



# Fabrication of metallic surfaces with long-term superhydrophilic property using one-stop laser method



Y.C. Guan<sup>a,b,\*</sup>, F.F. Luo<sup>c</sup>, G.C. Lim<sup>b</sup>, M.H. Hong<sup>c</sup>, H.Y. Zheng<sup>b</sup>, Bojin Qi<sup>a</sup>

<sup>a</sup> Beihang University, 37 Xueyuan Road, Beijing 100191, PR China

<sup>b</sup> Singapore Institute of Manufacturing Technology, 71 Nanyang Drive, Singapore 638075, Singapore

<sup>c</sup> National University of Singapore, Singapore 117576, Singapore

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## ABSTRACT

A simple method for fabricating stable superhydrophilic surface at metallic substrates is reported. This technique comprises irradiating the surface with multiple laser pulses. Surface wettability can be tailored through controlling laser parameters and processing conditions. The substrates were selected as aluminum alloy and stainless steel. Physical morphology and chemical composition of laser-textured surfaces were characterized by SEM, XPS, and 3D profiler measurements. Results showed that the longest wettability duration was achieved as more than 1 month for stainless steel and more than 200 h for Al alloy, respectively. The possible mechanism of hydrophilic behavior of laser-textured surfaces was discussed. The effect of surface topography on superhydrophilicity property was also evaluated. This study presents a promising method in fabricating long-term superhydrophilic surfaces, which is useful for improving adhesion or achieving water-assisted flow in industrial applications as well as developing cell-based technologies in biomedical applications.

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## 1. Introduction

Recently, extensive research has been conducted to realize metallic materials with good adhesion surfaces by considering significant applications for both scientific findings and practical industries. The significance of good adhesion surface for the metallic materials, especially for widely used stainless steel, titanium alloy, aluminum alloy, mainly lies in the strong bonding interface between the substrates and other materials, such as cell-based biosensors, cell–cell communication, coating fabrication, and water-assisted flow generation [1–3]. On basis of previous work, many methods including plasma treatment, UV irradiation, layer-by-layer assembly, ion exchange, chemical assisted techniques, electrochemical anodization, electroless deposition, and polymer-based coating have been successfully applied to various metallic surfaces to achieve complex surface functions for effective wettability enhancement [4–9].

Although widely implemented in research, these methods have fundamental limitations for practical use. Typical disadvantages are summarized as following [2,3,5–7,9]: (a) the need for multistep and slow procedures for implementation; (b) the need to expose

metallic substrate to liquid organic solvents and/or inorganic solutions; (c) limitations in materials for example UV light cannot be applied to stainless steel and polymer coating are unsuitable for high temperature applications; (d) limitations in substrate size and non-curved surfaces. Moreover, the stability of the hydrophilic property has attracted attention and has increased in importance. For example, the hydrophilicity of surface treated with plasma degrades in air very quickly, and the average valid time for plasma treated surface is estimated to be less than 2 h [1,2,9]. It can be explained by the rotation of chemical hydrophilic groups toward the substrate with aging according to careful analysis [9]. In addition, plasma treatment usually requires expensive equipment. Therefore, developing a simple, cheap and green method that overcomes these limitations for obtaining environmentally stable superhydrophilic surface with large-sized areas is still a problem.

In this work, we present a one-stop method for fabricating long-term superhydrophilic surfaces at metallic substrates by short pulse laser. Superhydrophilicity denotes a phenomenon where water drop is quickly sucked into the surface structure, ultimately spreading to a contact angle at or close to 0° [10–12]. We demonstrate this method to achieve the superhydrophilic surfaces on both stainless steel and aluminum alloy, and discuss how laser processing affects the textured-surfaces. We conclude by explaining the mechanism of long-term superhydrophilic surfaces, and indicate how it applies for potential applications.

\* Corresponding author at: Beihang University, 37 Xueyuan Road, Beijing 100191, PR China.

E-mail address: [guan0013@e.ntu.edu.sg](mailto:guan0013@e.ntu.edu.sg) (Y.C. Guan).

## 2. Materials and methods

### 2.1. Preparation of samples

The materials studied were commercial Al alloy 6061 and 316L stainless steel. The dimension of aluminum alloy was 30 mm by 20 mm by 3 mm, while the dimension of stainless steel is 30 mm by 20 mm by 1 mm. All specimens were ground with progressively finer SiC paper (180, 400, 800, 1200, 2400 and 4000 grit) to minimize the effect of incidence angle, and cleaned with alcohol to reduce variation of laser beam absorption by the contaminants.

### 2.2. Laser texturing

A nanosecond pulsed Nd:YAG laser (wavelength 1064 nm, pulse duration 10 ns, repetition rate 100000 Hz, spot size 20  $\mu\text{m}$ ) was used in this study. The optimized average power density was  $1.53 \times 10^6 \text{ W/cm}^2$ , and the optimized scanning speed was 70 mm/s. The irradiated area was  $10 \times 10 \text{ mm}^2$  in square using hatched scanning mode with 50% overlapping in the program. When laser was turned on, the specimen was placed in a well-sealed chamber under different environment as vacuum, nitrogen, oxygen, argon and air, respectively, as shown in Fig. 1. All samples were stored in open air both before laser processing and after laser processing.

