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Drag reduction of stable biomimetic superhydrophobic steel surface by acid etching under an oxygen-sufficient environment

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Abstract
Superhydrophobic surfaces have shown utility applications in drag reduction field. A novel method based on simulation analysis and test experiments is proposed to fabricate a superhydrophobic surface with 3D flower-like micro and nano-structures on a steel ball under an O₂ rich environment. The superhydrophobic steel surface has water CA of 166 ± 1.5°. The sliding angle is less than 2°. The experiment and the simulation of the superhydrophobic and the untreated steel ball fall under water are built to prove the validity of the method of reducing water resistance. The drag reduction ratio of the superhydrophobic steel ball is beyond 53% opposed to the untreated surface under water. A model simulation is built to simulate and analyze the solid-liquid interface drag reduction mechanism of superhydrophobic surface based on theoretical analysis. The result testifies the rationality of the drag reduction experiment.

1. Introduction

Due to the intimate involvements of superhydrophobic property with nature and human society proceeding, most of scientific researches and industrial productions always focused on comprehending behaviors and achieved drag reduction to benefit transport industry [1–7]. In nature, water-repelling surfaces of floating plants and semiaquatic insects are capable of utilizing produced superhydrophobic properties to easily move through water [8]. And on this basis superhydrophobic surfaces attract more attention in the fields of industrial production and academic research. Song et al design a serial wedge-shaped wettability pattern with the superaerophobic-superaerophilic region to realize long-distance spontaneous and directional pumpless transportation of subaqueous gas bubbles driven by unbalanced surface tension forces [9]. They also process a novel superhydrophobic surface preparation method to fabricate micro/nano structures on an Al mold with intensive conical holes by nanosecond laser drilling and HCl etching for droplet pancake bouncing [10]. Under the assistance of air film between water and hydrophobic solid surface, these structures can reduce the contact area between solid and liquid with obviously reducing the friction drag [11–15]. Therefore, achieving the favorable performance of the superhydrophobic surface has attracted more attentions of researchers while researches about drag reduction were still an urgent need in various fields [16, 17].

In recent years, the drag reduction effect of superhydrophobic surfaces has been studied both theoretically and experimentally [18, 19]. Cheng et al have fabricated a mechanically stable drag reducing coating composed of elastic polydimethylsiloxane and hydrophobic copper particles on model ships, which can reduce hull abrasion and reveal a durable drag-reducing effect [20]. Mohamed et al fabricated durable superhydrophobic polystyrene span-fiber coatings and studied the frictional resistance of water movement toward investigating the impact of environmental factors on the performance of superhydrophobic surfaces [21]. Doo Jin Lee et al analyzed that liquid slip increases with decreasing channel height and depth, resulting in drag reduction by
fabricating superhydrophobic nano Zinc oxide surfaces [22]. However, current research focuses on drag reduction experiments. Therefore, we propose a novel method to fabricate a stable biomimetic superhydrophobic surface on a steel substrate by chemical etching under an oxygen environment in diluted acidic solution, and build a model simulation to analyze the solid-liquid interface drag reduction mechanism of superhydrophobic surface for verifying the rationality of the drag reduction experiment based on theoretical analysis using COMSOL. A Fe₃O₄ film with 3D nanoflower-like structures is grown on the steel substrate treated with the acidic solution that the main components are hydrochloric acid and potassium chloride under an O₂ rich environment in the growth process. The as-prepared steel surface after modified with fluorinated silane shows superhydrophobic property with water CA of 166 ± 1.5° and the sliding angle is approximately 2°. Moreover, due to taking advantages of superhydrophobic properties of steel ball surface under the optimal operation conditions, it falls faster than untreated steel ball from the same position and reduced water resistance. Experimental results indicate that superhydrophobic surfaces have considerable friction drag reduction effect, correspond to the simulation results.

