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# Flame soot stably deposited on silicone coatings possess superhydrophobic surface



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## ABSTRACT

A flame soot deposited silicone coating was successfully prepared by butane flame along with a deposition process of flame soot. Field emission scanning electron microscopy equipped with an energy-dispersive X-ray analyzer and X-ray photoelectron spectroscopy were used to analyze the chemical compositions and structure of flame soot, showing that the surface is mainly composed of carbon nanoparticles being closely packed and forming tree branch-like network with micro- and nanoscale roughness. Meanwhile, the flame soot can stably deposit on the silicone coatings during the water-flow impact test. When the deposition time is 20 s, the silicone coating possesses a water contact angle of  $168 \pm 2^{\circ}$  combining with a sliding angle less than 1°, and a transmittance reduced less than 28% for wavelengths above 500 nm compared to glass substrate. The superhydrophobicity has a little increase with the extension of deposition time, but at the expense of transmittance.

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#### 1. Introduction

Superhydrophobic surfaces with a water contact angle (WCA) above  $150^{\circ}$  and a sliding angle (SA) less than  $10^{\circ}$  have attracted great interest over the past decade in both scientific research and engineering application [1-4]. Based on the studies of naturally superhydrophobic materials, such as lotus leaves, legs of water striders and spider silk, and artificially superhydrophobic materials, it has shown that surface roughness at both micro- and nanoscale combined with low surface-energy material is prerequisite to forming superhydrophobic surfaces [5–12]. Superhydrophobic surfaces have applications in self-cleaning, corrosion-resistant, and anti-adhesive coatings [2]. For engineering application, the superhydrophobicity should be combined with properties such as mechanical resistance, high thermal and chemical stability, durability and processability. And if the superhydrophobic surfaces are used in optic or electronic devices, the good transparency or the special optical and electrical properties should be taken into account as well [13,14]. Transparent superhydrophobic surfaces can be obtained based on silica [15–17], zinc oxide [17] and titanium dioxide [18] nanoparticles, and a transparent superhydrophobic film was also fabricated from stabilized carbon nanotube/silane sol mixture solution [19].

Silicone elastomers based on polydimethylsiloxane (PDMS) are widely used in many fields such as high-voltage outdoor insulation, anti-foul coatings, biomedical applications, encapsulation, seal ring and superhydrophobic materials [20], which are caused by the PDMS's combination properties, for instance, a hydrophobic surface, a wide range of operating temperature, low toxicity, longterm stability, ease of processing and good transparency. PDMS (Sylgard 184) was successfully applied to fabricate a transparent superhydrophobic coating mixed with silica nanoparticles [21].

Most of methods for preparing superhydrophobic surfaces were demanded special or expensive materials, special equipment; the complex process spent a lot of time. Nevertheless, a convenient and cheap way to fabricate superhydrophobic surfaces is provided by exposing the substrates to a flame. During the exposure, flame soot was deposited on the substrates leading to formation of a superhydrophobic surface. Superhydrophobic surfaces showing high water contact angle were achieved by burning candle [2], ethylene-air [22], and rapeseed oil [23]. However, the structure of the flame soot deposited on glass substrate is fragile because the particle–particle interactions are only physical and weak. When water rolls off the flame soot layer, the drop carries soot particles with it until nearly all of the soot deposit is removed and the drop undergoes a wetting transition [2]. Therefore, it is important to develop a new method to avoid this problem.

Herein, we present a novel method to prepare superhydrophobic silicone coatings by exposing it to butane flame. Flame soot deposits on the surface of uncured silicone coating along with a curing process. The surfaces of the as-prepared flame soot deposited





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silicone coatings exhibit tree branch-like heterogeneous structures with micro- and nano-scale roughness having an important influence on the wettability. Within a limited deposition time, a transparent and superhydrophobic flame soot deposited silicone coating can be fabricated. To the best of our knowledge, this is an extremely simple and convenient way to manufacture a superhydrophobic surface.

## 2. Experimental

#### 2.1. Materials

Sylgard-184, an elastomeric polydimethylsiloxane kit manufactured by Dow Corning had two independent components (Sylgard-184A and Sylgard-184B, respectively) and mixed them when used to obtain a silicone coating. The flame was obtained by commercially available lighter with the fuel of butane from Ningbo XINHAI Electric Co., Ltd.

