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Femtosecond laser-chemical hybrid processing for achieving substrate-independent superhydrophobic surfaces

WENG Wei-xuan(翁伟轩)[1], DENG Qin-wen(邓钦文)[1], YANG Peng-yu(杨鹏宇)[1], YIN Kai(银恺)[1, 2, 3]*

1. Hunan Key Laboratory of Nanophotonics and Devices, School of Physics, Central South University, Changsha 410083, China;
2. State Key Laboratory of Precision Manufacturing for Extreme Service Performance, College of Mechanical and Electrical Engineering, Central South University, Changsha 410083, China;
3. State Key Laboratory of Intelligent Manufacturing Equipment and Technology, Huazhong University of Science and Technology, Wuhan 430000, China

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Abstract: Superhydrophobic surfaces have attracted considerable interest due to their various functions and wide applications. Most of the existing methods for preparing superhydrophobic surfaces are only applicable to one or several specific substrate materials, which have the disadvantage of substrate-dependent. Here, an approach for the fabrication of substrate-independent superhydrophobic surfaces based on femtosecond laser-chemical hybrid processing is proposed. Micro/nanostructures are constructed on substrates via femtosecond laser direct writing technology, followed by modification with stearic acid. The laser-treated samples coated with stearic acid (LT*x*-SA, *x* presents different samples) surfaces have excellent superhydrophobic and self-cleaning properties. Moreover, it is worth noting that the LT*x*-SA surfaces remain stable superhydrophobicity after heating substrate from 20 °C to 100 °C, washing substrate 10 times, and exposing substrate to air for 60 days. This work provides an efficient and facile strategy for achieving substrate-independent superhydrophobic surfaces.

Key words: femtosecond laser; stearic acid; substrate-independent; superhydrophobic surface

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1 Introduction

Superhydrophobicity is one of the significant wettability of solid surfaces. Generally, superhydrophobic surfaces have both high roughness and low surface energy [1 − 2]. Water droplets appear spherical and easily roll on superhydrophobic surfaces with a water contact angle (WCA) greater than 150° and a sliding angle

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WENG Wei-xuan and DENG Qin-wen contribute equally to this work.

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Corresponding author: YIN Kai, PhD, Professor; E-mail: kaiyin@csu.edu.cn; ORCID: https://orcid.org/0000-0002-0763-3834

(SA) less than 10° [3 − 5]. The above interesting phenomena endow superhydrophobic surfaces with various excellent application prospects, involving fields such as self-cleaning [6−8], anti-corrosion [9−11], oil/water separation [12 − 15], and droplet manipulation [16 − 17]. In more detail, functional device surfaces are easily contaminated and corroded during operation, the performance of the device will be affected and damaged. With the combination of superhydrophobic surfaces and these functional devices, this problem can be effectively solved. Accumulated water droplets can carry away the dust on superhydrophobic surfaces while rolling, keeping their surface clean [18]. Moreover, the air layer acts like a mask on these surfaces, they realize anti-corrosion property by inhibiting direct contact between the substrate and corrosive substances [19−20]. The direct discharge of untreated oil-water mixtures will cause pipeline blockage and even environmental pollution. Under the action of gravity or magnetic field, superhydrophobic surfaces with superlipophilicity can adsorb oil and remove water from the oil-water mixture, thus achieving the goal of oil-water separation [21−23]. Meanwhile, these surfaces can also remove oily pollutants from water.

In recent years, superhydrophobicity has attracted extensive attention, many studies have proposed various methods for preparing superhydrophobic surfaces and conducted to fabricate them successfully. For example, MAYOUSSI et al [24] presented a facial way via 3D printing to fabricate thin superhydrophobic membranes, which have adjustable porosity. The obtained superhydrophobic membranes could be used in fields such as oil/water separation and salvinia layers exploration. ZHANG et al [25] developed a superhydrophobic ZnO/Cu-ZnMOFs@SA composite coating on Zn surface, through one-step hydrothermal method, exhibiting good antibacterial and anticorrosion performance. GHASEMLOU et al [26] fabricated lotus-inspired superhydrophobic surfaces by soft-imprinting lithography and spin-coating approaches. However, these mentioned preparation superhydrophobic surface methods suffer from complicated, time-consuming, or substrate-dependent procedures. Hence, it is of great significance to explore a facial strategy to fabricate substrate-independent superhydrophobic surfaces with excellent stability.

