Modification of wetting properties of laser-textured surfaces by depositing triboelectrically charged Teflon particles

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Abstract Hydrophilic laser-textured silicon wafers with natural oxide surfaces were rendered hydrophobic by depositing electrostatically charged submicrometer Teflon particles, a process termed as triboelectric Teflon adhesion. Silicon surfaces were micro-textured ($\sim 5\ \mu\text{m}$) by laser ablation using a nanosecond pulsed UV laser. By varying laser fluence, micro-texture morphology of the wafers could be reproduced and well controlled. Wetting properties of the triboelectrically charged Teflon-deposited surfaces were studied by measuring apparent static water contact angles and water contact angle hysteresis as a function of substrate roughness and the amount of Teflon deposited. A similar study was also performed on various micro-textured silicon carbide surfaces (sandpapers). If the average substrate roughness is between 15 and 60 μm , superhydrophobic surfaces can be easily formed by Teflon deposition with water contact angle hysteresis less than 8°. This environmentally benign solvent-free process is a highly efficient, rapid, and inexpensive way to render contact-charged rough surfaces hydrophobic or superhydrophobic.

Keywords Laser ablation · Triboelectric polymer · Teflon · Textured surfaces · Superhydrophobicity

Introduction

Micro- and nanostructuring of surfaces has been extensively used recently in order to control the wetting properties of surfaces with particular emphasis on making them either superhydrophilic or superhydrophobic [1, 2]. Such increased research interest is due to the wide range of applications in which these surfaces can be used, such as biological scaffolds, self-cleaning surfaces, microfluidics, lab-on-chip devices, coatings for automotive and aerospace vehicles, and textiles [3–5]. To generate micro- and/or nanostructures, and thus tailor the wettability of diverse surfaces, several physical and chemical patterning approaches have been employed, including photolithography, electron-beam etching, templated electrochemical deposition, plasma treatment, and selective growth of nanotubes, atomic force microscopy, soft lithography, laser micromachining, and microsphere nano-patterning [6–9]. Electron-beam etching induces high amounts of heat and potential damage to the surface, while photolithography involves multiple steps and requires the use of chemicals. Atomic force microscopy can be used to mechanically etch the surface, but the procedure is slow and is not suitable for large areas. Soft lithography techniques such as micro-contact printing, micro-molding, micro-transfer molding and replica molding, imprinting and injection molding have been extensively used due to their low thermal effect, low cost, and large area applicability [10]. However, these techniques involve multiple steps and require specialized masters. Microsphere nano-patterning, in which a laser light is focused through a layer of self-assembled microparticles, is able to form submicrometer periodic patterns but with a number of disadvantages

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including control of particle assembly and additional patterning over the processed particles.

Laser micromachining, on the other hand, has a number of advantages such as fast material processing speed, large scan area, and single-step capability [11]. Surface patterning using, for example, nano- and femtosecond lasers has many applications such as fabrication of MEMS/NEMS, CMOS, 3D-microstructures, micro-trenches, micro-channels, micro-holes, structured or aligned submicrometer gratings and nanophotonics as well as specialized surfaces for bacterial activity [12, 13]. It can also be used for patterning of magnetic and transparent thin films which can be used for microelectronics applications such as fabrication of flat panel displays and hard disk drives [14]. In this work, by using the concept of triboelectric charging of polymer particles [15–17] confined between two dielectric surfaces we demonstrate a solventless, rapid and simple technique to alter the wetting properties of UV nanosecond laser-ablated silicon wafer surfaces having natural oxide layers. Robust and hydrophobic thin Teflon layers can be formed on these rough surfaces when these submicrometer particles are electrically charged using commercial cellulosic foam. Our aim was to fabricate ceramic surfaces that mimic the surface architecture of self-cleaning lotus leaf surfaces. By varying the laser fluence, it is possible to control the surface roughness of the wafers in a systematic and reproducible way and thus the final wetting properties. The laser ablation technique allowed generation of average surface roughness of the order of 5 μm . In order to investigate the effect of a wider surface roughness range, we have used silicon carbide sandpapers of various grit sizes as model substrates as well. We found that when average surface roughness (in the form of protruding pyramid-like features) of the substrates is between 15 and 60 μm , superhydrophobic surfaces can easily be made with the present technique.

