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A simple fabrication method for mechanically robust superhydrophobic surface by hierarchical aluminum hydroxide structures

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ABSTRACT

Superhydrophobic surfaces with uniformly superhydrophobic surface where nano-scale structures were fabricated by alkali surface modification method and self-assembled monolayer coating. To enhance mechanical durability of the superhydrophobicity, we propose the fabrication process for dual-scale hierarchical structures combining both microstructure *via* sandblasting techniques and the nano-structured aluminum hydroxide layer. The superhydrophobic surfaces fabricated by both methods exhibited a high water contact angle and very low contact angle hysteresis. By forming the hierarchical structure, the mechanical durability of superhydrophobic aluminum hydroxide surface was improved. The resulting hierarchical structures are suitable for diverse applications of aluminum in various industrial areas, including self-cleaning, anti-frosting, and microfluidic devices for rigorous environments.

1. Introduction

Surfaces with a very high water contact angle larger than 150° and a low contact angle hysteresis of less than 10° are generally known as superhydrophobic surfaces. These surfaces have attracted much interest in industry, because many studies have shown that these surfaces have properties such as self-cleaning [1–3], anti-corrosion [4–6], anti-frosting [7–9], fluid drag reduction [10–12], non-sticky to oil and inks [13,14]. A superhydrophobic surface can be attained by forming a hierarchical roughness structure, that is, a nanometer-sized structure superimposed over a micrometer-structure, similar to those of the lotus leaf. Moreover, many recent studies have reported that a hierarchical roughness ensures super-hydrophobicity even after the surfaces are worn away [15–17].

Aluminum is widely used in various industrial fields as a basic material for numerous mechanical components. For this reason, making an aluminum surface superhydrophobic has many application possibilities. However, only a few methods for fabricating superhydrophobic aluminum surfaces have been reported. Qian et al. reported a dislocation-selective chemical etching technique for superhydrophobic aluminum surfaces with contact angles larger than 150° [18]. Guo et al. fabricated a superhydrophobic surface using chemical etching by immersing aluminum in sodium hydroxide (NaOH) and then decorating with perfluorononane [19]. On the other hand, anodization combined with a low-temperature plasma treatment was used to form hierarchical structures on aluminum surfaces [20]. Wu et al. realized a superhydrophobic aluminum surface by forming alumina nanowire forests *via* high field anodization [21]. Kim et al. [22] and Jeong et al. [23] reported a self-aggregation phenomenon for alumina nanowires by the anodization of aluminum. However, chemical etching induces serious problems such as damage to the aluminum substrate and a non-uniformly etched site. The anodization method consumes a large amount of electricity and causes defects in the alumina layer from a failure to control the current.

Recently, Seo et al. reported a fabrication method for an aluminum hydroxide layer that uses alkali surface modification [24,25]. The fabricated aluminum hydroxide layer is composed of gibbsite (γ-$Al(OH)_3$) and has a flake-like nanostructure. By means of this method, uniformly distributed nanostructures on an aluminum surface can be obtained quickly and simply. They demonstrated that the wettability of the fabricated surface is superhydrophilic.

Herein, we report uniformly superhydrophobic surface where nano-scale structures were fabricated by alkali surface modification

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method and self-assembled monolayer coating. And, we propose the fabrication process for dual-scale hierarchical structures combining both microstructure *via* sandblasting techniques and the nanostructured aluminum hydroxide layer to enhance mechanical durability of the superhydrophobicity.

This process has the following key advantages: industry compatibility, robustness, and a uniform superhydrophobic surface. We particularly investigated the wetting characteristics by measuring the water contact angle and the improvement in the mechanical robustness by an abrasion test.

2. Experimental details

The fabrication process for superhydrophobic nanostructured and hierarchical aluminum hydroxide surfaces are shown schematically in Fig. 1. Industrial grade aluminum sheets (99.5%, 50 mm × 40 mm × 1 mm) were used in all of the experiments. The formation of nanostructured aluminum hydroxide was carried out in a 0.05 M NaOH solution at 80 °C for 5 min. After this, the specimen was immediately immersed in 100 °C deionized water for the subsequent stabilization process. A microroughness structure was prepared by sandblasting the aluminum sheet with sand particles. The size of the sand particle was 500 mesh, and the particles were ejected from a nozzle using compressed air at a pressure of 6 kgf cm^{-2}. After sandblasting, the aluminum sheet was cleaned with deionized water. The hierarchical surface was fabricated by forming aluminum hydroxide layer on the microroughened aluminum specimen. Finally, superhydrophobic nanostructured and hierarchical surfaces were obtained after self-assembled monolayer coating with heptadecafluoro-1,1,2,2-tetrahydrodecyltrichlorosilane (HDFS, Gelest) was applied on the specimen. The specimen was dipped in a mixture of *n*-hexane and HDFS (volumetric ratio 1000:1) for 10 min. They were then washed with distilled water and dried in the oven (105 °C) for 1 h.