2. Experimental

2.1. Preparation

Steel foil was cut into 3 × 3 cm² iron, followed by 800#, 1200# and 1500# sandpaper for grinding. Using a hot press after flattening with acetone, ethanol and deionized water was ultrasonically cleaned for 5 min 200 ml of 0.2 mol l⁻¹ potassium chloride solution was added, and then hydrochloric acid was added dropwise to adjust the pH value to about 3. The cleaned steel foil was soaked in the solution, and oxygen was introduced and reacted for different etching time at 60 °C. The steel pieces were washed in ethanol and dried with nitrogen when the principal reaction was accomplished. Finally, these samples were immersed in an ethanol solution of 0.5 wt% FAS for 30 min and dried in an oven at 120 °C for 60 min. The surface structure was fabricated on the steel ball in the same way.

2.2. Characterization

The surface morphology is observed using a field-emission scanning electron microscope (FE-SEM, TESCAN VEGA). Element distribution of the sample is determined using energy-dispersive x-ray spectroscopy (EDS). The water contact angles (WCAs) and sliding angles (SAs) are evaluated with 10 μl droplets of water using an optical contact-angle meter system (Data Physics Instrument GmbH, Germany). The average WCAs and SAs values are obtained as a result of measuring each sample at a minimum of five different positions at room temperature.

3. Results

3.1. Surface morphology and structures

For the superhydrophobic surfaces, surface morphology and micro/nano-structures play significant parts in wettability of surfaces. Steel is hydrophilic materials with a native oxidized layer that has a contact angle far less than 90° for water. A rough substrate is a prerequisite to achieve the superhydrophobic surface. Therefore, acid etching and oxidation are utilized to prepare roughened surfaces under an oxygen-sufficient environment. The as-prepared steel ball is observed by scanning electron microscope (SEM). Figure 1 is the optical and SEM image of bare and treated steel ball. It shows that the surface of the steel ball gradually turned gray-green, and the color gradually deepened until it is removed at 25 min. The steel ball surface after the oxidation treatment had significant changes. The color is changed from silver (figure 1(a)) with metallic luster to dark black (figure 1(c)). When using iron tweezers to touch the steel ball, the surface shows a slight magnetism. From the figure 1(b), the bare steel ball has a relatively smooth surface. After the previous etching in a lower concentration, the surface of steel ball obtains rough morphology as showed in figure 1(d). The prepared surface can exhibit more roughness with complex and regular flower-like microstructures which the size of the protrusions is about 80–120 nm in the high magnification.

The morphologies of the products are different from those of the samples under the oxygen-sufficient environment. As a comparison, when there is no O₂ during the reaction, the surface of the steel foil grows some irregular cracks at the reaction time of 6 min, and produces blocky structures after acid etching which consist of relatively flat gullies by enlarging the image within the red box in figure 2(a). A few of nanoflowers structures grows on the surface after 20 min which make the surface become more roughness in figure 2(b). As shown in figure 2(c), the gradually increasing micro/nano structures have still been shapeless when the reaction time reaches 25 min. The size of petal-like projections structures has increased as time reaches 28 min and a number of nano-scale papillae can be observed in the red box image in figure 2(d). But the surfaces are relatively flatter
Figure 1. Optical photographs taken at two slightly different orientations perpendicular to steel ball surface. (a) Before and (c) after grow for 25 min in acidic solution with introduced oxygen bubbles. (b) and (d) Corresponding SEM images of the steel ball surfaces and Fe$_3$O$_4$ films.

Figure 2. SEM images of the prepared samples at 60 °C in the acidic solution for (a) 6 min, (b) 20 min, (c) 25 min, (d) 28 min in 60 °C acidic solution under oxygen-deficient environment.
than these under an oxygen-sufficient environment. Therefore, the results can prove the necessity of an oxygen-
sufficient environment. It has a great influence on the morphology of the product and superhydrophobicity.