Experimental

Fabrication of micro-textured silicon wafers

Commercial standard silicon wafers (one side polished) having natural oxide surface layers were used in this study. No chemical treatment or mechanical polishing was used to remove the natural oxide layers from the silicon wafers. Both polished and unpolished sides of the wafers were used to create micro-texture using laser ablation for Teflon deposition. A UV laser beam was focused using a cylindrical lens with a focal length of 75 mm. Different laser fluences were used ranging from 0.5 and 2.0 J/cm^2 all under a nanosecond pulse rate. The silicon wafers were kept in liquid baths such as methanol or distilled water, with 5 mm of liquid layer covering them during laser ablation process. The wafers

were moved in precise small steps using an x - y translation stage until a textured surface region of approximately 1 cm^2 area was obtained. Many such rough surfaces with approximately 1 cm^2 area can be fabricated on a single silicon wafer. After ablation, the wafers were removed from the liquid baths and left to dry under ambient conditions. More details about the present laser texturing technique and various other parameters effecting silicon wafer surface structuring can be found in [18].

Triboelectric deposition of Teflon on textured surfaces

Teflon powder was purchased from Sigma-Aldrich. The powder is composed of highly agglomerated submicrometer particles with an average particle size of ~ 150 nm. Certain amount of Teflon powder was spread over a clean cellulose-based foam surface. A commercial Scotch Brite (3M) medium duty cellulosic foam with dimensions 15 $\text{cm} \times 9 \text{ cm} \times 2 \text{ cm}$ was used. A clean metal rod was rubbed over the Teflon-deposited foam surface continuously for a few minutes to spread Teflon evenly over the foam surface. Following this, the foam was continuously rubbed against the textured silicon surfaces in circular motion for a few minutes. At the end of this process, almost all of the Teflon particles spread over the foam surface were transferred to the textured silicon surface forming a thin Teflon layer. It was found that Teflon particles were strongly attached onto the laser-formed structures, since post treatment by immersing in different solvents such as acetone, toluene, methanol, or chloroform did not cause particle removal from the surfaces. A slightly different approach was followed to generate hydrophobic/superhydrophobic surfaces on silicon carbide sandpapers. Submicrometer Teflon powder was directly deposited on the sandpapers and a clean cellulosic foam was rubbed over the surfaces directly for a few minutes to spread the Teflon particles over the rough surface. Typically, sand papers of 600, 800, and 1200 grit sizes were used as substrates. The weight of the samples was recorded before and after the Teflon deposition in order to find a possible correlation between the amounts of Teflon adhered to the surfaces and the resultant wetting characteristics.

Contact angle measurements

Wettability tests (sessile drop method) were performed using a video-based optical contact angle-measuring system (Kruss GmbH, Germany). A tilting plate method was used to measure the advancing (θ_A) and receding (θ_R) water contact angles on textured surfaces as well on Teflon-deposited surfaces. A water droplet was placed on the target surfaces, and then the sample was slowly tilted while a CCD camera recorded changes in the droplet's shape. θ_A and θ_R

were measured using the last recorded image before the droplet base started to move. All contact angles were measured automatically using the software of the contact angle-measuring system. Measurements were performed under ambient laboratory conditions using water droplets of 10–12 μL volume. All data are given as an average value from at least ten independent measurements including $\pm 3^\circ$ standard deviation.

Characterization of the surfaces

The morphology of the laser-textured surfaces was inspected with scanning electron microscopy (SEM) and atomic force microscopy (AFM) in non-contact mode. After Teflon deposition, surface micro- and nano-textures were mainly analyzed with AFM. In general, between 5 and 20 μm scan sizes were used with simultaneous acquisition of topography, height, error and phase images. A Park Systems XE-100 AFM is used in non-contact mode with a silicon cantilever. An adaptive scan rate between 0.15 Hz and 0.25 Hz was utilized for all samples.