Herein, we proposed femtosecond laser and chemical hybrid processing technology for achieving substrate-independent superhydrophobic surfaces. Femtosecond laser is used to construct micro/nanostructures on the substrate surface, increasing its roughness. Subsequently, stearic acid is coated on the laser-treated substrate via heating and curing in drying oven. Stearic acid in this process can effectively reduce the surface energy of the substrate, resulting in hydrophobicity improvement. The above preparation method is applicable to various substrates such as ceramic, titanium (Ti), silicon (Si), and quartz glass. The obtained laser-treated samples coated with stearic acid (LT*x*-SA, *x* presents different samples) all exhibit outstanding superhydrophobicity. Furthermore, after a series of tests, including heating or washing substrate, and long-term exposure of substrate to air, the superhydrophobicity of LT*x*-SA has not degraded, revealing good stability.

2 Experimental

2.1 Materials

The ceramic with an average thickness of 1 mm was purchased from Jingwei Special Ceramics Co., Ltd. (Jiangsu, China). Ti, Si, and quartz glass were bought from local markets. Stearic acid was obtained from Xilong Science Co., Ltd. (Guangdong, China).

2.2 Fabrication of superhydrophobic surface

The method proposed in this work for preparing superhydrophobic surfaces is applicable to various substrates. Here, ceramics were selected as the main research substrate to introduce the detailed preparation process. The first step is femtosecond laser processing. The laser beam (wavelength of 1035 nm, pulse width of 350 fs) was generated by a commercial femtosecond fiber laser system (HR-Femto-IR-50-40B, Huaray, China). Through a two-mirror galvanometric scanner system (basiCube 10, Scanlab, Germany) with an F-Theta lens (focused length of 125 mm), the laser beam was concentrated on the ceramic surface, which scanned along the *X*-direction first and then the *Y*-direction. The laser power, scanning speed, and scanning interval were set at 3.64 W, 50 mm/s, and 15 μm, respectively. Next, the stearic acid was placed on drying oven (101-1BS, Super Instrument, China) at 150 °C for 15 s. Finally, the laser-treated sample coated with a layer of melted stearic acid was vertically placed on drying oven at 240 °C for 20 min, and the LTC-SA surface was obtained.

2.3 Instruments and characterization

The different microstructures of various samples surfaces were characterized by using a field emission scanning electron microscope (SEM, MIRA3 LMU, Tescan, Czech Republic). The elemental compositions and maps were taken by an energy dispersive X-ray spectroscopy (EDS, Tescan, Czech Republic). The 3D morphology and cross-sectional profiles were investigated by a laser confocal microscope (LCM; Axio LSM700, Zeiss, Germany). WCA was measured by a contact angle measurement system (SDC-200S, Shengding Precision, China).

3 Results and discussion

3.1 Surface fabrication

The substrate-independent superhydrophobic surfaces were simply prepared by femtosecond laser-chemical hybrid process. As schematically illustrated in Figure 1(a), femtosecond laser direct writing technology was chosen as the first step to ablate substrates because of its high precision, simple fabrication process, and high efficiency, which is suitable to produce micro/nanostructures on different substrates [27−38]. Then, a drying oven was employed to melt stearic acid and adhere it to laser-treated substrates. After keeping samples in the

