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Fabrication of hydrophobic Ti_3SiC_2 surface with micro-grooved structures by wire electrical discharge machining

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ABSTRACT

The near-superhydrophobic Ti_3SiC_2 surfaces with regular and controllable micro-grooved structures were fabricated by wire electrical discharge machining (WEDM). The surface topographies and chemical compositions of smooth and micro-grooved Ti_3SiC_2 surfaces were characterized. The micro removal mechanism of Ti_3SiC_2 in the process of electrical discharge machining was also analyzed. The wetting mechanism of micro-grooved Ti_3SiC_2 surface was discussed along with the static contact angle, anisotropic wettability and contact angle evolution versus time. The relationships between parallel and perpendicular contact angles, depth-width ratio of micro-grooved structures and surface roughness of textured surface were investigated. The experimental results show that the parallel contact angle on the textured Ti_3SiC_2 surface increased by about 164% compared with the one on the smooth surface, and near-superhydrophobic surface with obvious anisotropy was roughly achieved. The experimental parallel contact angles were very close to theoretical contact angles calculated by Cassie-Baxter formula. It is confirmed that the depth-width ratio may be used to predict the parallel contact angle with the average prediction error of 2.4%. The perpendicular contact angles had a good correlation with the depth-width ratio and surface roughness.

1. Introduction

Ti_3SiC_2, a typical MAX phase, which is comprised of a C-Ti_6 octahedron and a close packed Si layer, resulting in nano-layered and hexagonal symmetry crystal structure. Owing to the combination of Ti-C covalent bond with weak bonds between Si and C-Ti_6, Ti_3SiC_2 exhibits both metal and ceramic properties, including light weight, good electrical and thermal conductivity, thermal stability, thermal shock resistance and self-lubrication [1,2]. Based on these remarkable properties, it has many potential applications in the fields of turbine blade [3], dumping material [4] and biomedical material [5], etc.

In recent years, the research on the application of Ti_3SiC_2 in biomedical material had been reported [5]. It had been confirmed that Ti_3SiC_2 was a promising implant material due to its biological non-toxicity and stability in living body. However, Ti_3SiC_2 had a corrosion after a long-term immersion in the Hank's fluid, limiting the applications in the implants contacted with body fluid. It had been reported that the hydrophobic surface with micro- or nano-structures exhibited better corrosion resistance than hydrophilic surface [6]. This is because the textured surface could well control the fluid contact by entrapping air inside the textured structure to reduce the fractional area of liquid-solid interface, resulting in difficulty for the penetration of corrosive species to the surface.

To figure out the relationships between contact angles and surface structure and topographies, two classical theories, namely, the Wenzel and Cassie-Baxter models, were proposed to describe the wetting state on the textured surface [7,8]. The Wenzel model described a non-composite wetting state, which the droplet could completely fill the grooves and wet the liquid-solid interface. The Cassie-Baxter (CB) model described a composite wetting state, which the droplet could hardly penetrate the grooves and form a composite interface of liquid-solid and liquid-gas contact.

Textured surfaces with micro-structures can be fabricated by many advanced processing techniques, including etching [9], micro-grinding [10], laser processing [11], and wire electrical discharge machining (WEDM) [12], etc. The etching machining technology may fabricate petal-like microstructure on the surface of stainless steel by modifying a fluoride film, but the mechanical stability of film was untested [9].

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Moreover, the equipment of etching machining was very expensive and its process was also considerable complicated. Although smooth and precise micro-grooved surfaces with depth of 25–80 μm were obtained by micro-grinding technology [10], the processing efficiency was very low due to repeating dressing and truing of diamond grinding wheel micro tip. It had been confirmed that the efficient and eco-environmental laser machining can evidently improve the hydrophobicity of textured Ti_3SiC_2 surface with a maximum contact angle of 83.6° [11]. However, it is very difficult to ensure the form accuracy and surface quality of micro-structures.

Compared with the processing techniques mentioned above, especially conductive materials, wire electrical discharge machining (WEDM) owned higher machining efficiency and could fabricate more regular and complicated structures with micro-nano sizes. The co-existed micro-nano structure may great improve the hydrophobicity of textured surface. For instance, Yu, et al. [13] had fabricated textured Al alloy surfaces with submillimeter-scale structures by high-speed wire electrical discharge machining (HS-WEDM), resulting in the super-hydrophobic surface with a water contact angle of 154.7°. At present, the HS-WEDM processing was usually employed to machine grooves with hundreds of micrometers in width. Compared with efficient HS-WEDM, low-speed wire electrical discharge machining (LS-WEDM) had higher machining accuracy and more dimensional scale. In addition, the micro-grooves with the size less than 100 μm can be only fabricated by LS-WEDM.

In order to investigate the influence of micro-grooved structure with the sizes less than 100 μm on the wetting behavior of Ti_3SiC_2 surface, efficient and precise LS-WEDM was employed to fabricate regular and controllable micro-grooved structures with different groove parameters in this paper. The wetting behaviors of water droplet on the surface with micro-grooved structures were investigated to reveal the wetting mechanism and anisotropic wettability. The chemical compositions were characterized to analyze influences of groove parameters on wetting state of textured surface. The relationships between contact angles and groove parameters were analyzed to reveal the variation of contact angles.

