maximum packing density[2]. The coverage would be a lot worse for the commonly used simplified process that involves putting a drop of FOTS next to the mold sealed inside a vacuum container.

Here we show that the FOTS coverage and thus the mold surface energy can be improved by repeated cycle of vapor-phase deposition and subsequent solvent removal of the physisorbed FOTS. Our approach originated from the observation that FOTS-treated mold showed an apparent surface energy increase after being washed with toluene, which we attributed to the removal of physisorbed FOTS by toluene. This surface energy increase is also responsible for the improved filling into mold structures of PDMS when diluted with toluene[3].

In the experiment, we first cleaned bare silicon wafer using solvent and oxygen plasma, and then treated the wafer with FOTS in a vacuum container for 3 hours at room temperature. The film was subsequently baked on a hotplate at 150^{o}C for 20 min. Water contact angle measurement was carried out after this first silane treatment. Next, the FOTS-coated mold was soaked in toluene for 2 min to remove the non-chemisorbed FOTS, followed by a second time of FOTS treatment again in a vacuum container for 3 hours. The same process was repeated one more time.

Figure 1 shows water contact angles after each FOTS surface treatment. Clearly the water contact angle increased and thus the surface energy decreased with the number of treatment. This is attributed to the removal of non-chemisorbed FOTS by toluene, which exposed the mold surface to be chemisorbed by FOTS during the following silane treatment.

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[1] W. R. Ashurst, C. Carraro and R. Maboudian, IEEE Transactions on Device and Materials Reliability, 3, 173 (2003).
[2] T. M. Mayer et al., J. Vac. Sci. Technol. B, 18, 2433 (2000).
[3] C. Con and B. Cui, "Effect of mold treatment by solvent on PDMS molding into nanoholes", Nanoscale Research Letters, 8, 394 (2013).

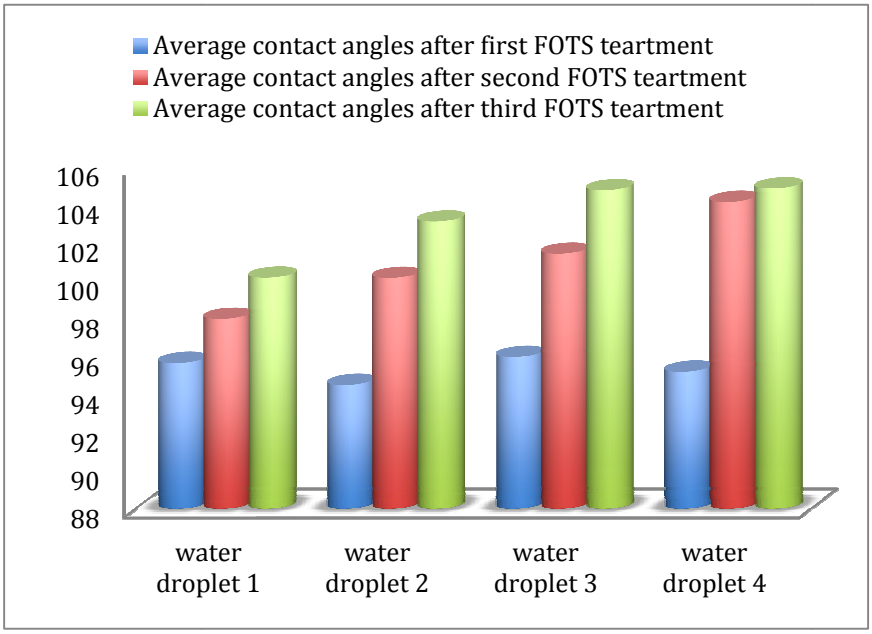

Figure 1 Water contact angles measurement of Si wafer after first, second and third FOTS treatment. The mold was cleaned with toluene before each additional treatment.

Figure 2. Photographic snapshots of water droplets during water contact angle measurements after first (a), second (b), and third (c) FOTS treatment.