### 2.3. Measurement and characterization

Contact angle measurement was carried out in atmospheric condition at room temperature by sessile drop method with VCA Optima (VCA-2500XE AST products, Inc.). The contact angle is defined as the angle between the solid surface and a tangent, drawn on the surface of liquid droplet, passing through the solid–liquid–vapor triple-point. 1  $\mu\text{l}$  deionized water droplets was used. The VCA Optima utilizes a precision camera and advanced PC technology to capture the image of the droplet and determine a tangent line for the WCA measurement. The axisymmetric-drop-shape analysis profile method was used for estimating the contact angle of water on the solid surface.

XPS analysis was carried out for the surface chemistry using Thermo Scientific Theta Probe XPS. Monochromatic Al K $\alpha$  X-ray ( $h\nu = 1486.6 \text{ eV}$ ) was employed for analysis at an incident angle of  $30^\circ$  with respect to the surface normal. Photoelectrons are collected at a take-off angle of  $50^\circ$  with respect to the surface normal. The analyzed area is approximately 400  $\mu\text{m}$  in diameter. Survey spectra and high-resolution spectra were acquired for surface composition analysis and chemical state identification, respectively.

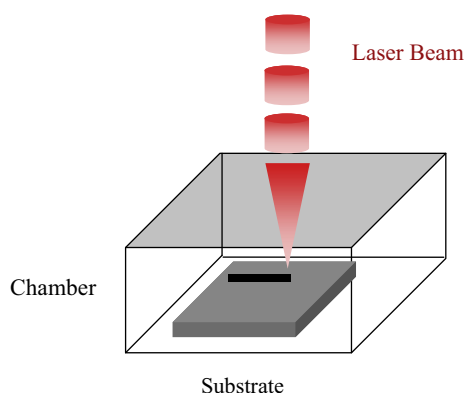


Fig. 1. Schematic outline of laser method used to produce superhydrophilic metallic surfaces.

Surface morphology was observed by Carl Zeiss EVO 50 scanning electron microscopy (SEM, LaB6 filament). Surface topography of the irradiated areas was carried out using Alicona optical three dimension non-contact metrology system (Infinite Focus). The optical three dimension non-contact metrology system is used to image profile and measure roughness of the textured surfaces.

## 3. Results and discussion

### 3.1. WCA measurement

We performed static contact angle measurements by placing water droplet on the surface including both as-received surfaces and laser-textured areas to study the effect of laser texturing on surface wetting, as shown in Fig. 2. For laser-textured surfaces for both Al alloy and stainless steel, we observed complete wetting, i.e., the water droplet brought into contact with the solid spontaneously formed a film with a water contact angle at or close to  $0^\circ$ . The laser-textured surfaces therefore display extreme superhydrophilicity. Table 1 further summarizes shows the evolution of WCA with increasing store time, showing that the superhydrophilicity occurred for all fresh textured surfaces. According to Young–Dupre equation for work of adhesion as  $W_{ad} = \gamma_{lv}(1 + \cos \theta)$ , where  $\gamma_{lv}$  is the liquid–vapor interfacial energy, and  $\theta$  is the contact angle [13]. Therefore, the smaller the contact angle is, the higher adhesion energy of water to the substrate is. However, self-drophobic recovery took place at some samples, especially for these proceeded in Ar environment during laser irradiation; the WCA was observed to change from hydrophilic to hydrophobic range.

### 3.2. XPS analysis

XPS was used to characterize the surface composition of both fresh laser-textured surfaces and laser-textured surfaces after 1 week. The typical XPS survey spectrum in vacuum condition for all elements at both fresh laser-textured surfaces and laser-textured surfaces after 1 week of Al alloy and stainless steel is shown in Fig. 3. It can be found that there is no much change for both surfaces with different store time in the spectrum, and the reason is probably due to zoom in effect for qualitative analysis of individual element in particular bonding energy range. Since XPS is a popular and powerful technique for the investigation of surface composition, it does provide qualitative and quantitative information on the chemical changes. Therefore, chemical compounds including metallic oxides as Al–O bond, Al–OH bond,  $\text{Fe}^{2+}$ –O bond,  $\text{Fe}^{3+}$ –O/ $\text{Fe}^{3+}$ –OH bond, and polar functional groups as hydroxides, C=O bond was chosen for quantitative analysis of main elements as C and O due to water solubility [12,14–17]. It is known that polar functional groups improve the wetting behavior significantly because –OH from water molecules is polar. Moreover, the water solubility of metallic oxides also affects the wetting behavior [12,14,15]. Fig. 4 shows the respective atomic percentage of the above bonds of laser-textured surfaces. Compared to samples proceeded under Ar condition, both samples proceeded under vacuum have longer duration of wetting, which is probably due to the higher content of polar functional groups as hydroxides and C=O bond at the surfaces (20.16 at.% vs 3 at.% and 14.46 at.% vs 4.73 at.% for stainless steel, while 46.08 at.% vs 40.16 at.% and 48.27 at.% vs 39 at.% for Al alloy). It is known that surface energy is determined by intermolecular forces and it comprises of polar interaction and dispersive component. Thereby, the work of adhesion  $W_{ad}$  can be also expressed as the sum of different intermolecular forces

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