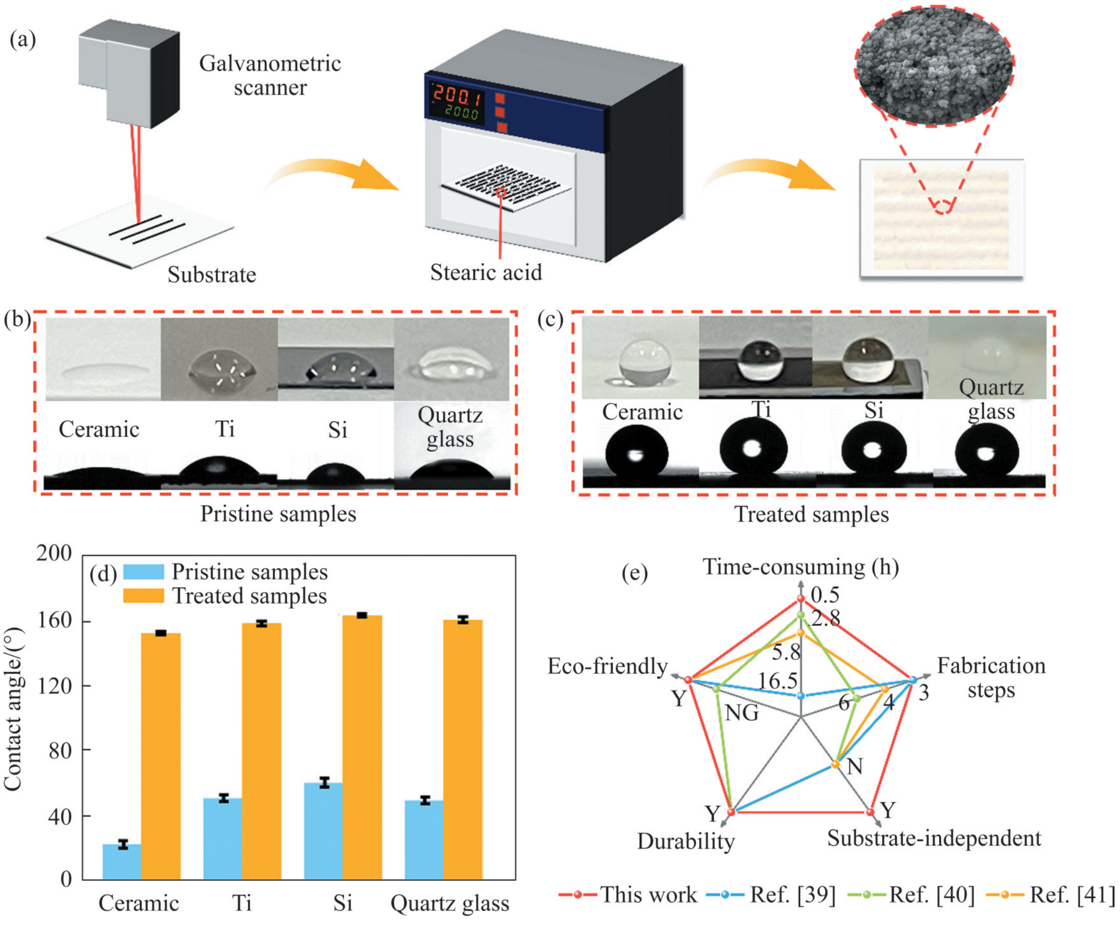

Figure 1 Fabrication of substrate-independent superhydrophobic surfaces: (a) Schematic diagram for fabrication of substrate-independent superhydrophobic surfaces; (b) Photos of the water droplets placed on the Pristine ceramic, Ti, Si, and quartz glass, respectively; (c) Optical photos and static contact angles of the water droplets placed on the LTC-SA, LTT-SA, LTS-SA, and LTQ-SA, respectively; (d) Comparison of WCAs on pristine and treated samples surfaces; (e) Comparison between our proposed method and previously reported other preparation methods for superhydrophobic surfaces

