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A novel approach of preparation and characterization for fourteen acid aluminum superhydrophobic films

In order to improve the preparation level of acid aluminium super-hydrophobic film coating, in this paper, a simple and rapid method for the preparation of super-hydrophobic films was proposed. Aluminium substrate was used as the substrate, and the surface roughness structures were first etched with dilute hydrochloric acid, and then configure the n-dodecyl alcohol, fourteen acid ethanol soaking solution, the treated substrate is immersed in the soaking solution by a tapering machine, and finally, a superfine hydrophobic film having a special surface is obtained by heat treatment. The experimental results show that the surface morphology of the film is observed by scanning electron microscopy, and then measured its hydrophobic angle up to 160.9 degrees.

Key Words: *Superhydrophobic, hydrochloric acid etching, fourteen aluminum acid*

1. Introduction

Organic matter fourteen acid is a kind of phase change material, which has a large phase change enthalpy and stable phase transition [1]. In recent years, the study of super-hydrophobic surfaces has become one of the more active research topics, which plays an important role in the preparation of new high performance functional materials [2]. Super-hydrophobic materials have great prospects for development, such as pipeline transport can reduce friction, outdoor wires can prevent snow, the ship shell can reduce the resistance to save energy and micro-syringe tip can eliminate the waste of expensive drugs.

The application of super-hydrophobic materials is so extensive, but there are still many problems in the quantitative preparation of super-hydrophobic materials [3], many researchers at home and abroad have studied it. Overall, most of the research results are costly, the preparation time is long and the preparation process is complicated.

In this paper, several simple, rapid and low-cost methods for the preparation of super-hydrophobic films were studied, by using pure aluminum as the experimental object, using fourteen and twelve thiol reagent acid on matrix processing, the main component of the surface of $Al[CH_3(CH_2)_{10}CH_2S]_3$ and $Al[CH_3(CH_2)_{12}COO]_3$ superior performance of super hydrophobic surface.

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2. Materials and methods

2.1 Experimental materials and design

$CH_3(CH_2)_{12}COOH$, molecular weight: 228.38), n-dodecyl mercaptan ($C_{12}H_{26}S$, molecular weight: 201.41), and soaking agent anhydrous ethanol (CH_3CH_2OH), substrate (pure aluminum) [4]. The raw materials used in the experiment are shown in Table 1.

Table 1: The main raw materials for the experiment

Raw material name	Specification
Tetradecanoic acid ($CH_3(CH_2)$12COOH)	Analysis of pure AR
N-dodecyl alcohol ($C_{12}H_{26}S$)	Analysis of pure AR
Anhydrous ethanol (CH_3CH_2OH)	Analysis of pure AR

The equipment used in the experiment is: FA2104N electronic balance, HJ-4 multi-head magnetic stirrer, self-made pull-type coating machine, electric heating oven (DHG-020-0), X-ray diffractometer (XRD, D8 advanced, contact angle meter, JSM-6390 scanning electron microscope (SEM), Fourier Infrared Spectrometer.

Among them, the contact angle measuring instrument is used to measure the contact angle of the solid sample surface liquid on the substrate. The scanning electron microscope is used to observe the surface morphology of the sample [5], XRD analysis element composition, infrared spectroscopy, chemical bond.

2.2 Substrate processing method

Pure aluminum is silver and white metal, hardness is small, the density of 2.7×10^3kg/m^3[6]. Pure aluminium in the air surface can generate a layer of alumina film, oxide film can prevent oxygen and aluminium contact to prevent further oxidation, so aluminium in the air has a strong corrosion resistance, the experiment with aluminium as a base. In order to facilitate the experiment will be cut into certain size aluminum [7], the specifications used in this experiment is 25mm × 75mm × 2mm.

