Tribological performance of microstructured surfaces with different wettability from superhydrophilic to superhydrophobic

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Abstract: The anti-friction function of superwetting surfaces with superhydrophobicity has been demonstrated. However, the influence regularity of wettability to tribological performance, and the underlying mechanism are still unclear. Here, two kinds of microstructured surfaces with different wettability are fabricated on the substrate of steel by controlling surface chemical compositions. The water contact angles on these surfaces range from 0° to 151°. The ball-plate tribological tests are performed under water lubrication. The results show that the tribological performance is closely related to surface wettability. The friction coefficient increases with the increase of contact angles when the surfaces are hydrophilic rather than superhydrophobic. In contrast, the friction coefficient on the hydrophobic surfaces decreases with the increase of contact angles. Furthermore, the best anti-friction capability is obtained on the superhydrophobic surfaces, and the anti-friction mechanism is elucidated. The lowest friction coefficient was 0.12 under the load of 10 N. This work provides strong evidence of an association between tribological property and wettability, which may inspire the fabrication and application of special wetting surfaces in friction control.

1 Introduction

The improvement of the tribological performance of metal surfaces has aroused considerable attention due to the strong demands in the fields of machinery, aviation, chemical engineering, electronics, and so on [1–3]. Researchers have made many academic achievements in tribological theory, among which lubrication is considered to be the most effective measure to reduce friction and control wear [4, 5]. A stable lubricating film can be formed by adding lubricant to avoid direct contact of the friction surfaces and build an interface layer with superior loading capacity, achieving friction reduction [6, 7]. However, with respect to microscale friction systems, such as high-precision machinery, micro-electromechanical systems, and micro-robots, the microscopic force on the friction interfaces plays a vital role in the tribological performance, due to the light weight, small gap, and large specific surface area of the friction systems. In addition, the tribological property is readily affected by the uneven distribution of lubricating fluid and the adhesive force between the lubricating fluid and the friction surface [8–10].

Over the past decades, researches have shown that the friction and adhesion in microscale friction systems are caused by many factors, such as the gap between friction interfaces, surface topography, surface free energy, and surface mechanical property. Nevertheless, few studies have investigated the relationship between tribological property and surface wettability [11]. In fact, after evolution for millions of years, organisms in nature display near-perfect coordination and unification in many aspects, for instance, structure, materials, and surface energy [12, 13]. Notably, some biological surfaces simultaneously possess excellent tribological performance under water lubrication and special wettability. For example, human joints show hydrophilicity and low friction coefficient, lotus leaves have superhydrophobic and low-adhesion properties, and \textit{Nepenthes} peristome depicts superhydrophobic and slippery characteristics [14–16]. This is because that the appropriate wetting and adhesive properties contribute to the lubricating of friction interfaces under aqueous conditions [17–19]. Therefore, it is a promising approach to improve tribological performance through regulating surface wettability.

Recently, some studies have focused on the wettability and tribological performance of biomimetic surfaces [9, 20]. Lu [21] prepared a biomimetic superhydrophobic surface with hierarchical structures to reduce the frictional drag at the solid–liquid interface and realise the superior lubrication. Conradi \textit{et al.} [22] regulated the surface wettability and tribological property by changing the surface morphology. Gong [23] fabricated a hydrophilic hydrogel with both excellent mechanical and anti-friction properties. However, most of these studies have only focused on the preparation of artificial surfaces with special wettability and the verification of anti-friction performance. To date, very little attention has been paid to the relationship between tribological performance and wettability. The effect of wettability on tribological property needs to be further studied [24, 25]. It is worth mentioning that surface wettability can be controlled by applying external stimuli, such as pH, light, electricity, and temperature, which provides a basis for investigating the influence of wettability on tribological performance [26–30].

Herein, we prepared a series of microstructured surfaces with different wettability. Subsequently, the tribological performance of these wetting surfaces ranging from superhydrophilic to superhydrophobic was investigated under water lubrication. The results demonstrated that the wettability had a significant effect on the tribological behaviour. The lowest friction coefficient was achieved on the as-prepared superhydrophobic surface. Further, the relevant tribological mechanism was analysed to provide a theoretical basis for the fabrication of excellent anti-friction surfaces.

2 Experimental section

2.1 Materials

Steel (45#) plates with the size of 25 mm (length) × 25 mm (width) × 10 mm (thickness) were obtained from a retail store.
All chemical reagents were obtained from Sinopharm Chemical Reagent Co., Ltd, China without further purification.