2. Experimental details

2.1. Wire electrical discharge machining (WEDM) method

As shown in Fig. 1, a commercial WEDM system (Sodick, AP250LS) was used to fabricate micro-grooved Ti_3SiC_2 surface. Commercially available Ti_3SiC_2 bulk was firstly cut in 15 × 15 × 5 mm bar and fixed on the vertical worktable. The brass wire with the diameter of 50 μm was loaded as an anodic electrode and constantly controlled by wire electrode feeding system to minimize the electrode wear. The workpiece was immersed into the dielectric oil (Glysantin G 48–24) during the machining process. Under the action of micro electrical discharge removal, the micro-grooved structures were machined by the wire electrode along the setting V-shape excision path. All the experiments were implemented under same electrical discharge parameters. The discharge voltage and inter-electrode gap were set as 70 V and 0.034 mm, respectively. Afterwards, these machined samples were cleaned ultrasonically by acetone and alcohol for three times.

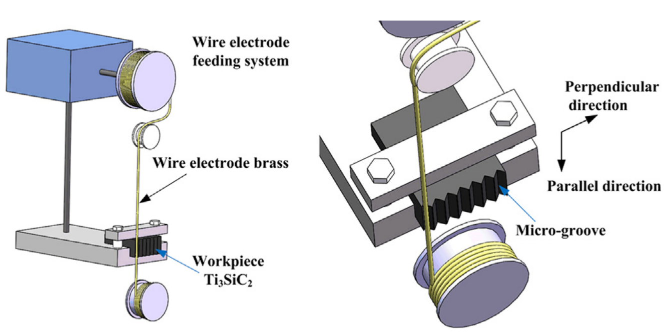

Fig. 1. The processing schematic diagram of LS-WEDM.

Table 1
The surface roughness R_a and groove parameters of textured surfaces.

Sample no.	Width b (μm)	Spacing a (μm)	depth h (μm)	Depth-width ratio λ (μm)	Surface roughness R_a (μm)
A	47.1	13.3	15.1	0.32	1.21
B	49.3	13.7	16.7	0.34	1.14
C	47.9	12.1	17.9	0.37	1.01
D	63.6	12.9	27.4	0.43	1.52
E	63.2	15.2	33.8	0.53	1.65
F	96.5	22.8	45.4	0.47	1.49

2.2. Characterization techniques

The phase constitutions of Ti_3SiC_2 surfaces before and after WEDM processing were analyzed by energy dispersive X-ray spectroscopy (EDS, INCAP FET-X3), X-ray diffraction (XRD, MiniFlex600, Rigaku), Raman spectrum (HR Evolution, Horiba) and X-ray photoelectron spectroscopy (XPS, Axis Ultra DLD, Kratos). The surface topographies were characterized by scanning electron microscope (SEM, Quanta FEG450, FEI) and 3D laser scanning confocal microscope (VK-250, Keyence). According to the measured 3D topography and profile, the groove width b, groove depth h and surface roughness R_a was obtained as shown in Table 1. The static and dynamic contact angles were measured by sessile droplet method using contact-angle measuring instrument (Theta, Biolin). The water droplet with a size of 4 μl was deposited on the sample at the constant temperature of 26 °C. The contact angle observed along parallel and perpendicular directions of micro-grooved were defined as the parallel and perpendicular contact angle, respectively (see Fig. 1).

3. Results and discussions

3.1. Topographies and profile curves of the textured surfaces

Fig. 2 shows the section profile curves of WEDM textured surfaces. Micro-scale grooves with groove width b, spacing a and depth h were successfully fabricated on the Ti_3SiC_2 surface. The WEDM textured surfaces with different pattern parameters were labeled and listed in Table 1. Sample E had the highest depth-width ratio λ (0.52) and surface roughness R_a (1.65 μm), while Sample F had the maximum groove width b (94.6 μm), spacing a (25.4 μm) and depth h (45.4 μm). From the section profile curves of textured surfaces, it can be observed that there were many secondary nano-structured grooves distributed on the surface of micro-grooves. This co-existed micro-nano groove features might increase the specific surface area of the textured samples and provide more opportunity to entrap air, resulting in increase of contact angle.

Fig. 3 shows SEM photos of micro-grooved Ti_3SiC_2 surface after WEDM processing. The regular and controllable micro-grooved array structures with different width and spacing sizes can be observed on WEDM textured Ti_3SiC_2 surfaces. The groove topographies of all the samples were roughly identical to the results as shown in Table 1. The inset in the Fig. 3(f) was the enlarged picture of the groove bottom on sample F. Many pores, micro-cracks and debris were also observed on the groove bottom. Because the grain defects were vulnerable to the impact of the cathode spots, grain boundaries on polycrystalline Ti_3SiC_2 was locally heated, resulting in production of pores accompanied with microcracks. The debris adhered on groove bottom might be attributed to the splash of the molten Ti_3SiC_2. The SEM analysis was consisted