3.2. Chemical composition of the prepared surfaces
Energy-dispersive x-ray spectroscopy (EDS) is used to determine the corresponding element distribution.
Figure 3(a) shows the EDS spectrum of the after-prepared samples, which indicates the presence of Fe, O and C
elements. It shows the EDS spectrum of the resultant Fe3O4 sample. The strong peaks for Fe and O are observed
in the spectrum with the atomic ratio of 1:1.29, which is on the verge of the theoretical value (1:1.33) that of
magnetite, indicating that iron surface after the reaction covered with black material should be iron oxide Fe3O4,
which shows a possible mechanism for the formation of magnetite nanosheets. Figures 3(b) and (c) show XRD
patterns of both as-prepared samples under the oxygen-sufficient and oxygen-deficient environment,
respectively. Diffraction peaks of the samples are all indexed to pure iron [PDF file: 85-1410] and magnetite
[PDF file: 79-0419]. Fe is at the existence after acid etching under oxygen-deficient environment from
figure 3(b). According to the XRD patterns of the sample, the three strong peaks located at 44.3°, 64.5° and 81.7°
are ascribed to (110), (200) and (211) planes of Fe, respectively. The indexed main peaks located at 30.2°, 35.6°,
43.3°, 57.3°, and 62.9° ascribed to (220), (311), (400), (511), and (440) planes of magnetite can be observed from
the XRD patterns in figure 3(c). It is indicated that Fe3O4 is the main composition of micro/nano structures
formed on the steel surface.

When the steel ball is immersed into the hydrochloric acid, Fe will pass into solution as Fe2+ and then
Fe(OH)2 is usually formed when O2 is required for the oxidation of steel. Under an oxygen-sufficient solution,
Fe(OH)2 is converted into Fe3O4 · H2O. This process can be described by the following composite chemical
equation [23]:

\[
6\text{Fe}^{2+} + 12\text{OH}^- + \text{O}_2 \rightarrow 2\text{Fe}_3\text{O}_4 + 6\text{H}_2\text{O} + 12\text{e}^- , \tag{1}
\]

3.3. Effect of reaction time on wetting behavior
The morphology of the surface structures on the steel substrate has been observed using SEM. It can be noted
that etching time and temperature can control the size and morphology of the resulting surface under an
oxygen-sufficient environment. From figure 4(a), the surface of the steel foil only sparsely grows some irregular
micro/nano scale wrinkles at the reaction time of 6 min, and these structures are composed of curled nanowalls
by enlarging the image within the red box. A further increase of the immersion time leads to an increased surface
density of the clusters and nanosheets, and the nanosheets start to grow upwards, a few of nano flowers are
formed on the surface of steel foil with the gradual increasing of the quantity and size (figure 4(b)). The micro/
nanostructures have been very dense in the microscope. When the reaction time reaches 28 min (figure 4(c)), a
number of nano papillae with a diameter of 1–3 μm can be observed in the red box image, but the surface is
relatively flatter than that of figure 1(d). The petals outside the papillae offer nano-scale roughness on the surface, leading to the formation of hierarchical nanoflakes structures. The shape, distribution and density of the surface topography are very close to that of the natural lotus leaf surface.

As the surface roughness plays a major role in the superhydrophobicity, the effects of surface roughness on the wetting properties are studied by comparing the WCAs of the steel surfaces obtained at different reaction temperatures after FAS modifications. The fluorinated binary structures have probably already trapped enough air to prevent the penetration of water droplets into the nanosheets. It will cause the superhydrophobicity on the surfaces. From figure 4(d), the contact angle increases with increasing reaction time, then reaches a peak at 25 min with a peak value of 166°. The contact angle will decrease by degrees because the thickness of the oxide layer becomes thick as the reaction time increases. There is some internal stress between the thicker oxide layer and the steel substrate. Therefore, due to stress the thick oxide layer has some cracks, leads to the decrease of the WCAs in the process of drying at high temperature after fluorination. An appropriate oxide layer thickness when reaction time is 25 min is optimum for the superhydrophobic property.