Results and discussion

The laser patterning technique used in this study is ideally suited for the generation of micrometer sized protruding structures on polished or unpolished silicon wafers. Figure 1 shows the SEM images of the laser-patterned surfaces obtained on the polished surface of a silicon wafer. Densely packed pyramid-like roughness features (pillars with pillar density of 0.8 pillar/ μm) of the order of 2 μm in size were produced on the silicon wafer surface. These surfaces were hydrophilic. Finite water contact angles could not be measured on the textured surfaces and for all practical purposes an apparent water contact angle of 0° was assigned to these surfaces. In Fig. 2, AFM topographical and error signal images are shown. Note that high-quality AFM image acquisition of surfaces with micrometer features is only possible when the deflection (or amplitude) signals are minimized. This is because the deflection and amplitude images are the error signals in AFM. If these signals are at a minimum, the error images obtained during scanning should look identical to topographical images. Indeed, both the topographical and the error signal images in Fig. 2 match perfectly, indicating that there exist no additional phases or impurities on the laser-textured surface as a result of laser exposure. Moreover, from these images, it is seen that, practically, there exists no single and isolated pyramidal roughness feature but rather the surface topography is made up of collective pyramidal structures which probably were carved out of larger surface bumps during laser ablation.

Fig. 1 Scanning electron microscope (SEM) images (*top* low magnification; *bottom* high magnification) from a laser-ablated area on a silicon wafer. Closely packed pyramid-like roughness features of average size of 2 μm can be distinguished

Figure 3 displays a high-magnification AFM topography of the patterned surface corresponding to Fig. 2 using a UV nanosecond laser at a fluence of 1.0 J/cm². Detailed morphology of the pyramid-like roughness features is very clear in this AFM image.

It is seen that four pyramid-like roughness features are packed into a 25 μm^2 area on the silicon surface. Also, the base of the pyramids is made up of a layered structure most probably originating from laser induced melting-solidification cycles [13]. Roughness analysis indicates that the base of the pyramids is about 2 μm wide and the top is 0.5 μm wide. This surface morphology namely, the size, depth and the frequency of distribution of the resultant protruding features, strongly depends on the laser fluence and the application technique. In the present case, the samples are prepared by irradiating the wafers with a line focus beam of about 20 mm long and 0.2 mm wide while rotating the wafer. The line focus is set radially starting slightly from

Fig. 2 AFM topographical (a) and error signal images (b) corresponding to a laser-textured surface using the UV nanosecond laser at a fluence of 1.0 J/cm^2

the center of the wafer, thus the actual shot dose, i.e., number of laser shots per unit surface area, is changing along the radius. The wetting of these surfaces was characterized as superhydrophilic, regardless of the applied laser fluence range. In fact, water droplets placed on these surfaces showed an apparent contact angle of 25° immediately after deposition; however, after a few seconds they

completely spread on the textured surfaces showing no finite contact angle.

When submicrometer Teflon particles are adhered to the surface by the triboelectric (contact friction) charging method described here, the surface morphology as well as the wetting of the original surface changes drastically due to the deposition of Teflon particles into the laser-formed asperities. Two main factors were found to influence the final composite surface wetting characteristics, namely, the substrate micro-roughness and the amount of Teflon adhered. Submicrometer Teflon particles used in the present study were well characterized and have been recently used in the fabrication of functional superhydrophobic polymer nanocomposites [19–22]. Figure 4 shows morphology of as-received Teflon powder. As-received powder is made up of agglomerated submicrometer Teflon particles as seen in Fig. 4. Figure 5 shows two 3D AFM topographic images of an untreated laser-textured surface and a triboelectrically deposited Teflon surface, respectively.