2.2 Preparation of microstructured surfaces with different wettability

Steel plates were first polished with 600#, 1500#, and 2000# grades sand papers for several minutes, respectively. The micropillar arrays were prepared on the steel surfaces using an electrical discharge machining system. Subsequently, the silver layer was electrodeposited on the microstructured surface. In detail, the electrolyte consisted of AgNO₃ (40 g l⁻¹), CH₃COONH₄ (77 g l⁻¹), KOH (50 g l⁻¹), and K₂CO₃ (75 g l⁻¹). The pH of the electrolyte was adjusted to 7 by adding HCl and NH₃·H₂O solution at 20°C. The electrodeposition time and current density were 40 min and 0.3 A dm⁻², respectively. After electrodeposition, the specimens were immersed in a mixed ethanol solution of HS(CH₂)₁₁CH₃ and HS(CH₂)₁₀COOH for 10 h. The mixture concentration was 1 mM l⁻¹, and the molar fraction of HS(CH₂)₁₁CH₃ was changed from 0 to 1. To obtain different hydrophilic surfaces, some samples were modified by the mixed solution in which the molar fraction of HS(CH₂)₁₁CH₃ was 0.6, and then were immersed in NaOH solution (pH=10–14) for 5 h.

2.3 Characterisation

The surface morphology of the samples was characterised by scanning electron microscope (SEM, ZEISS, EVO18) and laser scanning confocal microscope (LSCM, ZEISS, OLS3000). At room temperature, water droplets (5 μl) were dropped at three different positions of each sample, and the average contact angle was obtained using a contact angle meter (KRUSS, DSA25S).

2.4 Tribological test

The tribological performance was examined using a reciprocating ball-on-plate tribometer (CETR, UMT3). The diameter of the bearing steel ball (GCr15) was 10 mm. Before the testing, a water droplet (40 μl) was supplied onto the specimen, as shown in Fig. 1. The sliding time, sliding speed, and reciprocating stroke were 10 min, 10 mm s⁻¹, and 5 mm, respectively. To investigate the influence of load on tribological property, the tribological tests were performed under two kinds of load (5 N, 10 N). The average friction coefficient was obtained by measuring three different positions of the same surface.

3 Results and discussion

3.1 Surface morphology and wettability

The micropillar arrays with the width of 120 μm, and the inter-pillar spacing of 200 and 300 μm were designed. Then, the micropillar arrays on the steel plates were obtained by combining the electrical discharge machining and electrodeposition technologies. After preparation, we characterised the surface morphology by SEM and LSCM. It was observed from Fig. 2 that the surface was comprised of rectangular pillar arrays. The magnified SEM images showed that the top of the pillars was covered by micropapillae, which increased the roughness of the surface. From the LSCM images, it was found that the height of the two different micropillar arrays was ~70 and 55 μm, respectively. The results

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Fig. 1 Schematic illustration of the friction test

Fig. 2 Surface morphology of the two kinds of samples

a, b SEM images
c LSCM image of the micropillar array with the width of 120 μm and the inter-pillar spacing of 200 μm
d, e SEM images
f LSCM image of the micropillar array with the width of 120 μm and the inter-pillar spacing of 300 μm

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illustrated that uniform micropillar arrays were prepared on the steel substrate using the method.

Fig. 3a depicts the wetting property of the two kinds of microstructured surfaces. When the molar fraction of HS(CH$_2$)$_{11}$CH$_3$ was 0, the microstructured surfaces showed superhydrophilicity (contact angle = 0°). With the increase of the molar ratio of the methyl thiol, the contact angle increased, and the surface wettability changed from superhydrophilic to superhydrophobic. When the molar ratio of HS(CH$_2$)$_3$CH$_3$ was 0.2, 0.4, 0.6, 0.8, and 1.0, the contact angles of water droplets on the surface with the microstructure size of 120 μm × 200 μm were 11°, 12°, 14°, 14°, and 15°, respectively. With regard to the surface with the microstructure size of 120 μm × 300 μm, the corresponding contact angles were 8°, 126°, 130°, 138°, and 149°, respectively. The results showed that most of the as-prepared surfaces displayed hydrophobicity. Subsequently, we fabricated various hydrophilic surfaces by immersing the modified surfaces into NaOH solution (pH = 10–14) for 5 h. It can be seen from Fig. 3b that the contact angles of the microstructured surface with the size of 120 μm × 200 μm were 96°, 80°, 76°, 50°, and 0°, respectively. After being treated with NaOH solutions, the contact angles of the microstructured surface with the size of 120 μm × 300 μm were 90°, 78°, 51°, 35°, and 0°, respectively.

