



Preparation of high wear-resisting superamphiphobic robust film by self-assembled monolayer surface reaction

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ABSTRACT

Artificial superamphiphobic surfaces have been arousing great attention in recent years for improved durability in practical applications. In this study, composite of dual-sized porous silica of micro-/nanoparticles and epoxy resin was coated on the glassy substrate as the micro-/nanostructure, then 60 nm porous silica particles modified by 3-aminopropyltriethoxysilane were covered on it. The monolayer of low-surface-energy fluoride-chains was grafted by click reaction of 2-(((3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10- heptadecafluorodecyl)-oxy)-methyl)oxirane with amino groups in the micro-/nanostructure surface. The low free-energy long perfluorinated chain and topological features established a super-repellent surface. The surface was of the superamphiphobic property with a wide range of liquids (surface tension ranging from 72.0 to 27.2 mN m⁻¹). Because of EP's wearability and the long perfluorinated chains' automatically migrating from the inner surface of porous silica particles to the new surface, the super-repellent surface is of not only the self-cleaning feature like the lotus' effect, but also the durability, the self-healing and high wear-resisting abilities, even after it was heavily scratched by the knife or abraded by sandpaper. After the superamphiphobic coating had been rubbed, under a weight of 100 g, 40 sandpaper abrasion cycles, the coating still keeps its contact angles (CAs) to water; diiodomethane and ethylene glycol over 155 ± 2°, and all its sliding angles (SAs) lower than 2.7 ± 1°, respectively. These abilities are anticipated to have important practical applications because of their satisfactory of the long-life use in outdoor and large-scale fabrications.

1. Introduction

Many surfaces in nature, including various plant leaves [1,2], legs of the water strider [3], troughs on the elytra of desert beetles [4], and lotus leaf [5,6], are superhydrophobic, displaying apparent contact angles with water greater than 150° and low contact angle hysteresis. Scientists attribute it to the complementary roles of the two key surface parameters, surface energy and roughness [7–9]. Researchers had revealed that the superhydrophobicity of the surface, characterized as a water contact angle (CA) higher than 150° and a sliding angle (SA) lower than 10°, is responsible for this special wetting behavior [10]. Inspired by the extreme water-repellency and self-cleaning property of low surface energy bio-characteristics in the natural world, people are interested in the phenomenon that the water droplet can easily roll down from the leaf and takes the dirt away without wetting the surface [11]. Up to now, a great number of elegant methods have been reported to acquire Cassie-Baxter's state superhydrophobic surface, these

approaches can be generally divided into two categories. One is in situ creation of rough surface structures on substrates by chemical etching [12], solution or solvent treatment [13,14], microlithography [15,16], sandblasting [17], and so forth. However, these approaches are usually limited to special substrates, and sometimes, further treatment with low surface energy compounds has to be conducted [13,16]. Another is ex situ casting or deposition of superhydrophobic coatings on the substrates. This strategy is versatile for creation of a superhydrophobic surface and can be executed by three routes: (a) creating micro/nanostructure surfaces via deposition of dual-sized particles or nanoparticles and then casting a layer of low surface free energy materials [18,19]; (b) casting low surface free energy coatings and further creating rough structures on the outmost surfaces of the coatings via electron irradiation [20,21], plasma treatment [22], nanocasting [23]; (c) casting polymer solutions or polymer/particles (nano)composite solutions and subsequently drying to produce superhydrophobic surfaces by phase separation [24–26] or self-organization [27–31].

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Due to the promising application in reality, artificial superhydrophobic surfaces have been arousing great attention not only because of its scientific background but also for wide range of technological applications including self-cleaning [32,33], antifouling [34–36], anti-icing [37–39] and oil-water separation [40].

However, superoleophobic surfaces that resist wetting of liquids with much lower surface tension are extremely rare [41]. For instance, the springtail cuticle, which exhibits a highly textured, hierarchically arranged surface pattern by geometrical factors, can't reach the superoleophobic performance [42–45]. Superoleophobic surfaces can be rather complicated, but they have great potential applications in antifouling from hazard chemicals and biological contaminants [46]. Although any solid surface can be characterized as superoleophobic as long as its contact angle with an oily fluid is greater than 150° , the surface properties revealed from the contact angle measurement using different contacting oils could be considerably different. For example, a surface that is superoleophobic to certain oily fluids may have lower repellency or even be wettable by other oily fluids of a lower surface tension. It is normally easy to make a surface super-repellent to oils of a high surface tension, but difficult to prepare superoleophobic surfaces against oily fluids that have a surface tension below 35 mN m^{-1} . Additionally, most super-liquid-repellent surfaces have poor durability [47]. Chemical oxidation from exposure to air, a special chemical environment, strong light, or physical rubbing could cause the surfaces to lose their super-repellency permanently. It is imperative to improve the durability for practical applications [48]. The durability includes the corrosion resistance, the self-healing ability, the strong adhesive ability and the mechanical friction performance. The bioinspired self-healing ability has been proposed to be a promising solution to improve the durability of synthetic superhydrophobic surfaces [49,50]. Organic fluoride or polymer containing fluorine can effectively solve weather aging, chemical corrosion and chemical oxidation of super-liquid-repellent surface. Li et al. [51] reported a self-healing superhydrophobic coating that was prepared by chemical vapor deposition (CVD) of a fluoroalkyl silane on a layer-by-layer assembled porous surface, and self-healing ability was derived from the reacted fluoroalkyl silane embedded in the rigidly flexible coating layer. Wang et al. [52] also reported the formation of a self-healing superamphiphobic surface on anodized alumina by filling the intrinsic pores with one low-surface energy liquid. The strong adhesive ability is another essential for the superamphiphobic surface. Great progress has been made to develop mechanically robust superhydrophobic surfaces and laundering-durable superhydrophobic fabrics [53,54]. In fact, the biggest obstacle to the

