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SUPER-HYDROPHOBICITY OF NANO-PATTERNED POLYMER NEEDLE ARRAY

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Abstract — **We report the fabrication and characterization of a novel porous, nano-patterned SU-8 microneedle array as a transparent super-hydrophobic surface. The 33 × 33 SU-8 microneedle array (approximately 250 µm tall) was nano-patterned by plasma treatment and coated with fluorocarbon (FC) film, which significantly increased the contact angle as high as 161^{o} and in turn enhanced hydrophobicity.**

Index Terms – Microneedle, super-hydrophobic, contact angle, nano-pattern, SU-8

I. INTRODUCTION

Liquid repellent surfaces and micromachined super-hydrophobic surfaces have been studied intensively in biomimetics and droplet-based microfluidics for many technological applications including protective cloth coatings and windshield protection since Barthlott's original discovery of nature's lotus leaf self-cleaning surface mechanism [1-4]. Since natural water repelling surfaces including lotus leaves, butterfly wings, sundews, and duck feathers show a high contact angle of approximately 150^{o}, numerous studies have been carried out to create artificial lotus leaves with micro/nano scale structures for super-hydrophobicity. A hierarchical dual-scale structure design has been reported as an effective way to create super-hydrophobicity [5-6] and different types of wetting modes were introduced and analyzed theoretically [7]. Due to relative ease of fabrication, one of the most common materials used for combined micro/nano scale super-hydrophobic surface was silicon [7-9]. A variety of polymers such as ploy-methylmethacrylate (PMMA), polystyrene (PS) [10] and polytetrafluoroethylene (PTFE) [11] have also been investigated for super-hydrophobicity applications. For flexible and transparent super-hydrophobic surfaces, polydimethyl-siloxane (PDMS) based micro pillar structures were studied [12-14].

In this work, we report plasma treated nano-patterned surfaces on polymer microneedle array created by backside exposure of SU-8 [15, 16] as a combined micro/nano scale structures as a super-hydrophobic surface.

II. FABRICATION

A single mask was designed with a 33 × 33 circular geometry array with diameters of 70 µm and a pitch of 150 µm for high density. As shown in Figure 1(a), Pyrex 7740 (thickness 700 µm) was used as a substrate and prior to deposition of SU-8, a thin 17 nm layer of OmniCoat (MicroChem Corp, Newton, MA) was applied to improve adhesion between the Pyrex substrate and SU-8.

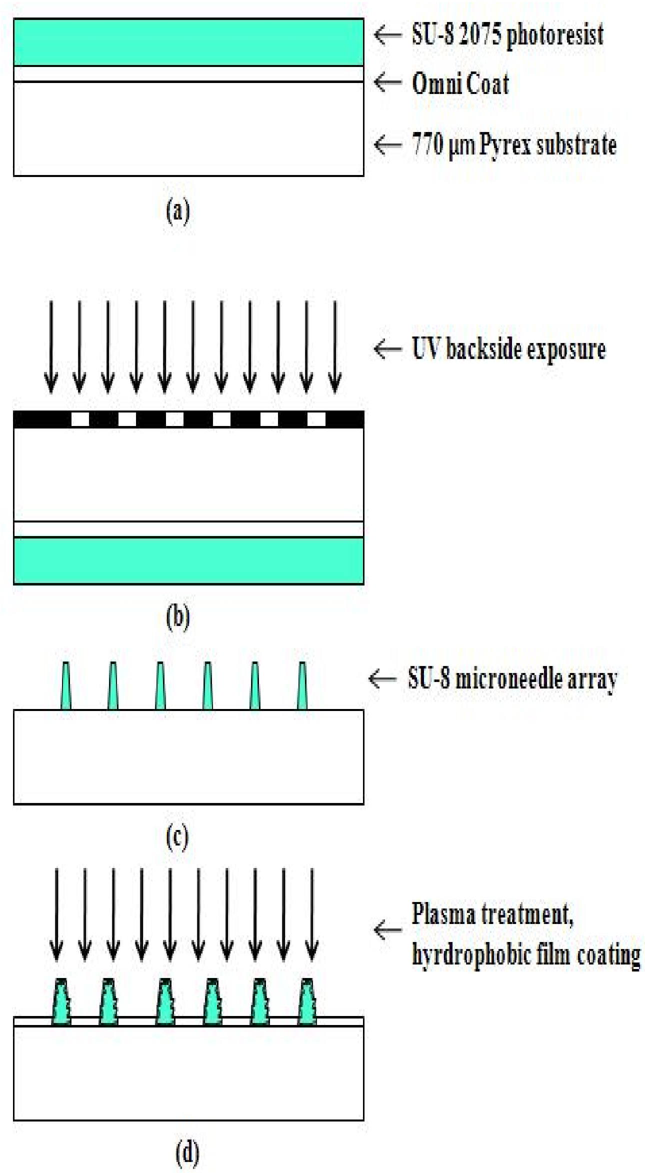
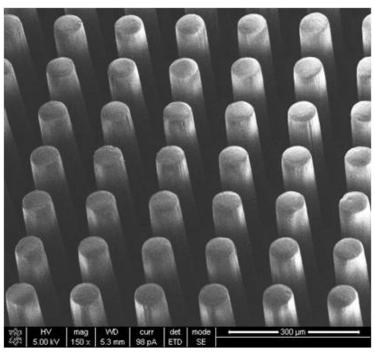
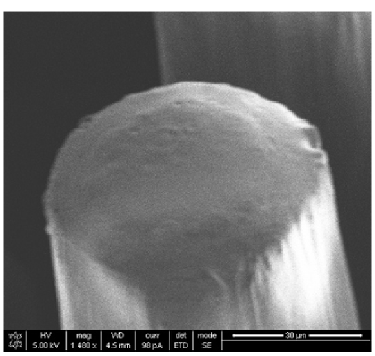