As shown in Fig. S1, we further analysed the formation mechanism of different wetting surfaces. Both HS(CH$_2$)$_3$CH$_3$ and HS(CH$_2$)$_{10}$COOH could be grafted to the Ag layer through the Ag–S bond. After grafting, the surfaces modified by HS(CH$_2$)$_{10}$COOH exhibited superhydrophilicity due to the hydrophilic carboxyl group. However, the addition of HS(CH$_2$)$_3$CH$_3$ enhanced the hydrophobicity because of the increase of the hydrophobic methyl and methylene groups. Therefore, the wettability of our microstructured surfaces was successfully regulated by changing the molar ratio of HS(CH$_2$)$_3$CH$_3$ and HS(CH$_2$)$_{10}$COOH. The wetting performance of the as-prepared surfaces could be further changed in response to pH. With the increase of pH (pH > 7), the carboxyl group of HS(CH$_2$)$_{10}$COOH was gradually ionised, and the formed hydrate layer would lead to the enhancement of the hydrophilic property. Finally, various surfaces with different wettability were obtained, which was helpful to further explore the tribological performance of the special wetting surfaces.

### 3.2 Tribological behaviour of the wetting surfaces

To investigate the influence of wettability on the tribological performance, we chose the samples with different wetting property for following tribological tests. For the samples with the microstructure size of 120 μm × 200 μm, the contact angles were 0°, 50°, 80°, 96°, 125°, and 151°, respectively. With respect to the samples with the microstructure size of 120 μm × 300 μm, the tribological behaviour of the surfaces with contact angles of 0°, 35°, 51°, 78°, 90°, 126°, and 149° was investigated.

Fig. 4a shows the friction coefficient of the specimens with different wettability under the load of 5 N. The friction coefficient first decreased, then increased, and finally decreased with the increase of contact angles. In detail, when the surfaces were hydrophilic, the friction coefficient of the surface with the microstructure size of 120 μm × 200 μm changed from 0.18 to 0.23 with the contact angle increasing from 50° to 80°. With increasing the contact angle of another microstructured surface from 35° to 78°, the friction coefficient shifted from 0.19 to 0.27. Conversely, as the contact angle of the hydrophobic surface with the microstructure size of 120 μm × 200 μm increased from 125° to 151°, the friction coefficient decreased from 0.32 to 0.14. When the contact angle of another microstructured surface increased from 126° to 149°, the friction coefficient decreased from 0.34 to 0.15. For superhydrophilic surfaces (contact angle = 0°), the lubricating liquid would fully penetrate into the gap between micropillars due to the strong capillary force, which affected the lubricating effect and resulted in an increase of the friction coefficient when compared to the hydrophilic surfaces. It should be noted that superhydrophobic surfaces showed the best anti-friction performance. Compared to the superhydrophobic sample with the microstructure size of 120 μm × 200 μm, the friction coefficient of the samples with contact angles of 0°, 50°, 80°, 96°, and 125° was increased by 128.57, 28.57, 60.71, 132.14, and 150%, respectively (Fig. 4b). Meanwhile, the friction coefficient of the specimens with contact angles of 0°, 35°, 51°, 78°, 90°, and 126° was increased by 93.33, 26.67, 66.67, 80.00, 126.67, and 86.67%, respectively, in comparison with the superhydrophobic specimen with the microstructure size of 120 μm × 300 μm. The results demonstrated the significant correlation between the tribological property and wettability.

As shown in Fig. 4c, the friction coefficient of the specimens under the load of 10 N exhibited a similar trend to that of the samples under the load of 5 N. For hydrophilic surfaces, as the contact angle of the surface with the microstructure size of 120 μm × 200 μm increased from 50° to 80°, the friction coefficient changed from 0.20 to 0.23. The friction coefficient of another microstructured surface varied from 0.17 to 0.26 with the increase of the contact angle from 35° to 78°. With respect to hydrophobic samples, the surface with the microstructure size of 120 μm × 200 μm decreased from 0.27 to 0.14 with increasing the contact angle from 125° to 151°. As the contact angle of another microstructured surface increased from 126° to 149°, the friction coefficient decreased from 0.28 to 0.12. When compared to the superhydrophobic sample with the microstructure size of 120 μm × 200 μm, the friction coefficient of the samples with

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**Fig. 3** Wetting property of surfaces after different chemical treatments

(a) Water contact angles of the surfaces as a function of the concentration of the methyl thiol

(b) Water contact angles of the surfaces immersed in NaOH solution with different pH
contact angles of 0°, 50°, 80°, 96°, and 125° was increased by 76.00, 60.00, 84.00, 116.00, and 116.00%, respectively (Fig. 4d). In addition, the friction coefficient of the surfaces with contact angles of 0°, 35°, 51°, 78°, 90°, and 126° was increased by 106.35, 34.92, 82.54, 110.32, 126.19, and 118.25%, respectively, compared with the superhydrophobic specimen with the microstructure size of 120 μm × 300 μm. It was discovered that the friction coefficient was relatively small under high loads. These results confirmed that surface wettability significantly affected tribological performance. One can control the friction behaviour by changing surface wetting property.