drying oven at 240 ℃ for 20 min, the LT*x*-SA surfaces were achieved successfully. In this work, four kinds of materials including ceramic, Ti, Si and quartz glass were selected as substrates to fabricate superhydrophobic surfaces. Pristine ceramic, Ti, Si, and quartz glass all exhibited hydrophilicity (Figure 1(b)). Interestingly, the wettability of these four substrate materials has been transformed from hydrophilicity to superhydrophobicity through femtosecond laser processing and stearic acid modification. It was obviously seen that the water droplets were almost spherical shape on the LTC-SA, LTT-SA, LTS-SA and LTG-SA surfaces (Figure 1(c)). The WCAs of four pristine samples were ~22.3°, ~50.9°, ~60.4° and ~49.5°, respectively. By comparison, the WCAs of four treated samples were ~152.5°, ~158.6°, ~163.5° and ~161.0°, respectively. We further compared the proposed process with other previously reported work about superhydrophobic surfaces preparation methods [39−41]. Our proposed fabrication process was brief and efficient. More importantly, the preparation method had superiority substrate-independent aspects (Figure 1(e)).

3.2 Characterization of surface morphology

Morphology and chemical composition of solid surfaces are the decisive factors for their wettability [42−45]. In order to explore the superhydrophobic mechanism of the four treated samples, we employed the scanning electron microscope (SEM) to observe micro/nanostructures on the four treated samples. Compared to the four smooth and flat pristine samples, numerous porous micro/nanostructures were uniformly distributed on corresponding treated samples, resulting in coarser surfaces. However, from high magnification SEM images, it was clearly found that there were some differences in the micromorphology of these treated samples. Specifically, the LTC-SA surface was coverd with dense hierarchical coral-shape micro/nanostructures (Figure 2(a)). The morphology of LTT-SA and LTS-SA surfaces was composed of tightly packed particles with a particle size of 6.25 and 12.5 μm (Figures 2(d), 2(g)). There were various nanowires and nanocavities evenly coated on the LTQ-SA surface (Figure 2(j)). Furthermore, the four treated samples were also analyzed using energy dispersive X-ray spectroscopy (EDS) to verify the composition of chemical elements. Carbon (C) and oxygen (O) elements were homogeneously distributed on the surfaces of the four treated samples, indicating the existence of stearic acid (Figures 2(b), (e), (h) and (k)).

The contents of aluminum (Al), C, O elements on the LTC-SA surface were 28.72%, 9.72%, 61.56 %, respectively (Figure 2(c)). The content of Ti, C, O elements on the LTT-SA surface were 41.68%, 4.48%, 53.84%, respectively (Figure 2(f)). Both LTS-SA and LTQ-SA surfaces contained silicon (Si) element with the content of 42.02% and 24.31% (Figures 2(i) and (l)). The above characterization results validated that femtosecond laser processing and stearic acid modification could synergistically improve surface roughness and reduce surface energy to enhance surface hydrophobicity effectively.

The laser confocal microscope (LCM) was further carried out to characterize the 3D topography and cross-sectional micro profile of the LTC-SA, LTT-SA, LTS-SA, and LTQ-SA surfaces. As shown in Figures 3(a)−(d), the four treated sample surfaces were all very rough. Consistent with the results of SEM images, it was apparently observed that the hierarchical micro/nanostructures were regularly covered on these surfaces. The depth of their hierarchical micro/nanostructures were 5−15 μm. During the process of femtosecond laser-chemical hybrid treatment, the hierarchical micro/nanostructures contributed to increase the contact area between melted stearic acid and substrates, promoting the decrease of surface energy. As a result, the superhydrophobicity of surfaces would be further enhanced.

3.3 Characterization of surface wettability

To confirm prepared surfaces with superhydrophobicity, we selected LTC-SA surface as representative to conduct various wettability tests on it. For example, a water droplet was suspended from a microsyringe and slowly moved downwards. When the droplet contacted with the LTC-SA surface, it was slightly deformed. Subsequently, the droplet left the surface as the microsyringe rising, it restored initial shape (Figure 4(a)). Meanwhile, a water droplet can readily slide down the LTC-SA