The realization of the method is to tear off the surface of the plastic protective film, the aluminium surface with water frosted paper (1500 #) polished until the surface of the aluminium surface matter rough, no longer reflect the light so far. The acetone is then washed to remove the surface grease and washed with distilled water. Physical grinding is to remove the oxide film on the surface of aluminium, to prevent the surface of aluminium oxide oxidation of alumina impact on the experimental results [8]. From the previous theoretical knowledge, super-hydrophobic surface is not flat, but the convex structure, only a certain rough structure of the surface in order to achieve hydrophobic effect.

For the construction of rough surface structure of the problem, the method is to clean the aluminium plate with a clip caught in the new system of 5% - 25% dilute hydrochloric acid soaking solution, to be the surface of the black and gray out, distilled water rinse, dry. After the reaction was dark gray material, that is, experimental structure of micro-nano-scale rough structure. Analysis of black and gray matter according to XRD data is aluminium powder [9]. Aluminium tablets placed in hydrochloric acid products are aluminium chloride, aluminium chloride and soluble in hydrochloric acid, after cleaning the remaining material for the aluminium powder. The aluminium powder produced is easily converted to alumina by reaction with oxygen in the air, so the composition of the black and gray micro-nanometer roughened structure is alumina powder [10]. The reason for the formation of black and gray is because the generated material is micro-nano-aluminum powder attached to the surface of solid aluminum, because the particle size is very small nano-effect, the particles are too small will not reflect the light so are black and gray alumina.

2.3 Soaking solution configuration

Take appropriate amount of raw materials, configure the soaking solution. The concentration of each group and the required raw material quality are shown in Table 2.

The immersion process required for the experiment is as follows:

1. Take 100 ml small beaker, 100 ml of the cylinder cleaning, dry;

TABLE 2: COMPUTING VELOCITIES IN DIFFERENT MASS RATIOS

Group	Concentration (mmol/L)	Quality of n-dodecanediol (g)	Quality of tetradecanoic acid (g)
1	1	0.02024	0.0228
2	5	0.1012	0.114
3	10	0.2024	0.228
4	15	0.3036	0.342
5	20	0.4048	0.456
6	25	0.5060	0.560
7	30	0.6072	0.684

2. The positive mass of twelve mercaptan in the meter was measured by electronic balance;
3. Use the electronic balance weighing table corresponding to the quality of the four acid, into the beaker;
4. Use the cylinder to take 100 ml of anhydrous ethanol into the beaker;
5. Stir with a multi-head stirrer to fully soak solution, mixing;
6. Paste the corresponding concentration label.

2.4 Substrate surface functionalization

The method used herein is pulling the impregnation method. First, the treated substrate was placed on a pull-off coating machine and placed in a soaking solution for 5 minutes [11]. The soaking solution was sufficiently fused with the surface of the substrate, and the reactants were attached to the surface. The substrate was pulled out at a rate of 8 cm/min with a coating machine so that the soaking solution could be uniformly adhered to the substrate, and the plating was carried out twice. Until the substrate surface after drying paste the label, into the electric thermostat oven, preset a good temperature, time. Heat treatment for 1 hour, close the box-type resistance furnace, until its natural cooling close to room temperature, to detect the hydrophobic and record data.

3. Experimental results and discussion

After the preliminary analysis, the surface hydrophobicity is related to the treatment of the substrate. The surface micro-nanometer rough structure is the prerequisite for hydrophobicity, followed by the proportion of various coating materials. In this paper, the heat treatment of the substrate after the measurement and analysis, obtained contact angle effect diagram, surface morphology (SEM), surface product XRD diagram, infrared spectroscopy, according to these image analysis experiments, draw the experimental conclusion.

3.1 Contact angle analysis

Contact angle measuring instrument is the use of optical contact angle measuring instrument to measure the contact angle of the sample, measured at room temperature, the water droplets used in the measurement are 10 μL, the contact angle of the five different positions of the substrate surface was measured, and the average value thereof was recorded as the detection result. Fig.1 is an example of the experimental hydrophobic effect of the image. In order to observe the convenience, the ink stained with small water droplets on the hydrophobic coating, and then line of sight with the water droplets and coating contact surface parallel observation of contact angle,this method can only roughly estimate the size of the hydrophobicity. Fig.1 is a better hydrophobic substrate; the hydrophobic angle can reach more than 150°.