Next, we further analysed the typical friction process under the load of 10 N. Fig. 5a shows the friction coefficient of the surfaces with the microstructure size of 120 μm × 200 μm. Obviously, the surface with the contact angle of 151° exhibited the lowest and most stable friction coefficient. The friction coefficient of the other surfaces was unstable, and the running-in period was relatively long. As for the tribological behaviour of the surfaces with the microstructure size of 120 μm × 300 μm, the friction coefficient of the surface with the contact angle of 149° was the lowest (Fig. 5b). In addition to the superhydrophobic surface, the hydrophilic sample with the contact angle of 35° also displayed a lower and more stable friction coefficient, and a shorter running-in period than other surfaces. It was concluded that the superhydrophobic surfaces could provide the best anti-friction performance.

The typical wear behaviour of the samples with different wetting property was investigated. Fig. 6 displays the worn morphology of
As shown in Fig. 7c, the enhancement of hydrophobicity weakened the adhesion of lubricating fluid on the surfaces, leading to the instability of lubricating films during the friction process. The thickness and stability of lubricating films had a great influence on the tribological behaviour. As the wetting states changed from hydrophobic to superhydrophobic states, the friction coefficient decreased to the minimum value. On the one hand, the great wettability difference promoted the flow of lubricating liquid from the superhydrophobic specimens to the steel balls, contributing to the formation and stability of the lubricating film on the balls (Fig. 7d) [17, 32]. Even if the lubricating film was destroyed during friction, it would be easily recovered. Under this condition, although there were no lubricating films adsorbed on the superhydrophobic surfaces, the lubricating film adhering to the steel balls could avoid the direct contact of rough peaks, and reduce the friction coefficient. On the other hand, water lubricant took away the wear debris from the superhydrophobic specimens [31]. The timely removal of the wear debris was beneficial to shorten the running-in period, and then obtain stable and low friction coefficient.

4 Conclusions

In summary, various surfaces with different wettability ranging from superhydrophilicity (0°) to superhydrophobicity (151°) were fabricated through combining surface texturing and chemical modification. Then, the tribological tests were carried out under water lubrication. The results showed that the friction coefficient of hydrophilic surfaces increased with the increase of contact angles. With respect to superhydrophobic surfaces, the friction coefficient was higher than some hydrophilic surfaces. This was because that the lubricating films on the superhydrophobic surfaces were thinner than that on the hydrophilic surfaces. The friction coefficient dropped quickly and reached at the lowest value, as the wettability of the as-prepared surfaces changed from hydrophilicity to superhydrophobicity. The great wettability difference between friction interfaces and the capability of water to take away wear debris caused the low friction coefficient. We envision that the discovery of the relationship between wettability and tribological performance can inspire the fabrication and application of more wetting surfaces for friction control.

5 Acknowledgments

The authors thank the National Natural Science Foundation of China (No. U1601203), and Young and Middle-aged Science and Technology Innovation Team Project of Jilin Province (No. 20180519007JH).

6 References

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3.3 Analysis of the tribological mechanism

For superhydrophilic specimens, water lubricant could rapidly spread on the surfaces, and thereby lubricant films were formed. However, the friction coefficient of these superhydrophilic surfaces was higher than that of some hydrophilic surfaces. When a small water droplet was dropped on the superhydrophobic surface, most of the liquid was trapped in the gap between micropillars, and a little water was used to form a thin lubricating film (Fig. 7a). Hence, the superhydrophilic surfaces showed relatively large friction coefficient. With the increase of contact angles, the friction coefficient decreased. This was because that more water could be remained on the sample surfaces, and a thick lubricating film was formed (Fig. 7b). Nevertheless, when contact angles continued to increase, the friction coefficient exhibited an upward trend.

the surfaces with the microstructure size of 120 μm × 200 μm under the load of 10 N. It was observed that the top of the micropillars became smoother, indicating the destruction of the surface microstructure. Compared to other wetting surfaces, there was no obvious wear debris around the micropillars of the superhydrophilic surface. This was because that the lubricant was easy to flow on the superhydrophobic surface, and thereby the wear debris was removed [31].

Fig. 6 SEM images of the worn surfaces with different contact angles

- a 0°
- b 50°
- c 80°
- d 96°
- e 125°
- f 151°

Fig. 7 Tribological mechanism of the samples with different wettability

- a superhydrophilic
- b hydrophilic
- c hydrophobic
- d superhydrophobic
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