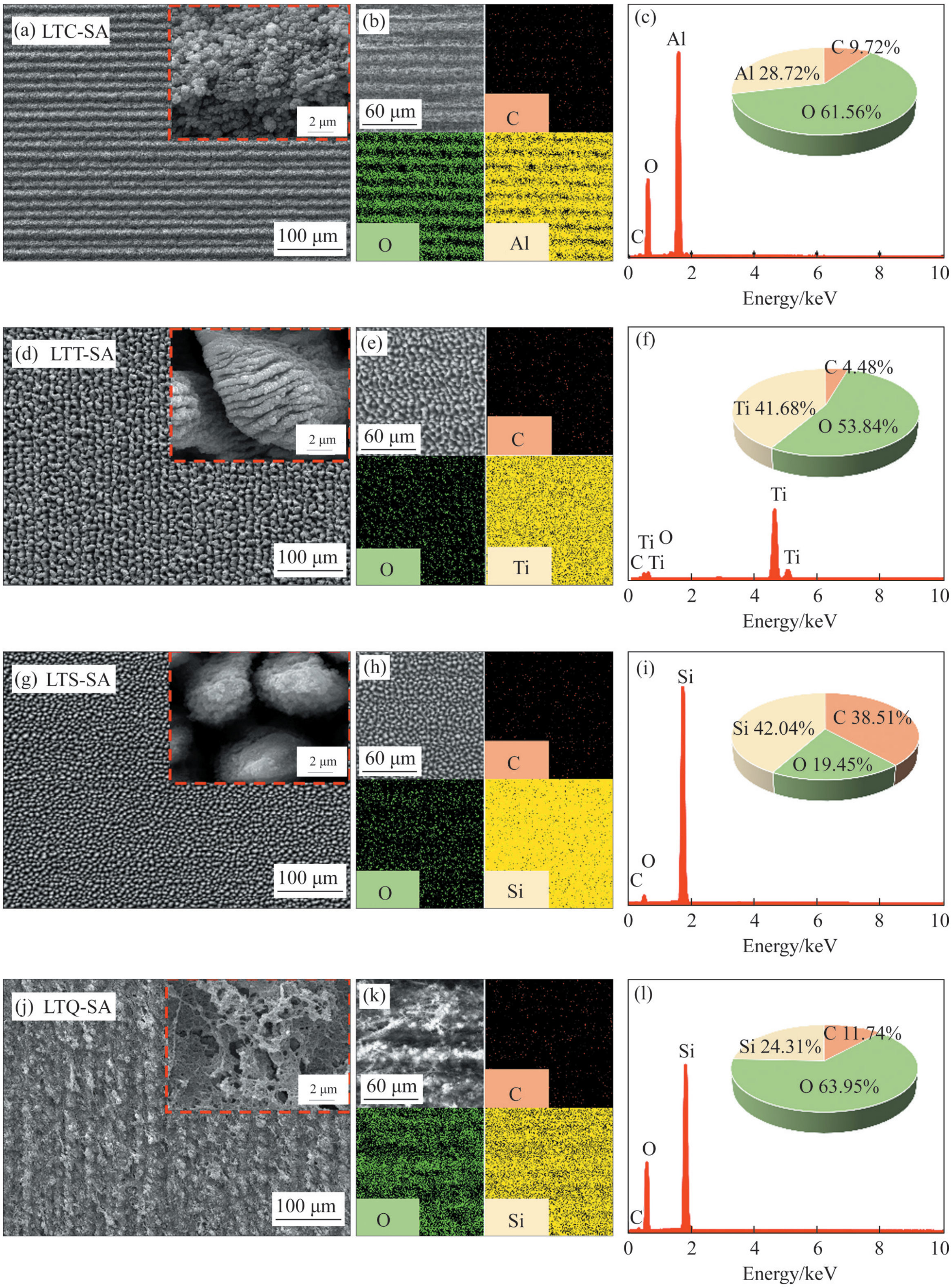

Figure 2 SEM and EDS characterization: SEM images of (a) LTC-SA, (d) LTT-SA, (g) LTS-SA, and (j) LTQ-SA, respectively. EDS and elemental mappings of (b, c) LTC-SA, (e, f) LTT-SA, (h, i) LTS-SA, and (k, l) LTQ-SA, respectively

surface with a sliding angle of 3° (Figure 4(b)). A high-speed camera was employed to capture the dynamic process of a water droplet impacting on the LTC-SA surface. As shown in Figure 4(c), the droplet experienced four stages of falling, spreading, retracting, and rebounding within 70 ms.