deployment of the artificial superhydrophobic surfaces in practical applications is their vulnerability to mechanical abrasion [51,55–57]. Mechanical abrasion on the superhydrophobic surfaces not only can destroy the microscopic rough structures that are essential for superhydrophobicity [48,54,58], but also can remove most of the hydrophobic surface layer on the superhydrophobic surfaces. Both factors lead to a decline in their nonwetting property [59,60]. So, the superamphiphobic surface being of the self-healing ability, the strong adhesive ability and the mechanical friction performance, especially, after abrasion, it is still superamphiphobic, is promising for the wide application of the reality. To our joy, Lu [32] prepared robust self-cleaning surfaces by commercial adhesives which can endure knife-scratch, and even 40 abrasion cycles with sandpaper.

Tailoring the chemical compositions of material surfaces is the other key factor to prepare the low-energy surface except the key factor of surface micro-/nanometer roughness. Self-assembled monolayer (SAM) by chemical modified the micro/nanostructure surface is an effective method. Usually, self-assembled material with low surface free energy is just monomolecular layer. So, it not only spares the expensive composition, but also keeps the original concave-convex surface. Perfluoroalkylsilanes are common compounds used for low surface energy chemical modification. The head group of perfluoroalkylsilanes, i.e., trichloro-, trimethoxy- or triethoxysilane, is responsible for the anchoring of the molecules onto the substrate. Hydrolysis of head group such as trichloro-, trimethoxy- or triethoxysilanes and subsequent reaction with the $-\text{OH}$ groups on the surface to obtain a polymerized network is an important step. However, the self-assembly process depends on several parameters, such as the water content, the utilized solvent, the age of the solution, the deposition time and the applied temperature. All of these factors play an indispensable role in the formation of the monolayer and might have a significant influence also on the monolayer formation process itself. Parameters and influence on monolayer formation process are rather difficult to control. It is imperative to find out the method to make the high-yield and no-by-products self-assembled monolayer.

In the study, we reported one kind of superamphiphobic coating. Firstly using the click reaction of amino group with epoxy group of 2-(((3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-heptafluorodecyl)-oxy)-methyl)oxirane (HDFO) to self-assembled monolayer on the micro-/nanostructure surface, we fulfilled the roughness and low-free-energy surface. The coating is self-healing, robust, especially high wear-resisting. It is particularly advantageous because it is efficient, rapid and stoichiometric reaction. It neither requires expensive or toxic catalysts

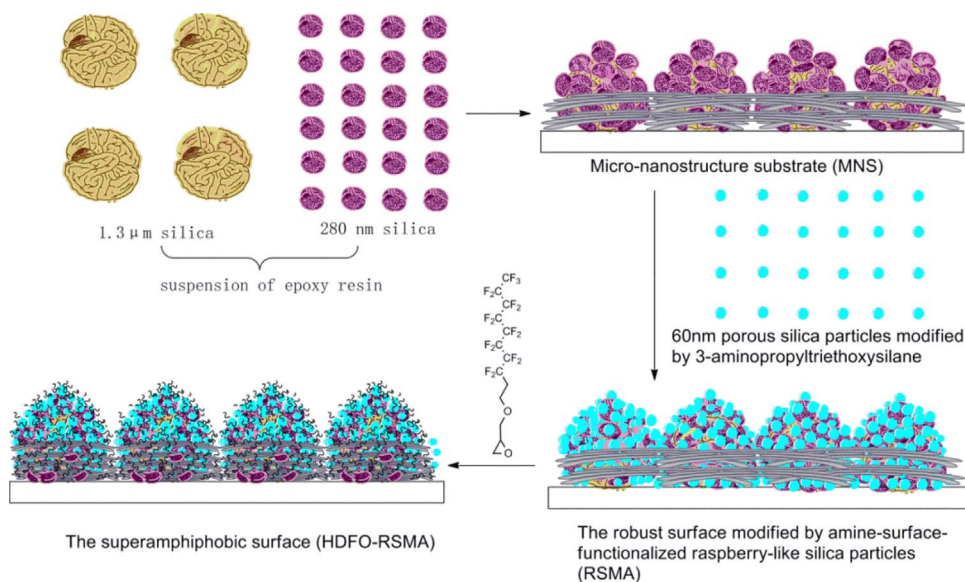


Fig. 1. Schematic illustration of fabricating high wear-resisting superamphiphobic robust surface by self-assembled monolayer surface reaction of amino group with fluorinated epoxy.

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