



Fabrication of pinecone-like structure superhydrophobic surface on titanium substrate and its self-cleaning property

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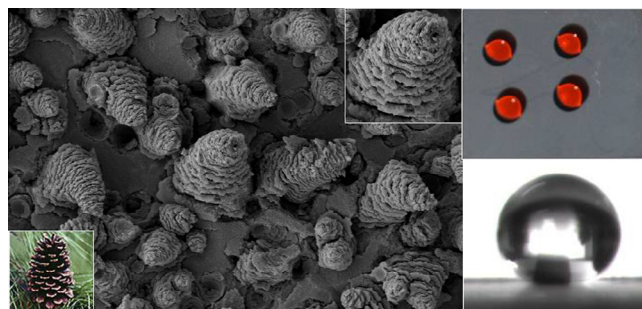
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HIGHLIGHTS

- A pinecone-like superhydrophobic surface was fabricated on the Ti substrate.
- Superhydrophobic surface with long-term stability and self-cleaning property.
- The largest static contact angle of 161.4° and ultra low slide angle of near to 0° were obtained.
- The surface show superhydrophobic properties for other droplets (tea, vinegar, coffee and milk).

GRAPHICAL ABSTRACT



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ABSTRACT

A superhydrophobic surface was fabricated via an economical, simple, and highly effective anodization process combined with a low surface energy modifying. The surface exhibited rough microprotrusions consist of nanoflakes morphology, which is quite similar to the structure of the pinecone. The pinecone-like microstructures and the wettability of the surfaces could be easily adjusted by anodization parameters including anodization time and voltage. Besides a maximum water contact angle of $161.4^\circ \pm 1.5^\circ$ and an extremely low sliding angle of about 0° , as prepared surface with pinecone-like hierarchical rough structure together with a chemical modification exhibited superhydrophobicity with other liquids such as tea, vinegar, coffee and milk. Moreover, this type of superhydrophobic surfaces prepared by this method was proven to have a good long-term stability, mechanical robustness, self-cleaning effect and anticorrosion.

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1. Introduction

In natural, many plants and insects exhibit excellent wettability performances, such as lotus leaves [1], rose petals [2], rice leaves [3], water striders [4], butterfly wings [5], bird's feathers [6], and so on. These biological organisms with unique surface wettability

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have been demonstrated, especially the intrinsic superhydrophobic and self-cleaning property of lotus leaf. Inspired by nature, the research of superhydrophobic faces, typically defined as a droplet water angle (CA) higher than 150° as well as a sliding angle (SA) less than 10° , have gained the attention of both the scientific and industrial communities due to considerable potential application values, such as antifouling [7], self-cleaning [8], anticorrosion [9], anti-icing [10], oil–water separation [11], and drag-reduction [12].

Among various metals and their alloys, titanium (Ti) and its alloys, as one of the most widely used metal materials, have

attracted considerable interest in the aerospace, medical care, and electronic industries for their superior physical and chemical performance. Although Ti has high anticorrosion due to a compact and chemically stable oxide film formed spontaneously on the metal surface, there is a need to raise the anticorrosion property of the Ti in some harsh environment condition such as water, corrosive medium or moist environment. Therefore, superhydrophobicity (self-cleaning and anticorrosive properties) will improve surfaces comprehensive performance and extend its applications as engineering materials, such as providing effective corrosion barrier for Ti parts used in special environment like ship construction [13], and reducing the fluidic drag force and fluid resistance [14] for prolonging the service life of the whole system. It is well documented that the wettability of a solid surface is governed by chemical compositions and geometric structures. Thus, superhydrophobic surfaces can be usually achieved by two typical ways, which are creating hierarchical micro-/nano-structures on the hydrophobic substrates [15,16] or an appropriate rough surface modified with low surface energy materials [17]. The surface energy can be easily tailored by the low surface energy compound (e.g., long-chain fatty acid or fluorosilane). Therefore, the key factor is the creation of the micro/nano roughness to obtain surfaces superhydrophobic. To get superhydrophobicity property of surfaces, various fabrication methods have been investigated, such as sol-gel technique [18], chemical etching [19], chemical vapor deposition [20], spin coating [21], solution-immersion [22], anodization method [23–25] and thermal oxidation [26]. For example, Fadeeva et al. [27] and Jiang et al. [28] created superhydrophobic Ti surface without modification by femtolasers ablation, which the water CA reached as high as 166° and 158°, respectively. Qu et al. [19] was obtained the superhydrophobic surface using chemical etching. Zou et al. [29] developed a stable superhydrophobic surface on Ti substrates using sandblasting and ion etching. Gnedenkov et al. [30] used plasma electrolytic oxidation with nanocomposite superhydrophobic coating to create an anticorrosive film on Ti substrate. Lu et al. [31] prepared superhydrophobic surface after modifying with low surface energy material on Ti substrate using anodization technique. However, many of these methods have potential shortcomings for practical applications, such as severe conditions, complex processing steps and devices, expensive materials, and uncontrollable geometric structures, etc. In order to overcome the aforementioned disadvantages, a facile and cost effective approach is highly desirable for the fabrication of superhydrophobic surfaces.