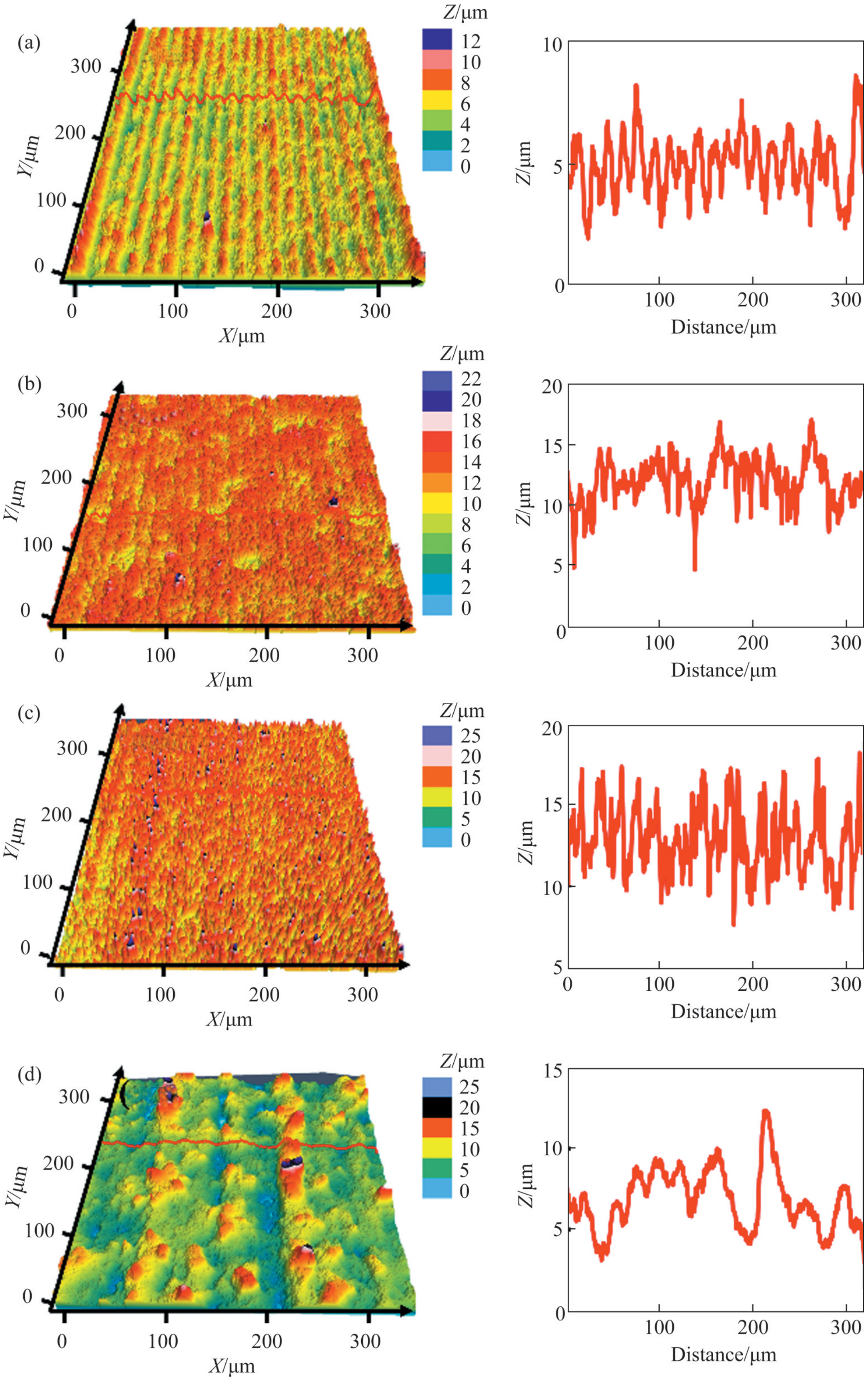

Figure 3 LCM characterization: 3D topography and cross-sectional micro profile of (a) LTC-SA, (b) LTT-SA, (c) LTS-SA, and (d) LTQ-SA, respectively