Fig.2 is the angle of the hydrophobic film measured by the hydrophobic angle meter.

Fig.2(a) is the untreated hydrophobic angle of 70.5°;

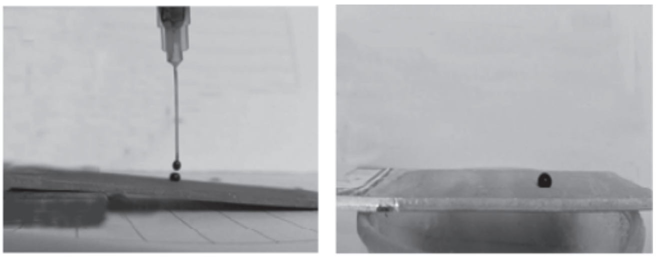

Fig.1 The real figure

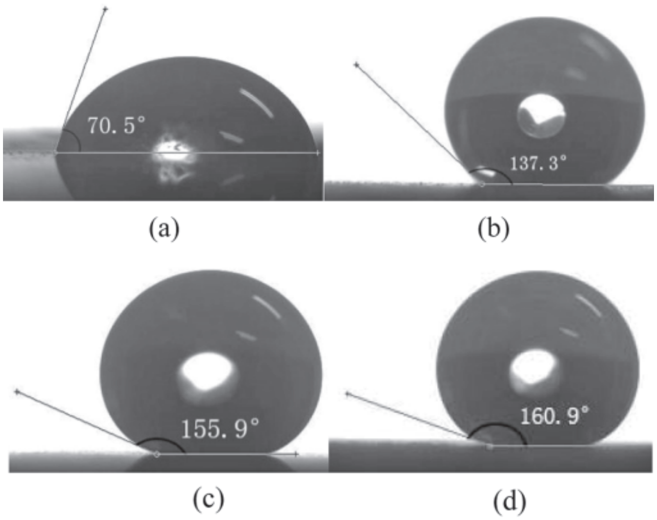

Fig.2 Hydrophobic angle diagram

Fig.2(b) is 15.3% hydrochloric acid etched hydrophobic angle of 137.3°; Fig.2(c) is the corresponding concentration of 10 mmol/L hydrophobic angle of 155.9°; Fig.2(d) is the corresponding concentration of 15 mmol/L of the hydrophobic angle of 160.9°.

3.2 SEM surface morphology analysis

Fig.3 is a scanning electron microscope to detect the hydrophobic coating on the aluminium surface structure, it can be seen from the figure after the aluminum surface of the test did form a rugged surface structure, the structure is very compact, the stack of layers, irregular arrangement, the surface structure of the structure to reach the level of microns.

Wherein, Fig.3(a) is 15% hydrochloric acid etching; SEM soaking solution concentration of 15 mmol/L image; Fig. 3(b) is 15% hydrochloric acid etching; SEM soaking solution concentration of 30 mmol/L image.

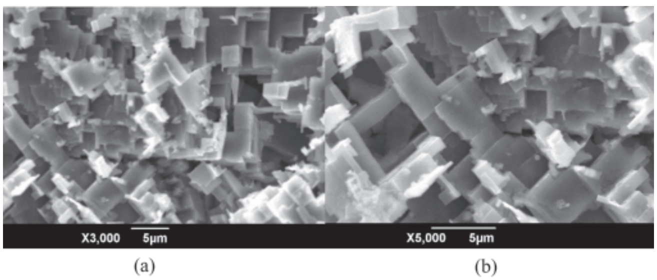

Fig.3 SEM scanning electron microscopy

3.3 Effect of surface roughness on super hydrophobicity

The early section of the paper mentioned that the surface roughness of the hydrophobic film has a great influence on the hydrophobic effect because the surface roughness is due to the reaction of the aluminium group in the dilute hydrochloric acid, so the mass fraction of hydrochloric acid is related to its roughness importance. Fig.4 is the relationship between the hydrophobic angle of the hydrophobic effect of aluminium in the dilute hydrochloric acid in the new 5%-25% system.