Fig. 3 Top AFM topography of a $5 \times 5 \text{ μm}$ area of the laser micro-textured silicon wafer surface. Bottom Typical roughness profile of the micro-textured surface

Fig. 4 Scanning electron microscope (SEM) image of the as-received Teflon powder before triboelectric charging for surface deposition

Fig. 5 **a** A 3D AFM topography (*large area scan*) of laser-textured silicon wafer surface before triboelectric Teflon adhesion and **b** 3D topographical detail of the resultant surface roughness after Teflon deposition. The resulting apparent water contact angle on the surface shown in (**b**) was measured to be 135°

The composite hydrophobic surface shown in Fig. 5b displayed an apparent water contact angle of 135° with contact angle hysteresis of 25° .

Since Teflon is intrinsically hydrophobic, the Teflon-coated surface also becomes hydrophobic but due to the underlying roughness the static contact angles exceed that of smooth Teflon surface which is around 110° . Detailed static water contact angle and contact angle hysteresis measurements showed that laser-textured silicon surfaces having an average surface roughness of $\sim 5 \mu\text{m}$ do not become superhydrophobic (contact angles $>150^\circ$) when coated with triboelectrically charged Teflon particles. Composite hydrophobic surfaces obtained this way showed relatively high contact angle hysteresis compared to self-cleaning superhydrophobic surfaces ($<10^\circ$) which on average was around 35° . In Fig. 6, detailed static water contact angle and contact angle hysteresis measurements are presented for Teflon-coated surfaces. Four different textured surfaces were used as the substrates as a result of four different laser fluences (0.5 , 1.0 , 1.5 and 2.0 J/cm^2). Within the fluence range studied, no major changes in the silicon wafer surface roughness were observed although the shape of the individual pyramid-like micro-features could be modified. However, this had no direct effect on the final wetting properties of the Teflon-coated surfaces. As seen, apparent static water contact angles center around 130° for all surfaces on average. Contact angle hysteresis, on the other hand, is approximately 35° on average. Although, these surfaces lack the ability to self-clean, indicated by the water contact angle measurements being less than 150° and contact angle hysteresis being greater than 10° , water droplets can still slide off the surfaces when they are adequately tilted $\sim 40^\circ$. This wetting property of the composite surfaces outperforms certain hydrophobic polymers such as smooth Teflon or poly(dimethyl siloxane) (PDMS). Smooth PDMS surfaces have high contact angle hysteresis close to 90° .

Within the laser fluence range studied, solventless triboelectric Teflon deposition technique results in surfaces with almost identical hydrophobicity. The main reason for this is attributed to the fact that the triboelectric coating creates

about $1.5\text{-}\mu\text{m}$ -thick Teflon films with the 150-nm Teflon powder. Therefore, a conformal coating was not possible with the resultant silicon wafer surface roughness range ($2\text{--}5 \mu\text{m}$) towards forming a hierarchical surface roughness necessary for superhydrophobicity.

In order to circumvent this problem, we have used silicon carbide sandpapers of various grit sizes to analyze the effect of surface micro-texture on the degree of final hydrophobicity as

Fig. 6 Static water contact angle (*top panel*) and water contact angle hysteresis on Teflon-coated laser-textured silicon wafers. Different colors in the graphs indicate surfaces created using different laser fluences ranging from 0.5 to 2.0 J/cm^2 . Ten random measurements were taken on each surface to investigate the wetting homogeneity on the surfaces

Fig. 7 Effect of amount of Teflon (PTFE) deposition on the degree of hydrophobicity as a function of surface roughness. The gray box indicates the roughness scales which could be fabricated on smooth silicon wafer surfaces with the present UV laser ablation technique. Higher surface roughness scales outside of this region are due to use of silicon carbide sandpapers of different grit sizes. The data set given ($CA > 150^\circ$, $CAH < 15^\circ$) indicates self-cleaning superhydrophobicity

a result of triboelectric Teflon particle deposition. Micro-textured SiC surfaces such as sandpapers have been recently used to fabricate highly conductive polymeric surfaces as well as oil repellent nanostructured polymer composites [15, 23]. Figure 7 shows that depending on the amount of Teflon adhered per unit area, surface hydrophobicity can be tuned from hydrophobic to self-cleaning superhydrophobic as a function of surface micro-roughness. In general, for micro-rough surfaces up to 60 μm , surface roughness less than 1.5 mg/cm^2 Teflon attachment is enough to render them superhydrophobic. The rectangular region in Fig. 7 indicates the roughness range which could be created by our UV laser ablation technique on silicon wafer surfaces compared to

