



Research paper

A novel strategy for durable superhydrophobic coating on glass substrate via using silica chains to fix silica particles



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ABSTRACT

The practical application of superhydrophobic coatings on glass is usually restricted by their poor wear resistance due to the insufficient adhesion. A double-silica-layered structure was proposed to reinforce the coating adhesion on glass substrate. The wettability, surface morphologies, and chemical composition were investigated by water contact angle measurement, scanning electron microscopy (SEM), and fourier transformed infrared (FTIR) spectroscopy. The prepared superhydrophobic coating displays a good wear-resistance by emery paper and sand abrasion, which also has excellent thermal stability and UV resistance. This strategy shows a bright future for durable superhydrophobic coating on glass.

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1. Introduction

The magical and unique features in ecology environment arouse scientific interests to transfer them into practical applications [1]. Water-repelling as one of the features, similar to the superhydrophobicity of lotus leaf [2,3], is desirable and promising in various applications such as moisture protection for building materials [4], efficient transport for microfluidic devices [5], self-cleaning for solar apparatus [6], and oil/water separation for water treatment [7]. An outstanding property of water-repelling can be fulfilled by superhydrophobic surfaces with micro/nano-sized roughness and hydrophobic chemicals that can render a water contact angle above 150°. However, the micro/nano-sized roughness and hydrophobic chemicals on the surface are usually very brittle due to the poor mechanical strength and inferior thermal/chemical stability [8]. They thus could be easily destroyed by various attacks such as sand abrasion (e.g. sand storm), thermal heat (e.g. high-temperature solar heat collector), and ultraviolet degradation in outdoor application, which can tremendously degrade the hydrophobicity and further restrict their commercial use.

A satisfactory superhydrophobic surface must be wear resistant, long-term stable at high temperature and UV irradiation, as well as easy to fabricate. So far, the preparation of durable superhydrophobicity is still a severe challenge, and much effort has been made to improve the coating robustness and durability [9]. For example, mechanical durability can be promoted by taking advantage of high-adhesion epoxy resin [10]. Hydrophobic chemicals as a surface modifier without any decomposition and softening at high temperature can present a good thermal stability for nano-silica-based superhydrophobic coatings [11]. UV resistance can be attained by employing UV inert substance (e.g. fluorosilane and silica) and passivating the photoactive species (e.g. SiO₂-coated TiO₂) [12].

Wear resistance is one of the most attractive properties for surface durability. With regard to superhydrophobic surface, it is recently reported that durable superhydrophobic coatings were prepared by silica nanoparticles embedded in different matrixes such as epoxy [13], polydimethylsiloxane [14], and polysiloxane [15] to increase the mechanical strength by their interconnection. On the other hand, the nanoparticles can be fixed on an interlayer that acts as a binder to connect the particles with the substrate [16]. This strategy is preferable for the exposure of the stacking particles roughness to the coating surface and lead to better hydrophobicity. Extending this scope for durable superhydrophobic coating, double-silica-layered structure was proposed here to reinforce the coating adhesion to glass substrate. In the double-

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silica-layered structure, a compact interlayer beneath a top layer of silica nanoparticles was formed by the hydroxyl condensation of silica chains which fixed the silica particles by their further mutual hydroxyl condensation. Thus, the prepared superhydrophobic coating can reserve the micro/nano-sized roughness of silica particles externally and obtain excellent mechanical robustness due to the strong fixation of the silica particles.

2. Experimental details

2.1. Materials

Ethyl alcohol (EtOH), tetraethoxysilane (TEOS), concentrated hydrochloric acid (HCl, 67%), and ammonium hydroxide (NH₄OH, 25 vol%) were supplied by Tianjin Da Mao Chemical Co., Ltd. (Hep tadecafluoro-1122-tetradecyl)trimethoxysilane (FAS) was purchased from Macklin Chemical Co., Ltd.

2.2. Experiment

Sol A: 20 mL of TEOS was dissolved in 200 mL of EtOH in a beaker, where 2 mL of concentrated HCl as catalyst was then added. After the solution was aged for 24 h, 4 mL of FAS was then dropwise added and stirred for 120 min. The final mixture was aged for another 5 days.

Sol B: 10 mL of TEOS was mixed with 200 mL of EtOH in a beaker. 10 mL of NH₄OH as catalyst was then added to the solution with continuous stirring for 90 min. Then, 2 mL of FAS was dropwise added to the above mixture and stirred for 120 min.

Superhydrophobic coating: Glass slides were cleaned by ultrasonic treatment in deionized water and EtOH, respectively. Sol A was deposited on the glass slides by dip-coating at a withdrawal speed of 80 mm/min (to ensure film thickness and uniformity) [17] and dried at 60 °C. Then, sol B was repeated the above procedure as sol A. Consequently, superhydrophobic coating with double silica layers on glass was obtained.