3.4. Falling ball drag reduction experiment

On the basis of the preparation of superhydrophobic surface, the iron sheet is replaced by a steel ball with a diameter of 5 mm. In order to estimate the drag reduction property of the as-prepared surface, the experimental setup of the liquid/solid friction drag is built in this paper. The force of the steel ball in the water is analyzed. As shown in figure 5(a), when a steel ball enters the water, it is subjected to three forces, gravity, buoyancy and water resistance to a steel ball, respectively. The quality and volume of steel ball before and after the treatment will not be changed so that the resistance can be obtained by measuring the resultant force, namely the acceleration in the water minus the value of the gravity and buoyancy. From figure 5(b), the distance between a cylinder mouth and the liquid level is $h_1$ when the height of the liquid level is $h_2$. When the experimental balls with different hydrophobicity do free-fall motion from the graduated cylinder mouth, the time going through $h_1$ in air should be the same. By measuring the time passed by $h_2$, the resistance of the steel balls in the water can be obtained by the calculated resultant subtracting gravity and buoyancy force. The calculation method is described as follows:

Hypothesise two balls go down from the mouth of the graduated cylinder into the liquid with the speed of $v_0$ and then the following physical formula is available:

![Figure 4. SEM images of the nanosheets film prepared at 60 °C in the acidic solution for (a) 6 min, (b) 20 min, (c) 28 min (d) The water CAs of the as-prepared steel balls under the different reaction time in 60 °C acidic solution under O2 rich environment. Surfaces have been modified by fluorinated silane after chemical etching and oxidation.](image)
According to energy conservation law:

\[ mgh = \frac{1}{2} m v_0^2, \]  

The acceleration of the steel balls can be solved:

\[ a = \frac{2 \left( \frac{h_1}{t^2} - \frac{\sqrt{2gh_1}}{t} \right)}{\frac{h_2}{t^2} - \frac{\sqrt{2gh_1}}{t}}, \]

Then, the resistance of the steel balls in water can be described as:

\[ F = mg - \rho g V = 2m \left( \frac{h_1}{t^2} - \frac{\sqrt{2gh_1}}{t} \right). \]

Therefore, it can acquire water resistance to two balls just measuring the quality of the balls, the volume, the time of the balls falling in the water and the water level in the graduated cylinder.

We fill the cylinder with deionized water, and then release the superhydrophobic steel ball with treatment that the best reaction condition is at 60 °C and 25 min and the steel ball without treatment from the mouth of the graduated cylinder at the same time. The high speed camera is recorded the falling time. Each steel ball is measured 5 times and then the average value can be obtained. The resistance of each steel ball can be computed via equation (5).

Meanwhile, steel balls with superhydrophobic surface are surrounded by bubbles when they fall in water. The gas forms a gas-liquid interface at the groove in its surface. The steel balls with different processing time are released from the top of the graduated cylinder. The resistance of the steel ball at the different reaction time to water can be obtained in figure 6(a). Figure 6(b) shows drag reduction ratio versus different reaction time of the falling superhydrophobic steel ball with the different CAs. It indicates that the highest drag reduction ratio is about 84.1% when the reaction time of the steel ball is 25 min.

3.5. Simulation model and theoretical analysis

To testify the rationality of the drag reduction experiment, a 3D finite-element method (COMSOL Multiphysics v5.2a) was used to build a model to simulate this situation (figures 7(a) and (b)). The simulation model uses fluid solid coupling physical field (FSI), and transient simulation to simulate the process of falling of steel balls in the water. According to Reynolds equation, at the maximum of the flow velocity, when Re is less than 2000, the numerical imitation is carried out in the laminar state. For stable state incompressible laminar flows, the fluid motion relative to solid can be described by the continuity equation.

