

Durable Lotus-effect surfaces with hierarchical structure using micro- and nanosized hydrophobic silica particles

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ABSTRACT

Surfaces with a very high apparent water contact angle (CA) and low water contact angle hysteresis (CAH) exhibit many useful characteristics, among them extreme water repellency, low drag for fluid flow, and a self-cleaning effect. The leaf of the Lotus plant (*Nelumbo nucifera*) achieves these properties using a hierarchical structure with roughness on both the micro- and nanoscale. It is of great interest to create durable surfaces with the so-called "Lotus effect" for many important applications. In this study, hierarchically structured surfaces with Lotus-effect properties were fabricated using micro- and nanosized hydrophobic silica particles and a simple spray method. In addition, hierarchically structured surfaces were prepared by spraying a nanoparticulate coating over a micropatterned surface. To examine the similarities between surfaces using microparticles versus a uniform micropattern as the microstructure, CA and CAH were compared across a range of pitch values for the two types of microstructures. Wear experiments were performed using an atomic force microscope (AFM), a ball-on-flat tribometer, and a water jet apparatus to verify multiscale wear resistance. These surfaces have potential uses in engineering applications requiring Lotus-effect properties and high durability.

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1. Introduction

Surfaces with superhydrophobicity (apparent contact angle greater than 150°) and self-cleaning effect (contact angle hysteresis less than 10°) are instrumental in many technical and industrial applications, such as self-cleaning windows, low-drag surfaces for microchannels, and surfaces requiring antifouling characteristics [1–4]. The leaf of the Lotus plant (*Nelumbo nucifera*) has a hierarchically structured surface, which is responsible for its superhydrophobic and self-cleaning qualities [5–7]. The microscale roughness is formed by convex cell papillae, and the nanoscale roughness is formed by hydrophobic wax tubules. The Lotus leaf has a contact angle (CA) of 164° and contact angle hysteresis (CAH) of 3° . The very low CAH value allows a water droplet to roll off the surface instead of slide, taking contaminants along with it. A hierarchical structure allows for increased formation and stability of air pockets, reducing the contact area of an applied water droplet with the surface. This results in low CAH, tilt angle, and adhesive force [1–3,8,9].

Surfaces inspired by the Lotus leaf have been fabricated using a number of techniques, such as soft lithography, self-assembly, electrodeposition, and imprinting [3,10–18]. For industrial applications, these surfaces must possess mechanical durability. For applications such as window glass or self-cleaning toilets, surfaces

must retain superhydrophobicity after impingement of water. Durable, Lotus-effect surfaces have previously been created, among other methods, by depositing carbon nanotubes (CNTs) onto epoxy micropatterns [19].

In the present study, hydrophobic silica particles of different sizes and with high hardness were used for both micro- and nanoscale roughness to create mechanically durable, Lotus-effect surfaces with hierarchical structure. Superhydrophobic surfaces using silica particles have been created by drop casting, chemical deposition and sol-gel processes [11,20,21]. In this study, hierarchical structures resistant to mechanical wear were created with silica particles using a simple spray process. First, silica nanoparticles of two different sizes were deposited onto micropatterned surfaces to confirm superhydrophobicity and low CAH. Then, silica microparticles were substituted for the micropattern to create the microscale roughness. The pitch between pillars for the micropatterns is known, and the average pitch between microparticles on a surface can be determined through SEM imaging. The CA/CAH behaviors of micropatterns and surfaces with microparticles are examined across a range of pitch values to compare the use of microparticles and micropatterns. Hierarchical surfaces were also created using a combination of microparticles and nanoparticles. Wear experiments were conducted for the microparticles and nanoparticles using an atomic force microscope (AFM), a ball-on-flat tribometer, and a water jet apparatus. In all experiments, the wear of an epoxy resin on a flat surface is used as a benchmark for comparison.

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2. Theoretical basis

Combining roughness on both the micro- and nanoscale results in a hierarchically structured surface, which is the basis of the Lotus effect. Fig. 1 [19] illustrates the wetting behavior of flat, nanostructured, microstructured, and hierarchically structured surfaces. The lowest area of contact between the droplet and the surface occurs in the case of the hierarchical structure, which can be expected to have highest CA and lowest CAH as a result. In addition, the multiscale roughness allows for more stable air pocket formation, guarding against destabilizing factors on both the micro- and nanoscale.