The static contact angle, contact angle hysteresis, and sliding angle on the fabricated surface were measured using a drop shape analysis system (DSA-100, Kruss). The contact angle hysteresis was obtained by measuring the advancing contact angle and receding contact angle at the maximum sliding angle by which the drop would move by gravity. A 5-μl droplet of distilled water was used for this purpose. The contact angle results were averaged over at least ten measurements on different areas of each specimen at room temperature. Scanning electron microscopy (SEM; JSM-7401F FE-SEM, JEOL) images were obtained to investigate the surface morphology.

In order to evaluate the robustness of the superhydrophobic surface, abrasion test illustrated in Fig. 2 was performed. The fabricated superhydrophobic surface was tested facing an abrasive film (1 micron grade Imperial™ lapping film, 3 M). Applying weights to the specimen, the surface was moved in one direction with 5 mm/s at a stroke of 15 cm. The preload was applied on each specimen and increased up to 1000 g. The static contact angles and hysteresis changes of the superhydrophobic surface were measured after abrasion test.

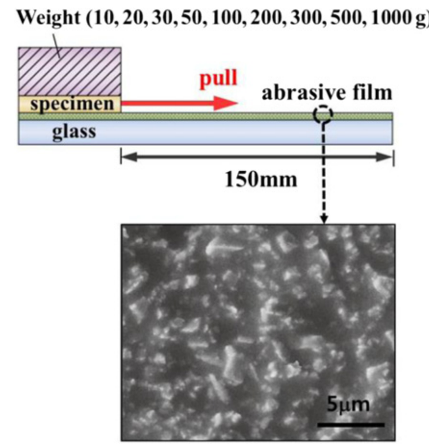

Fig. 2. Schematic of abrasion test. (Inset: SEM image of abrasive film). The abrasion tests were conducted by changing applied weights from 10 g to 1000 g.

3. Results and discussion

The surface morphologies of several specimens were examined using SEM, as shown in Fig. 3. Fig. 3(a) shows the surface of industrial grade normal aluminum. Fig. 3(b) shows that the aluminum surface had microscale unevenness and its morphologies changed significantly after sandblasting. Fig. 3(c) shows that the surface of the aluminum was covered with a flake-like aluminum hydroxide layer. The thickness of this aluminum hydroxide layer was around 500 nm with a nanoscale surface morphology. Fig. 3(d) reveals that the hierarchical structure included the microscale structure formed by sandblasting and nanoscale aluminum hydroxide structures over the microroughened structure.

The wettabilities of several specimens were characterized in detail using contact angle measurements, as shown in Fig. 4(a). The normal aluminum specimen exhibited slight hydrophilicity. For a droplet in contact with a rough surface, the contact angle was explained by the Wenzel state [26]. According to the Wenzel state, the contact angle of the aluminum surface became more hydrophilic because of the increase in its roughness after sandblasting.

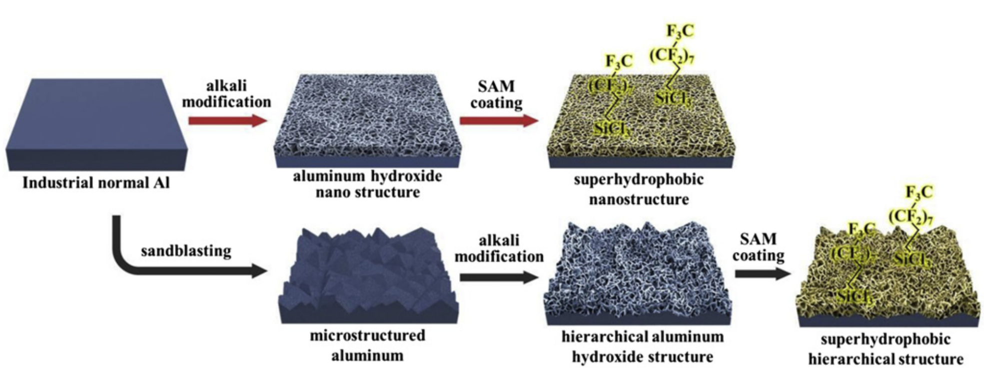

Fig. 1. Steps in the fabrication of superhydrophobic nanostructured and hierarchical aluminum hydroxide surfaces.