Fig. 1. Process flow for the micro/nano patterned SU-8 microneedles: (a) spin coating of 250 µm SU-8, (b) backside exposure to create an array of tapered SU-8 structures, (c) developing, (d) plasma treatment to create the nanopatterns on the needle array.

A 250 µm thick layer of SU-8 2075 (MicroChem Corp, Newton, MA) was spin-coated at 700 rpm for 1 minute and the sample was left on a flat surface for two hours for stress relaxation and planarization. The SU-8 was then soft baked at 65 ^{o}C for 20 minutes, then gradually ramped to 95 ^{o}C for a continuous bake of 80 minutes on the hot plate where, upon completion, the sample was gradually cooled down to room temperature. After the soft bake, as shown in Figure 1(b), UV (365 nm, i-line) backside exposure [15, 16] was carried out with a dose of 1500 mJ/cm^2 using a single mask. Then, a post-exposure-bake (PEB) was performed at 65 ^{o}C for 10 minutes, then gradually ramped to 95 ^{o}C where it stayed for 45 minutes on a hot plate. After the post bake, as shown in Figure 1(c), the sample was developed in a SU-8 developer, propylene glycol methyl ether acetate (PGMEA) bath, for approximately 60 minutes with a gentle stirring condition, and was cleaned by isopropyl alcohol (IPA) 2~3 times. The sample was then cleaned by oxygen plasma (200 Watt, 100 % O_2) for 60 seconds. Figure 2 shows the SEM images of a 250 µm tall fabricated SU-8 microneedle array.



(a)



(b)

Fig. 2. SEM images of backside exposed SU-8 microneedles: (a) microneedle array, (b) close-up view of tip area.

After the formation of the tall SU-8 microneedle array, the sample was dry etched for 1 minute by O_2 / CF_4 (90 % : 10 %) plasma with a power of 300 W under 1,330 mTorr using a microwave plasma etcher (PVA TePla America, Inc.). As shown in Figure 3, this process made the corner of the microneedle tip area round and at the same time creating nano scale porous sructures on the microneedle surface.

Optionally, 100 nm Parylene C or 100 nm fluorocarbon layer film (C_xF_y) which is commonly used in deep reactive ion etching (DRIE) as a passivation layer was coated on the sample surface. The fluorocarbon film was deposited by C_xF_y / Ar (75 % : 25 %) plasma with a bias power of 25 W and the ICP power of 600 W under 20 mTorr using an ICP plasma etcher.