The above tests all demonstrated that the LTC-SA surface has excellent superhydrophobicity. We further used chalk powder to simulate dust and sprinkled it on the LTC-SA surface. It was clearly observed that the dust can be easily carried away by water droplets, maintaining the surface clean (Figure 4(d)). The result displayed outstanding self-cleaning performance of the LTC- SA surface.

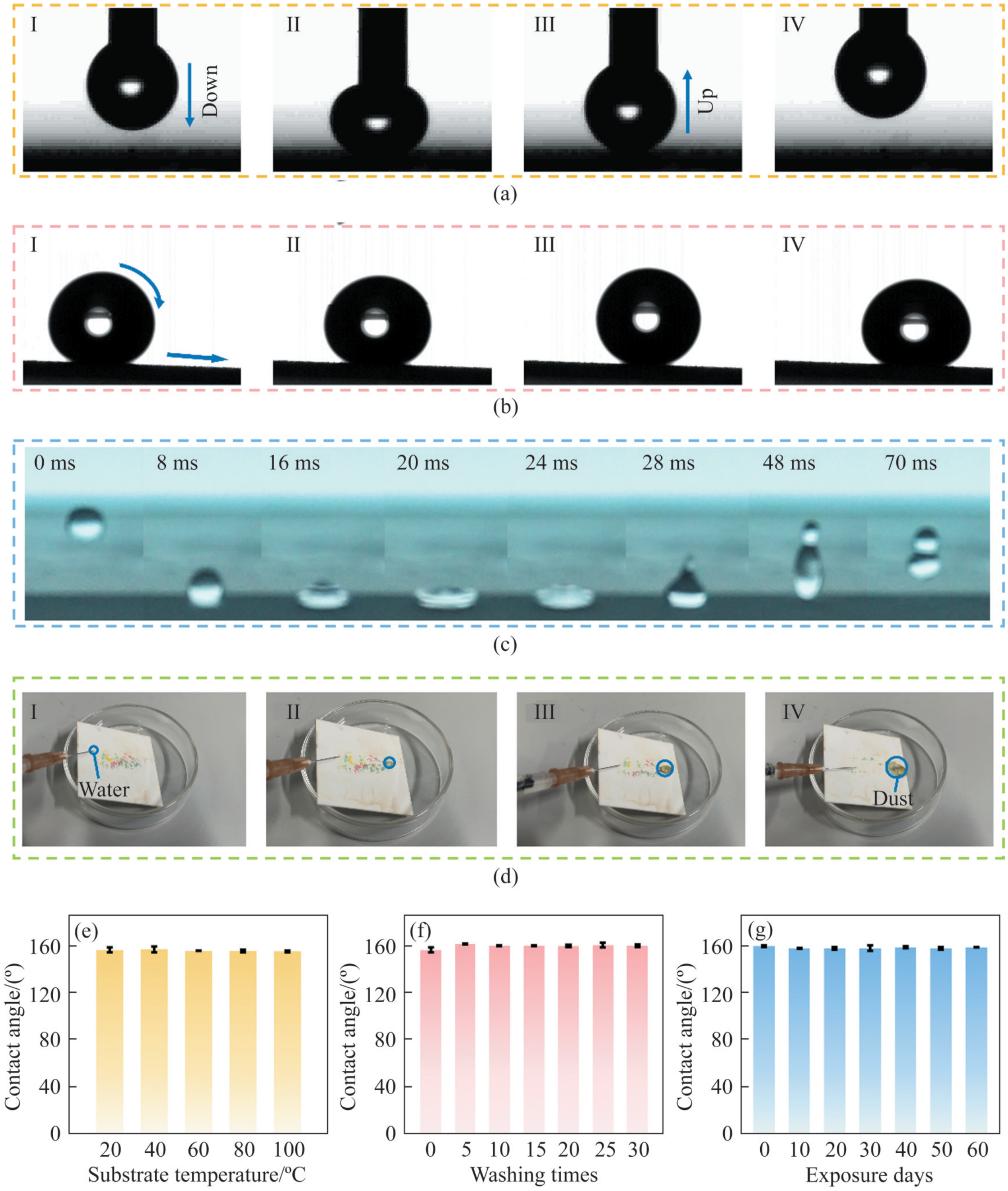

Figure 4 Wettability and stability characterization: (a) Dynamical adhesive behaviors of a water droplet contacting and leaving the LTC-SA surface; (b) Pictures of a water droplet sliding (~3°) on the LTC-SA surface; (c) Sequential photographs of water impact on the LTC-SA surface; (d) Self-cleaning effect of the LTC-SA surface; (e) Heating stability test of the LTC-SA surface; (f) Washing stability test of the LTC-SA surface; (g) Long-term air exposure test of the LTC-SA surface

The stability of superhydrophobic surface is crucial for its practical applications [46 − 47]. Therefore, a range of experiments including heating stability, washing stability, and long-term air exposure tests were conducted on the LTC-SA surface to evaluate its durability. The LTC-SA surface was placed on a hot stage with temperature increasing from 20 ℃ to 100 ℃. WCAs have basically not changed, which were still greater than 150°, indicating good heat resistance performance of LTC-SA surface (Figure 4(e)). After washing the LTC-SA surface 10 times by water, there was no change in superhydrophobicity of the surface (Figure 4(f)). Results showed that LTC-SA surface has positive wash resistance function. Moreover, the LTC-SA surface was expoed to air for 60 days, WCA values of water droplet on the surface substantially presented consistent every day, verifying its air stability (Figure 4(g)). Above all, the superhydrophobicity and stability of the LTC-SA surface is outstanding.