2.3. Characterization

Fourier transform infrared spectrophotometer (FTIR, Tensor27 Bruker) and scanning electron microscopy (SEM, HITACHI-4800) were used to investigate the chemical modification of silica sols and the morphology of the hydrophobic surface, respectively. The water contact angles were measured at ambient temperature via the sessile-drop method using an optical contact angle meter (Dataphysics OCA 20LHT, water droplet is 5 μ L). Abrasion resistance was determined by loading a weight of 500 g on the coated glass slide (7.5 \times 2.5 cm²) and displacing it against an emery paper abrasion (800 mesh). Sand abrasion tests were performed by different loads of sand impacting on the coating at a speed of about 3.16 m/s. Tape test (CNS 11684), also known as peel test, was carried out to evaluate the adhesion of the coatings on the substrate. The degree of adhesion was defined as the percentage of coating residing on the plate after the peel test. UV resistance test was performed by exposing the coating to a UV lamp (700 W) with a distance of 20 cm.

3. Results and discussion

Non-wetting behavior depends on both low surface energy and properly nano/micro-sized surface roughness. Thus for low surface energy, FAS with fluorine-containing groups is employed to modify the surface of silica aggregates in sol A and B. FTIR spectra were conducted to investigate fluorine-containing groups on the silica surface that can render the prepared silica layers with low surface

energy. Fig. 1a shows the samples of sol A and B with the same signals, indicating similar chemical bonds in the silica aggregates from acid and base catalysis. The vibrations of Si—O—Si are located at the range from 1120 to 1200 cm⁻¹. Besides, it also denotes that the silica was modified by FAS due to the appearance of —CH₃ and —CH₂ stretching vibration (1450 and 1398 cm⁻¹) and the vibration of C—F bonds (1245, 1211, and 1145 cm⁻¹) [16]. Furthermore, aiming at proper surface roughness, it is essential to control the surface morphologies of the prepared coating. Competition between the reaction rates of hydrolysis and dehydration condensation of TEOS can lead to various structures of silica aggregates from linear chains to particles [18]. Deposition of chain- or particle-shaped silica species on glass can cause different surface roughness that in addition influences the final obtained coating hydrophobicity. By acid catalysis for TEOS in this system, the reaction rate of its hydrolysis is lower than that of dehydration condensation so that the silica may grow along one direction and produce highly slim silica chains of molecular scale in sol A (as illustrated in Fig. 1b). Fig. 1c reflects stacking of the slim silica chains on glass that lead to a compact layer with a relatively smooth surface due to their close arrangement. On the contrary, by base catalysis in sol B, the reaction rate of hydrolysis is faster and the silica particles were obtained (Fig. 1b). The particle sizes of silica particles are affected by pH value that is determined by the concentration of ammonium hydroxide [18]. In the open system, NH₄OH may volatilize and lead to decrease of pH value. As a result, the obtained silica particles are heterogeneous and distribute approximately in the range of 40–200 nm, which is mainly attributed to heterogeneous nucleation during the reaction stage of dehydration condensation by altered base catalysis. Deposition of these silica particles on glass by dip-coating can lead to a loosened layer, where the uneven-sized particles nonuniformly stack together and thus exhibit micro/nano-sized surface roughness (Fig. 1d), which is necessary for the superhydrophobicity. Due to the distinct micro/nano-sized surface roughness, the coatings of sol B on glass have greater water contact angle (WCA) of 165.2° (the insets of Fig. 1d) than that of sol A (the insets of Fig. 1c).

Hydroxyls on the glass surface have a possibility to chemically bond with the hydroxyls of the silica species by dehydration, causing the formation of Si—O—Si linking that offer adhesion between coating and substrate. With close arrangement, the compact silica layer of sol A has a larger contact area with the glass substrate than the layer of silica particles does (sol B). Thus, it can be expected that the compact silica layer possess a better adhesion to glass due to more contact area for Si—O—Si linking. However, the compact silica layer does not present a sufficient surface roughness for superhydrophobicity. A double-silica-layered structure was thus proposed to combine the advantages of sol A and B, and improve the robustness for durable superhydrophobic coating. In the structure, micro/nano-sized roughness of FAS-modified silica particles on the top can render the coating with superhydrophobicity, and the compact silica interlayer below can act as a binder and reinforce the adhesion of the top silica particles to the glass substrate by their largely mutual hydroxyl condensation (as shown in Fig. 2a). SEM cross-section view of the coating confirms the double-silica-layered structure, where the silica particles were fixed to the interlayer surface (Fig. 2b). As expected, tape test shows 100% of coating residing on the glass after peeling by the tape (3M™ Scotch-600), revealing an excellent bonding force. Moreover, the digital photo shows that the coating of double-silica-layered structure is superhydrophobic and semi-transparent (Fig. 2c).

To demonstrate the improvement of coating wear resistance by the compact silica interlayer (sol A), the single-layered coating of sol B is selected for comparison with the double silica layers proposed above, both of whom have the same external surface and

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