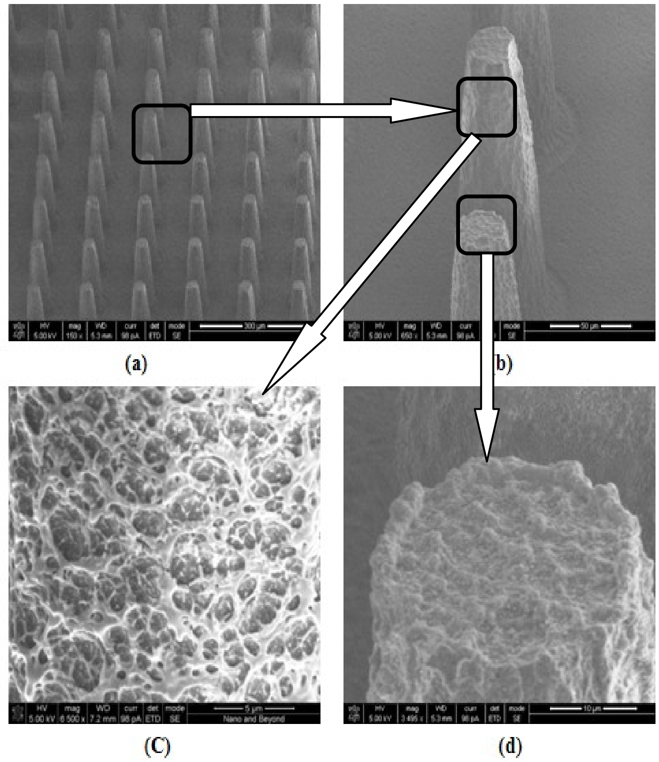


Fig. 3. SEM images of the nano-patterned SU-8 microneedle: (a) after plasma treatment, (b) close-up view, (c) side wall image, (d) close-up image of the tip.

III. CHARACTERIZATION

Before the characterization on the nano patterned microneedle array surface, various flat surfaces were first characterized for their surface morphology. Figure 4 shows the root mean square (RMS) of the surface roughness measured by an AFM (Vecco, 3100 Dimension V).

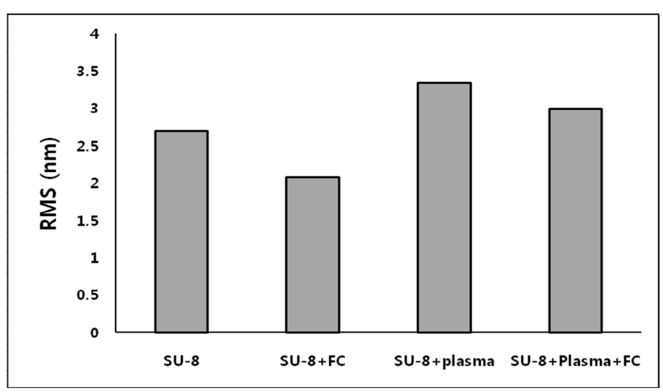


Fig.4. A comparison of the surface roughness on various films treated SU-8 flat surfaces.

Contact angle was measured by a goniometer (Rame-hart model 290). 0.5, 2 and 4 μl deionized (DI) water were used for the contact angle measurement and the measured contact angle was averaged from three measurements. First, we measured contact angles on various planar surfaces (Fig. 5). The bare SU-8 layer showed a contact angle of approximately 79~89°. Once the flat SU-8 layer was plasma (C_xF_y and O_2) treated, although nano-patterned SU-8 surface was formed, SU-8 surface became hydrophilic and the contact angle was lowered (40~45°). Parylene C (PC) coated on a planar SU-8 layer also did not show noticeable improvement in contact angle. In contrast, SU-8 flat surface coated with the fluorocarbon layer showed great improvement in contact angle to approximately 120°.