It can be seen from the figure that the aluminium sheet is put into the new 15% dilute hydrochloric acid etching reaction with the largest hydrophobic angle, indicating that the surface roughness at this time is ideal.

3.4 Effect of soaking solution concentration on superhydrophobic

According to the angle of the hydrophobic film measured by the concentration profile of the immersion liquid and the hydrophobic angle measurement instrument, the relationship between the corresponding concentration and the hydrophobic angle is obtained, as shown in Fig.5.

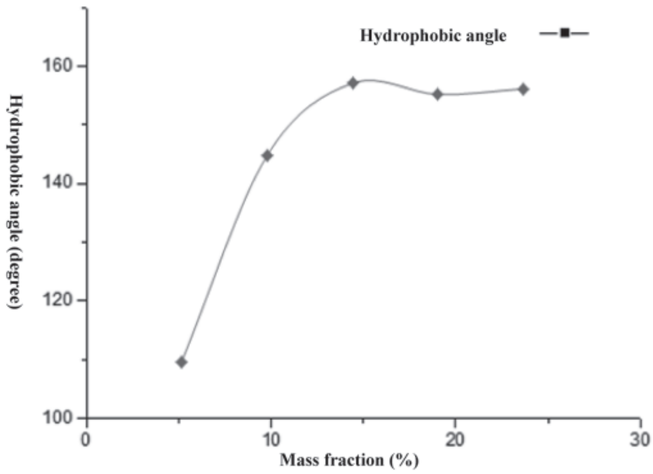

Fig.4 Effect of hydrochloric acid mass fraction on hydrophobic effect

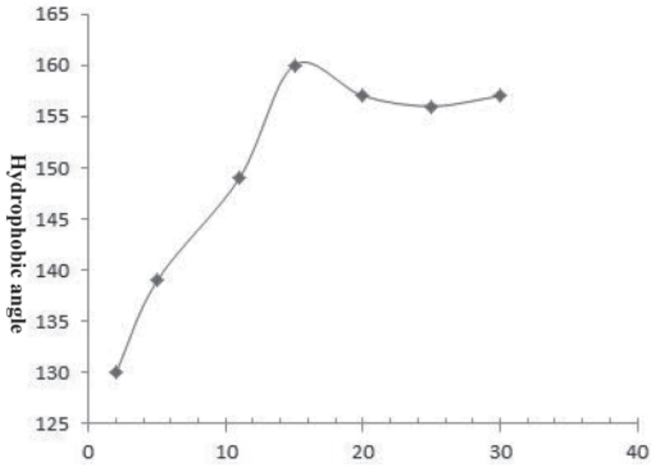

Fig.5 Effect of soaking solution concentration on hydrophobic effect

It can be seen from the figure that the aluminium sheet is put into the new 15% dilute hydrochloric acid etching reaction with the largest hydrophobic angle, indicating that the surface roughness at this time is ideal.

3.5 Experimental results

According to the above we can see: the physical polished pure aluminium in 15% dilute hydrochloric acid etching reaction structure micro-nano-level rough surface structure, and then in the soaking solution concentration of 15mmol / L group (that is, the quality of n-dodecyl alcohol is 0.3036g, the quality of tetradecanoic acid is 0.342g) with a trolley coating reaction, heat treatment temperature of about 120^{o}C, with a hydrophobic angle measuring instrument to measure the hydrophobic angle of 160.9°.

3.6 XRD analysis

Fig.6 shows the XRD pattern. As can be seen from the analysis of XRD patterns, there are titanium oxide, iron oxide, aluminium oxide and aluminium, and it can be determined that these mixed oxides are the main constituents of the hydrophobic structure. The key is iron oxide, only when the iron oxide content in a certain range will have a better hydrophobic effect.