#### 2.2. Preparation of flame soot deposited silicone coatings

The preparation of unexposed silicone coatings and flame soot deposited silicone coatings is schematically presented in Fig. 1. Sylgard-184A and Sylgard-184B were mixed by a mass ratio of 10:1. The mixture was then coated on glass substrates with a dimension of about 25 mm  $\times$  25 mm, forming 30  $\mu$ m thick uncured silicone coatings (Fig. 1a). The uncured silicone coatings on glass substrates were point-blank exposed immediately to the top of flame with a height of 20 mm. During the exposure, flame soot underwent a deposition on the uncured silicone coating surface, and wettability of the surface was changed as a result. Keeping several seconds' deposition for one place and repeated the deposition for other places of the uncured silicone coating surface until the whole surface was almost deposited with flame soot. The flame soot deposited silicone coatings with a total deposition time of 20 s and 40 s were prepared, respectively. The silicone coating was also being cured during the deposition process. The fuel flow rate was controlled by placing the lighter's valve in the middle position to keep the flame with a same height. The unexposed silicone coatings cured at 100 °C for 1 h with no exposure was used as references compared to the flame soot deposited silicone coatings. Meanwhile, the sample of aluminum foil with a 40 s flame soot deposition was also prepared for the study of flame soot's chemical compositions and structure. All samples had the same original dimension of about  $25 \text{ mm} \times 25 \text{ mm}$  and were cut into an appropriate size for characterization.

#### 2.3. Characterization

The water contact angle and sliding angle were measured by a DATA Physics System OCA20 from Germany at room temperature. A 4 µL water droplet was used for the measurement of WCA. The SA values (another important evaluation criterion for superhydrophobic surface) were measured by dropping water droplets  $(10 \,\mu L)$  onto the tilted surfaces from about 4 mm height above the surface, and determined as the tilting angles at which the water droplets rolled off. More than three different positions were measured for each sample. Field emission scanning electron microscope (FE-SEM) (Hitachi S-4800, Japan, operated at 3 kV) equipped with an energy-dispersive X-ray (EDX) analyzer was taken to observe the surfaces' morphology of flame soot deposited silicone coatings and flame soot deposited aluminum foils, and analyze the chemical compositions of the flame soot. X-ray photoelectron spectroscopy (XPS) analysis for the samples was performed in a VG ESCALAB MARK II using the monochromatic Mg Ka X-ray source (hv = 1253.6 eV). The transmittance of superhydrophobic silicone coating was characterized using the Varian Cary 100 Bio UV-vis spectrophotometer. The optical images were taken with a Canon 500D digital camera. A SM-114 (Teclock, Japan) thickness meter was used to measure the thickness of silicone coatings. The stability of flame soot on silicone coatings was studied by water-flow impact test.

# 3. Results and discussion

#### 3.1. Superhydrophobicity of flame soot deposited silicone coatings

Black surface of the 40s flame soot deposited silicone coating induced by the deposition of flame soot on silicone coating clearly showing superhydrophobicity with the behavior of water droplets on it (Fig. 1b). Fig. 2 demonstrates the surfaces' superhydrophobicity of flame soot deposited silicone coating. As shown in Fig. 2b and c, water droplets  $(4 \mu L)$  deposited on the 20 s and 40 s flame soot deposited silicone coating form extremely high WCAs of  $168 \pm 2^{\circ}$ , and  $171 \pm 2^{\circ}$ , respectively. It is impossible to measure the WCA if water droplets were not stayed contact with the dispensing needle because of the remarkable water repellency. In contrast, the unexposed silicone coating (Fig. 2a) shows a much lower WCA of  $118 \pm 2^{\circ}$  compared to the deposited silicone coating. Owning to the extremely low adhesion between the flame soot deposited silicone coatings and water droplets, water droplets could easily roll off at little tilting angles. Water droplets (10 mL) rolled off the 20 s and 40 s flame soot deposited silicone coating surface showing the SAs both less than 1° (Fig. 2d and e). All of this confirmed that



Fig. 1. Schematic diagram of preparation of unexposed silicone coatings and flame soot deposited silicone coatings. (a) Process of coating the uncured silicone on glass substrate. (b) Optical images of water droplets on a 40 s flame soot deposited silicone coating.

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