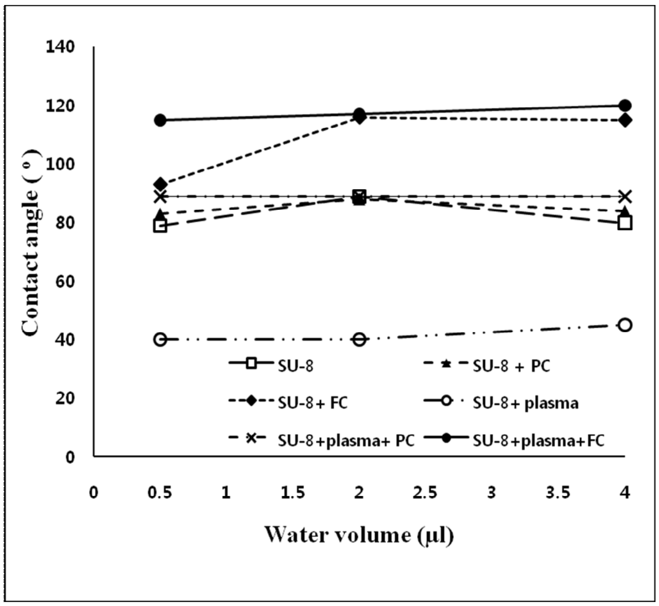


Fig. 5. A comparison of contact angle on various SU-8 planar surfaces.

After the measurement of contact angles on the flat surfaces, we measured contact angles on various films treated SU-8 microneedle array (Fig. 6). The SU-8 microneedle array showed contact angles of 87°, 136° and 118° for 0.5, 2 and 4 μl droplets, respectively. Increased contact angles for 2 and 4 μl droplets are believed to be due purely to the microstructure effect. O_2/CF_4 plasma-treated microneedle arrays again showed low contact angles (~40°) just like plasma treated flat SU-8 surface. The Parylene C layer coated SU-8 needle array showed contact angles similar to the bare SU-8 microneedle array. Again, the fluorocarbon film coated SU-8 microneedle array clearly showed improvement in contact angle (~136°). What is interesting is that the plasma-treated nano-patterned SU-8 needle array both with the fluorocarbon layer and the Parylene C layer showed great improvement on hydrophobicity with contact angles as high as 161° (Fig. 4). The fluorocarbon film coated nano-patterned SU-8 needle array's contact angle was very high regardless of the droplet sizes.

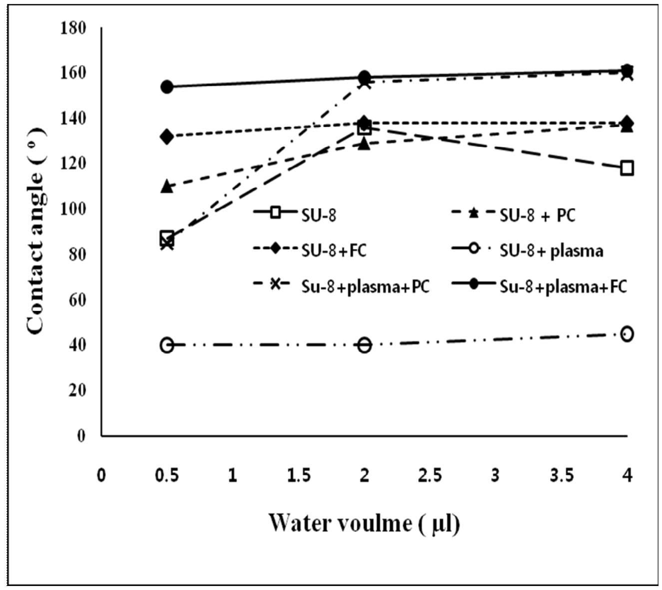
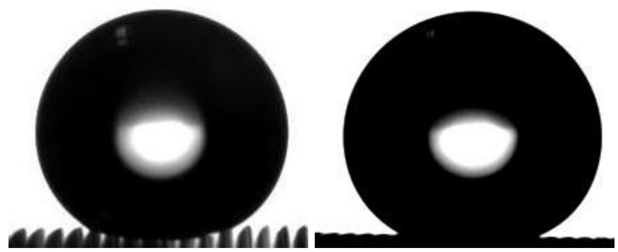


Fig. 6. A comparison of contact angle on various surfaces treated SU-8 microneedle array.



(a) (b)

Fig. 7. 4 μl water drop images on the SU-8 microneedle array: (a) surface coated with Parylene C on the plasma treated SU-8 microneedle array showing contact angle of 160^o, (b) surface with fluorocarbon film coated on the plasma treated SU-8 microneedle array showing contact angle of 161^o.

IV. Conclusion

We have successfully demonstrated super-hydrophobic surfaces using nano-patterned SU-8 microneedle array with surface coated with Parylene C and fluorocarbon. This optically transparent nano-patterned polymer microneedle array's super-hydrophobicity may provide a technology base for water repelling surface applications.

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