Fig. 8 Effect of amount of submicrometer Teflon deposited on a sandpaper with an average roughness of 16 μm on the degree of final hydrophobicity expressed in terms of apparent water static contact angles and contact angle hysteresis. PTFE measurements were accurate to 0.1 mg/cm^2

Fig. 9 Effect of amount of submicrometer Teflon deposited on a laser-textured surfaces with an average roughness of 2 μm on the degree of final hydrophobicity expressed in terms of water static contact angles and contact angle hysteresis

typical commercial sand paper roughness grits. In Fig. 8, we show the changes in water contact angle and contact angle hysteresis as function of submicrometer Teflon deposited on a sandpaper having an average roughness of 16 μm . Best results (water contact angles exceeding 155° and contact angle hysteresis less than 20°) are obtained when approximately 0.5–0.7 mg Teflon per cm^2 is deposited on the surface.

A similar analysis conducted on laser micro-textured silicon wafer surface shows that much less amount of Teflon per unit area (0.05–0.15 mg/cm^2) is necessary to render the surface hydrophobic as shown in Fig. 9. As seen, the overall changes in both the apparent water contact angles and contact angle hysteresis due to the amount of Teflon deposition is not as significant as the silicon carbide sandpaper surfaces with higher roughness scales as shown in Fig. 8.

Finally, in Fig. 10, a photograph of a sandpaper (600 grit; average roughness, 26 μm) part of which was coated with the triboelectric Teflon deposition method is shown. In the photograph, the red line shows the border between

Fig. 10 Photograph of 600-grit silicon carbide sand paper part of which was rendered superhydrophobic by the triboelectric Teflon deposition method (inner region encircled by the red line). The untreated region is completely wetted by water whereas water droplets on the treated region is in a superhydrophobic state

the treated and the untreated regions. The untreated (hydrophilic) region was completely wetted by water whereas the treated region is completely superhydrophobic. The surface roughness ($\sim 26\text{ }\mu\text{m}$) corresponding to this 600 grit silicon carbide sandpaper is within the roughness range 15–65 μm which is considered ideal for forming superhydrophobic surfaces with the present solventless triboelectric Teflon deposition method.

Conclusions

Using the principle of triboelectric charging of insulating polymers, we have demonstrated a solvent- and chemical treatment-free technique to render rough surfaces hydrophobic and superhydrophobic with low contact angle hysteresis. Two types of rough surfaces were employed: (a) micro-machined ($\sim 5\text{ }\mu\text{m}$) silicon wafers using a nanosecond UV laser and (b) coarse ($>16\text{ }\mu\text{m}$) silicon carbide sandpapers of various grit sizes. These surfaces were superhydrophilic before the treatment. Submicrometer Teflon particles were initially spread on these rough surfaces in a coarse manner and then a clean cellulosic foam surface was rubbed against the Teflon-deposited surfaces to smooth and spread out the particles, and at the same time electrostatically charging them. These particles adhered strongly to the textured surfaces due to electronic charging during rubbing. Depending on the underlying surface roughness, the surfaces could also be rendered superhydrophobic. Laser-textured surfaces did not achieve superhydrophobicity due to their low surface roughness ($\sim 5\text{ }\mu\text{m}$). Contact angles as high as 130° were measured on these surfaces with contact angle hysteresis around 35° . On the commercial sandpaper surfaces with higher surface roughness, superhydrophobicity can be achieved only if the surface micro-roughness is within a certain range which was found to be 15–60 μm .

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