Wetting of a structured surface can be generally described in terms of different wetting regimes, the main two of which are the fully wetted (Wenzel) and composite (Cassie–Baxter) regimes [22,23]. In the Wenzel regime, the liquid fully penetrates the gaps between asperities. In the Cassie–Baxter regime, the droplet sits on top of the asperities, creating air pockets. The micropatterns used in this study consist of cylindrical pillars with uniform height (H), diameter (D), and pitch (P). The droop of the droplet (δ) between pillars (Fig. 2) can be found if these parameters are known. It has been shown that for this geometry, the droop is expected to fully penetrate the air gaps when

$$\frac{(\sqrt{2}P - D)^2}{R} \geq H \quad (1)$$

where R is the droplet radius [24]. This represents the transition from the Cassie–Baxter to the Wenzel regime. For a given height and diameter, the pitch value can be determined above which transition to the Wenzel regime will occur. Because of the sudden loss of air pockets and increased liquid–solid interface, a droplet that has

transitioned to the Wenzel regime can be expected to have lower CA and higher CAH compared to before the transition.

A microstructure can also be formed by depositing micro-sized particles on a flat surface. Fig. 3 illustrates the use of both particles and a patterned surface to form microstructures and hierarchical structures. Knowing the geometry of the particles, transition to the Wenzel regime can similarly be predicted to occur above a certain pitch value. If a surface with microparticles is providing an adequate microstructure, it should be expected to exhibit similar trends in CA/CAH with varying pitch as those seen on a uniform micropatterned surface.

3. Experimental details

3.1. Samples

Epoxy micropatterns were fabricated by replicating a Si micro-patterned surface through soft lithography. This method has been used by Jung and Bhushan [19], in which a negative replica is created using polyvinylsiloxane dental wax, and a positive replica is then made with liquid epoxy. The epoxy is weakly hydrophilic, with a CA of $80^\circ \pm 2^\circ$ for a flat epoxy surface with no micropattern.

Hierarchically structured surfaces using micropatterns were created by depositing nanoparticles onto the micropatterned epoxy substrates using an established spray method [19]. Nanostructured surfaces were also created by spraying particles onto flat substrates. Silica nanoparticles of 10 nm (± 1 nm) and 50 nm (± 15 nm) were created through continuous flame hydrolysis of SiCl_4 , and hydrophobized through silane treatment (Evonik-Degussa Corporation, Parsippany, New Jersey). In order to spray the particles onto the surfaces, they were first dispersed uniformly in

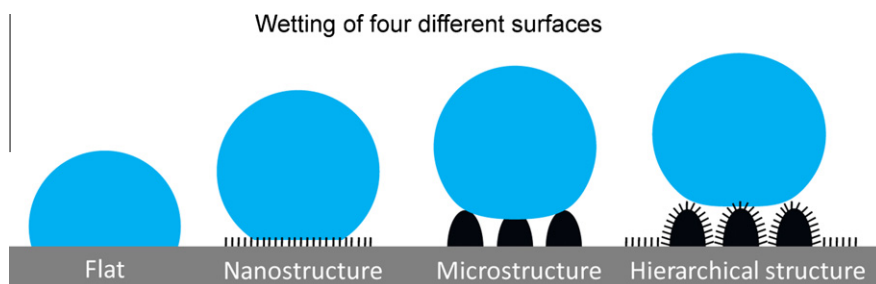


Fig. 1. Schematic of four different surface structure types and their wetting behaviors. The largest contact area between the droplet and the surface occurs on flat and microstructured surfaces, but is reduced on nanostructured surfaces and minimized on hierarchically structured surfaces [19].

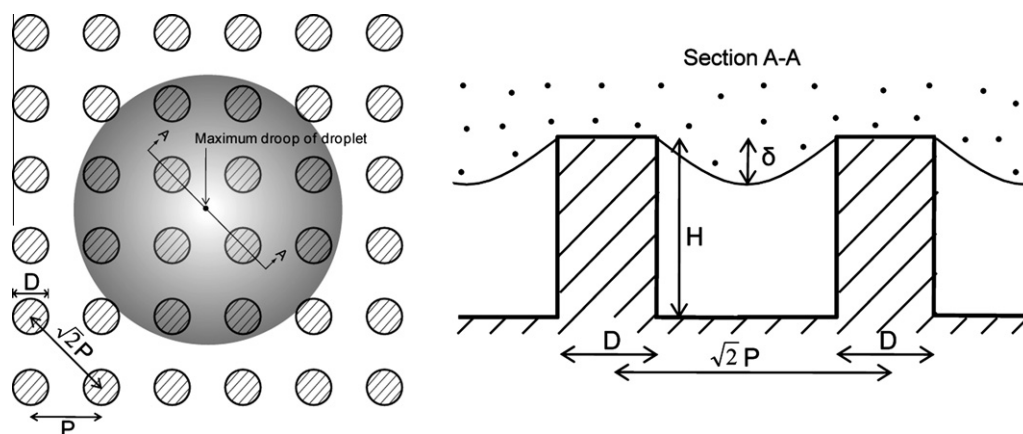


Fig. 2. Schematic of a small water droplet suspended on a superhydrophobic surface consisting of a uniform array of cylindrical pillars. The maximum droop of the droplet occurs in the center of the square formed by four pillars.

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