\[ \frac{\partial u}{\partial x} + \frac{\partial v}{\partial y} + \frac{\partial w}{\partial z} = 0, \]
Momentum equation is as follows:

\[
\rho \left( u \frac{\partial u}{\partial x} + v \frac{\partial u}{\partial y} + w \frac{\partial u}{\partial z} \right) = -\rho \frac{\partial p}{\partial x} + \mu \left( \frac{\partial^2 u}{\partial x^2} + \frac{\partial^2 u}{\partial y^2} + \frac{\partial^2 u}{\partial z^2} \right)
\]

\[
\rho \left( u \frac{\partial v}{\partial x} + v \frac{\partial v}{\partial y} + w \frac{\partial v}{\partial z} \right) = -\rho \frac{\partial p}{\partial y} + \mu \left( \frac{\partial^2 v}{\partial x^2} + \frac{\partial^2 v}{\partial y^2} + \frac{\partial^2 v}{\partial z^2} \right)
\]

\[
\rho \left( u \frac{\partial w}{\partial x} + v \frac{\partial w}{\partial y} + w \frac{\partial w}{\partial z} \right) = -\rho \frac{\partial p}{\partial z} + \mu \left( \frac{\partial^2 w}{\partial x^2} + \frac{\partial^2 w}{\partial y^2} + \frac{\partial^2 w}{\partial z^2} \right)
\]

Where \( \mu, v, w \) are the velocities (m s\(^{-1}\)) in the \( x, y, z \) directions; \( \mu \) is the dynamic viscosity (Pa·s); \( \rho \) is the density (kg m\(^{-3}\)); and \( p \) is the pressure (Pa) [24].

In the original 3D model (figure 7(a)), the size of the water area is 60 cm \( \times \) 80 cm, and the diameter of the steel ball is 5 cm. The upper part of the water is an open boundary, and other parts of the boundaries is set without sliding, which can minimize the influence of the water boundary on the calculation. The steel ball’s material is a kind of steel in a density of 7870 kg m\(^{-3}\). Both steel ball and water are affected by gravity. The steel ball moves downward under the action of gravity. In the contrast model, a laminar level set of physical field is added based on the original physical field, the surface of the steel ball is set as wetting walls with contact angles of

Figure 6. (a) Friction resistance versus the reaction time of the falling superhydrophobic steel ball. (b) Drag reduction ratio versus different reaction time of the falling superhydrophobic steel ball with the different CAs.

Figure 7. (a) and (b) the simulated analysis of the established untreated and superhydrophobic steel ball virtual model. (c) Friction resistance versus different CAs of the superhydrophobic steel ball after different reaction time (d) Drag reduction ratio versus different reaction time of the falling superhydrophobic steel ball of different CAs.
Thomas Young studied the free energy of solid surface and proposed the calculation of the contact angle formula, which is called Young’s equation [25]:

$$\cos \theta = \frac{\lambda_{sv} - \lambda_{sl}}{\lambda_{lv}},$$

where $\lambda_{sv}$, $\lambda_{sl}$, and $\lambda_{lv}$ represent solid-gas, solid-liquid, and liquid-gas interfacial tensions, respectively. $\theta$ is the balance CA or the intrinsic CA of the substrate materials. The CA is determined by the interactions across the three interfaces on the smooth surface.

In the actual situation, steel ball surface roughness can be considered by the modified Cassie—Baxter model in which the water droplets are suspended in the solid surface and are not infiltrated into the rough structures [26]. The angle corresponding to the lowest free energy of this ball falling system is given by the Cassie—Baxter equation [27]:

$$\cos \theta_{sw} = f_1 \cos \theta_1 + f_2 \cos \theta_2,$$

In this equation, $\theta_1$ and $\theta_2$ are the apparent CAs of the liquid on solid and air, respectively. $f_1$ and $f_2$ are the apparent area fractions of solid and air ($f_1 + f_2 = 1$).