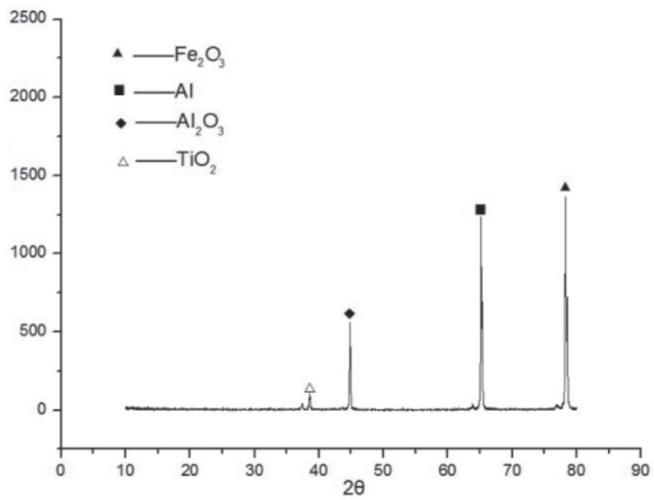

Fig.6 XRD pattern

4. Reaction equation

According to XRD and Fourier transform infrared spectroscopy, the following equation is written as follows:

The reaction equation of n-dithiol and roughened alumina:

$6CH_3(CH_2)_{10}CH_2SH + Al_2O_3 \rightarrow 2Al[CH_3(CH_2)_{10}CH_2S]_3 + 3H_2O$(1)

Determination of tetradecanoic acid with roughened alumina:

$6CH_3(CH_2)_{12}COOH + Al_2O_3 \rightarrow 2Al[CH_3(CH_2)12COO]_3 + 3H_2O$(2)

The adsorption capacity of n-dodecanethiol is lower than that of tetradecanoic acid, which makes the n-dodecyl mercaptan preferentially react with alumina and then react with tetrasodium acid and alumina to fill $Al[CH_3(CH_2)_{10}CH_2S]_3$ occupied by the alumina surface. $Al[CH_3(CH_2)_{12}COO]_3$ is on the membrane surface to improve the surface of the membrane micro-nano-structure of the compact, so the aluminium-based surface hydrophobic material is alumina roughened structure after the four acid, n-dodecyl thiol impregnated after the formation of substances:

$Al[CH_3 (CH_2)_{10}CH_2S]_3$ and $Al[CH_3(CH_2)_{12}COO]_3$.

5. Conclusions

In this paper, the fourteen hydrophobic aluminium films were successfully prepared. The experimental process is simple, the cost is low, and the operability is strong. It is significant for the industrialization and large-scale development of the super hydrophobic film. The hydrophobic effect of the hydrophobic film is better, but the durability and durability of the hydrophobic film need to be improved, and the surface is easy to be scratched.

Good surface stability, high strength and high hardness is also an important indicator of the hydrophobic film, is good or bad. So I guess you can prepare a good layer of hydrophobic coating and then add a layer of material or add a substance, provided that the damage has not yet formed Surface structure can enhance the basis of the hydrophobic surface strength, hardness, making wear resistant, can be stored for a long time use, which is the future direction of improvement.

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THEORETICAL ANALYSIS AND NUMERICAL SIMULATION OF DETONATION-DRIVEN DIRECTIONAL DISPERSING OF METAL PARTICLES

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5. Conclusions

The present study proposes a detonation-driven model to achieve directional dispersing of metal particles. Based on this model, an empirical formulas explaining the relationship between the dispersing velocity of particles and the mass ratio of explosive/particle is provided by using one-dimensional detonation wave theoretical analysis. Moreover, the three-dimensional finite element simulations of the detonation-driven particles dispersing under different mass ratio of explosive/particle are conducted and show good directionality, little scattering of the dispersal of metal particles. However, there is a comparative approximation in the results of theoretical analysis and numerical simulation, which indicates the availability of the detonation-driven model developed in this investigation.

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