4 Conclusions

In summary, fabrications of superhydrophobic surfaces on various substrates are achieved by hybrid femtosecond laser direct writing technology and stearic acid treatment. Femtosecond laser processing produces numerous hierarchical micro/nanostructures on the substrate that enhance its roughness. The laser-treated substrate is further modificated with stearic acid to reduce surface energy. The synergistic effect of these two preparation processes endows the substrate surface with excellent superhydrophobicity. Furthermore, the as-prepared superhydrophobic surfaces have remarkable stability. Through heating or washing superhydrophobic surface, and exposing superhydrophobic surface to air, there is no change in wettability of the superhydrophobic surface essentially. We expect that our work may provide some insights into designing superhydrophobic surface on various substrates and further promoting the development of superhydrophobic surface preparation field.

Contributors

YIN Kai provided the concept and discussed the results. WENG Wei-xuan and DENG Qin-wen designed and conducted the experiment, edited the manuscript. YANG Peng-yu designed the experiment, discussed the results.

Conflict of interest

WENG Wei-xuan, DENG Qin-wen, YANG Peng-yu, and YIN Kai declare that they have no conflict of interest.

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中文导读

飞秒激光-化学混合制备多基底超疏水表面

摘要：超疏水表面因具有多种功能和广泛的应用引起了人们极大的兴趣。目前制备超疏水表面的方法大多只适用于一种或几种特定的基底材料，具有依赖基底的缺点。本文提出了一种基于飞秒激光-化学混合加工制备与基底无关的超疏水表面的方法。通过飞秒激光直接写入技术在基底上构建出微/纳米结构，然后用硬脂酸改性。硬脂酸涂覆激光处理后的样品(LT*x*-SA，*x*表示不同的样品)表面具有优异的超疏水和自清洁性能。此外，值得注意的是，将基底从20 ℃加热到100 ℃，或清洗基板10次，或将基板暴露在空气中60 d后，LT*x*-SA表面仍然保持稳定的超疏水特性。这项工作为制备与基底无关的超疏水表面提供了一种有效而简单的策略。

关键词：飞秒激光；硬脂酸；多基底；超疏水表面