This equation can be applied to the system composed of solid, liquid and air, because the steel ball surfaces with rough groove structures can preserve air bubbles. Bubbles can affect the state of fluid motion on a solid surface. The fluid motion in the surrounding air area satisfies the Navier–Stokes equation [28].

Figure 7(b) indicates the velocity field (where the red arrow is pointing) and flow field distribution (where the green arrow is pointing) of the falling superhydrophobic steel ball surface. The colour change proved that the velocity around the superhydrophobic steel ball surface is bigger than that of the other from the inset of the velocity field. Gas/liquid volume fraction distribution demonstrates the presence of an air bubble layer at the solid–liquid interface and the surface arrow represents the velocity vector near the wall area of the falling steel ball from the other inset. Fluid flow of the superhydrophobic surface is composed of the slip flow in the grooves and the nonslip flow between the grooves. According to the classic no slip boundary, the wall velocity of flows is approximately zero. The air bubble layer within the grooves that resides between the no-shear solid-liquid interface generates the boundary slippage. Grooves spacing distribution in superhydrophobic surfaces can enhance slippage to the significant benefit of the increasing velocity of falling steel ball in the water compared with smooth steel ball surface [29–32]. Water velocity vector is concentrated in the middle of each groove and peak flow velocity appears near the midpoint of the groove. Therefore, figure 7(b) demonstrates that the superhydrophobic steel ball can fall faster than untreated steel ball from the same position.

In the Cassie–Baxter model of steel ball surface, viscous resistance as the main resistance of the superhydrophobic surface is produced at the velocity gradient near wall area and groove structures only affect the flow field near wall area; pressure resistance is caused by the low vortex formed in the grooves by the pressure differentials between the flow surface and the back flow. The latter plays a significant role in the presence of air in the grooves. Total resistance of the superhydrophobic surface is comprised of them. Velocity direction of low vortex in the grooves is the opposite of flow velocity direction so that the friction resistance in the bottom of the grooves is opposite to the flow direction as an additional driving force for drag reduction between the liquid and the solid surface.

Drag reduction property changes with different roughness surfaces which have different CAs. With the CA increasing, the low vortex produced by the rougher surface increase flow velocity through the superhydrophobic surface and reduce the shear force on the air-water interface. This phenomenon produces drag reduction effect. The resistance and drag reduction ratio show are shown in figures 7(c) and (d). The lowest resistance for 0.0019 N and the highest drag reduction ratio is 63.4% when the CAs is 166°. In general, superhydrophobic surface has considerable friction drag reduction effect the modeling results are aptly in accordance with the experimental results. The drag reduction model of superhydrophobic steel ball verifies the correctness of the experiment.

4. Conclusions

In conclusion, we use the oxygen wet oxidation method to prepare a rough micro/nano structures surface, and further confirm that the surface after fluorination has superhydrophobic property. The experimental result proves the necessity of an oxygen-sufficient environment compared with the samples under oxygen-deficient environment. It has a great influence on the morphology and superhydrophobicity. The composition of the
oxide layer is determined as Fe₃O₄ under the oxygen-sufficient environment, and its reaction mechanism is analyzed by morphology and characterization. Then the influence of reaction condition on the superhydrophobic surface is studied by the control variable method. Finally, it determines that those optional parameters included the 0.2 mol l⁻¹ potassium chloride solution with pH 3, under oxygen rich environment, at 60 °C and 25 min reaction time satisfy the optimum reaction condition. The steel ball treated with the superhydrophobic surface has a faster moving speed than that of the untreated steel ball in water. The highest drag reduction ratio is 84.06% and the biggest CA is 166° indicating that the resistance of the water on the surface of the steel ball is indeed reduced. Based on theoretical analysis, a simulation model is built to analyze solid-liquid interface drag reduction mechanism and the effect of superhydrophobic steel ball on drag reduction. As a result, the highest drag reduction ratio is 63.4%, validates the experimental method and shows drag reduction property is effective and robust in functionality and applications.

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