Accordingly, in comparison, the anodization method is considered to be one of the most promising techniques to produce large areas with the featured advantages of low cost, precise control, facile and rapid fabrication of surface roughness. Although the fabrication of the superhydrophobic Ti surface by anodization method has been reported, a higher temperature (60–70 °C) [31], a longer time [13] or a strong corrosive electrolyte [24,25] was needed in anodization processes. In the present work, the anodization method was used to fabricate pinecone-like structures on Ti substrate at room temperature for a short time and followed by low surface energy modifications to fabricate superhydrophobic surface. The pinecone-like structures that influence the hydrophobicity were easily controlled by anodization parameters such as anodization time and voltage. However, compared with the commonly reported nanotube array structures [24], the necessary microgeometric structures of non-stick superhydrophobic surface are more easily obtained. Furthermore, the as-prepared surface not only exhibited superhydrophobic stability after a long-term exposure to air and water jet impact test, but also exhibited excellent superhydrophobic properties in harsh conditions such as tea, vinegar, coffee, and milk, which is important to a wider range of future applications in the industry and in real life.

2. Experimental

2.1. Materials

High purity Ti sheets (99.99%) were used as substrates, provided by SUNTEC Titanium LTD, Dalian. Ammonium fluoride (NH₄F) and oxalic acid (C₂H₂O₄·2H₂O) were purchased from Sinopharm Group Chemical Reagent Co. Ltd. The other experimental chemicals used with an analytic grade were purchased from the Tianjin Kermel Chemical Regent Co., China, and distilled water lab-made.

2.2. Specimen preparation

Anodization was performed in a two-electrode configuration with Ti sheet as the working electrode and the graphite sheet served as the counter electrode under a computer-controlled programmable AC/DC Power Sourcemeater (APS-1102) at room temperature. Prior to anodization, the aluminum plates were cut into small pieces with a size of 25 mm × 12 mm × 0.25 mm and ultrasonically cleaned sequentially in acetone, anhydrous ethanol and distilled water for 15 min, respectively, then the cleaned Ti sheets were chemically polished in a mixed solution of HNO₃ and HF (8:1 in volume) for 2–4 min, followed by rinsing with deionized water and dried in air. Next, polished Ti sheets were anodized in the mixture electrolyte of different concentration NH₄F and C₂H₂O₄ under the range of DC voltages from 10 to 70 V for different time from 5 to 120 min to obtain different microstructured surface. After anodization, the samples were rinsed thoroughly with deionized water and subsequently dried with cold air. To achieve the superhydrophobic Ti surface, the samples were immersed in a 5 mM ethanol solution of stearic acid (STA) and then taken out of the solution, rinsed repeatedly with deionized water to remove the unnecessary STA and subsequently naturally dried in air. Finally the superhydrophobic surface was obtained.

2.3. Specimen characterization and test

The topography of the surface was observed using a Zeiss Neon 60 field emission scanning electron microscopy (SEM). The surface compositions were characterized by X-ray energy dispersive spectroscopy (EDS) associated with SEM. The Fourier transform infrared (FTIR) spectrum of the surface was recorded on a FTS-3000 with KBr pellet technique in the transmission mode. The CAs were measured on a JC2000D2 contact angle/interface system at ambient conditions and room temperature. The CAs reported here were the mean values measured with a 5 μL water droplet at five different positions on each sample. The optical pictures were obtained by a digital camera (Canon). The mechanical stability of the surface was evaluated by high velocity water jet impacted onto the overall surface for at least 60 s. The electrolyte temperature was adjusted using thermostatic heating magnetic stirrer (Yuhua Instrument Co., Ltd., China). The self-cleaning behavior of the surface was studied by sprinkling with sparse plant ashes on the surface, which was all washed off by adding water drops dropwise onto the upper side of the plant ashes surface with a tilting angle of about 7–10°. The corrosion resistance of the as-prepared superhydrophobic surfaces was evaluated based on immersion test in 3.5% NaCl solution by measurement of CAs.

3. Results and discussion

3.1. The fabrication of the pinecone-like structure and XRD patterns

Type large-area SEM images of the resultant TiO₂ surface microarray synthesized in the mixture electrolyte of 0.3 M

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