

## A Preparation Method of Super-hydrophobic Coating Material and its Application

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### Abstract

Silver coating with nano-emulsion structure was prepared by electrolysis with AgNO<sub>3</sub> solution as electrolyte under certain conditions, followed by adding citric acid, sodium dodecyl sulfate and other substances. The coating material makes the substrate highly hydrophobic, and has strong anti-fouling ability and self-cleaning effect. The substrate was first treated by electrochemical deposition of chromium and nickel, and then silver plating was carried out by the above method. The water resistance and vapor barrier layer was further deposited on the surface of silver plating layer by atomic layer deposition. The super-hydrophobic material is provided with anti-corrosion protection by nickel plating layer and anti-fouling protection by silver plating layer. As the transition layer between the substrate and the nickel plating layer, the chromium plating layer can increase the binding force between the nickel plating layer and the non-metallic substrate, and the water vapor barrier layer can isolate air and water, further improving the corrosion resistance of the material. The test shows that the material has super-hydrophobicity, and the anti-corrosion and self-cleaning ability of the coating material also achieve the expected effect.

### Keywords

Super-hydrophobicity; Coating Material; Anticorrosion; Self-cleaning, Ocean.

### 1. Introduction

China has a vast ocean area and huge marine economic potential. With the acceleration of China's smart ocean construction process, the application of marine sensors, radar and other marine equipment is more and more frequent, and the pollution of marine equipment is becoming more and more prominent. According to incomplete statistics, China's annual direct economic losses caused by seawater corrosion and microbial pollution are nearly trillion yuan. Therefore, the tide, antifouling and anticorrosion of marine equipment has become one of the technical bottlenecks that seriously restrict the development of marine engineering and marine equipment.

During the working process of marine equipment, due to the high corrosion characteristics of seawater [11], the outer surface of marine equipment is vulnerable to corrosion damage, reducing service life and increasing maintenance costs; At the same time, marine microorganisms will adhere to the surface of marine equipment [12], accelerate the corrosion rate of the surface of marine equipment, and even change the performance of marine

equipment, resulting in distortion of monitoring data. It is difficult to comprehensively and objectively reflect the change of seawater quality [13], which is easy to cause greater economic losses. At present, it can not only prevent the corrosion of seawater on marine equipment, but also effectively prevent the attachment of marine fouling organisms. The most effective method is to brush anti-corrosion coating and antifouling coating on the shell of marine equipment. The anti-fouling agent added in the traditional anti-fouling coating is mainly heavy metals (such as copper, tin, etc.), which can poison and pollute organisms to prevent their attachment [15]. However, the released metal ions are difficult to decompose, seriously polluting the marine environment, and the timeliness of the coating is short [16], resulting in the increasingly serious marine environmental pollution [1-5], and the seawater quality has been damaged to varying degrees [6-8].

Green and environmentally friendly marine equipment protective coating is the basis of marine safety, marine management and marine development and utilization. In this paper, aiming at the environmental weather resistance of marine equipment, the preparation process and method of super-hydrophobic coating for waterproof, moisture-proof, anti-corrosion and anti-biological adhesion of marine equipment are developed based on ALD atomic layer deposition technology. The coating is composed of a papilla structure and a ALD high barrier film. It can be widely used in the environmental weather resistance treatment of metal components, marine sensors, radars and other marine observation / monitoring equipment and intelligent operation equipment surfaces, which effectively slows down the pollution rate of marine equipment, improves the service life of marine equipment and obtains the reliability of marine data.

## 2. Experimental Materials and Methods

### 2.1. Experimental reagents

Experimental reagents include :  $\text{AgNO}_3$  solution ( 0.25-0.50mol/L ), sodium hypophosphite (  $\text{NaH}_2\text{PO}_2$  ) ( 0.05-0.09mol/L ), citric acid ( 0.12-0.20mol/L ), sodium dodecyl sulfate ( SDS ) ( 2.0-3.0g/L ), 1,4-butanediol (  $\text{C}_4\text{H}_6\text{O}_2$  ) ( 0.15-0.20g/L ), sodium sulfate ( 0.52-0.60mol/L ), P salt complexing agent, aluminum oxide.

### 2.2. Preparation of super-hydrophobic coating material

Using  $\text{AgNO}_3$  solution (0.25-0.50mol/L concentration) as electrolyte, complexing agent is sodium hypophosphite ( $\text{NaH}_2\text{PO}_2$ ) (0.05-0.09mol/L concentration), PH = 5.2-5.5, current density is 10-15mA/cm<sup>2</sup>, electroplating time is 10min-30min, electrolysis temperature is 60-65 °C. Then citric acid concentration was 0.12-0.20mol/L, sodium dodecyl sulfate (SDS) concentration was 2.0-3.0g/L, 1,4-butanediol ( $\text{C}_4\text{H}_6\text{O}_2$ ) concentration was 0.15-0.20g/L, sodium sulfate concentration was 0.52-0.60mol/L.

In order to ensure the maximum experimental effect, the concentration and reaction conditions of each solution can be further optimized. The concentration of citric acid was controlled at 0.15-0.20 mol/L ; Sodium dodecyl sulfate ( SDS ) concentration was controlled at 2.5-2.8 g/L ; the concentration of 1,4 - butynediol (  $\text{C}_4\text{H}_6\text{O}_2$  ) was controlled at 0.18-0.20 g/L ; sodium sulfate concentration was controlled at 0.58-0.60 mol/L ; Electrolyte PH regulator preferred ammonia, so that the electrolyte PH = 5.2-5.5, as the preferred PH = 5.2 ; Saturated calomel electrode ( SCE ) as reference electrode, platinum electrode ( Pt ) as auxiliary electrode, the best electroplating time is 10min-30min, the best current density is 10-15mA/cm<sup>2</sup>, the best electrolysis temperature is 60-65 °C.

The addition of P salt complexing agent in the electrolysis process can control the appearance structure of the silver plating layer, so that the surface of the silver plating layer forms a regular and controllable papilla structure of the nanostructure. Then, the appearance structure of the

silver plating layer is controlled by adjusting the processing parameters, so that the surface of the silver plating layer can obtain a papilla with a diameter of 30-80 nm, the best diameter of 30-40 nm, a spacing of 80-100 nm, and a height of 20-30 nm. The shape of the papilla is the upper tip and the lower thick structure, which is similar to the cone shape (the distance between the papilla is the distance between the two adjacent nipple heights).

### 2.3. Application method of super-hydrophobic coating material

Firstly, a 200-300nm chromium coating was deposited on the surface of the treated substrate. The thickness of the chromium coating was  $220 \pm 10$ nm. Electrodeposition of nickel coating with thickness of  $200 \pm 50$  nm on chrome plating layer is preferred as the thickness of  $200 \pm 10$  nm ; The electrochemical deposition thickness of silver coating on the nickel coating was 20 – 500 nm, which was used as the preferred silver coating thickness of  $300 \pm 20$  nm. The coating structure is shown in Figure 1.

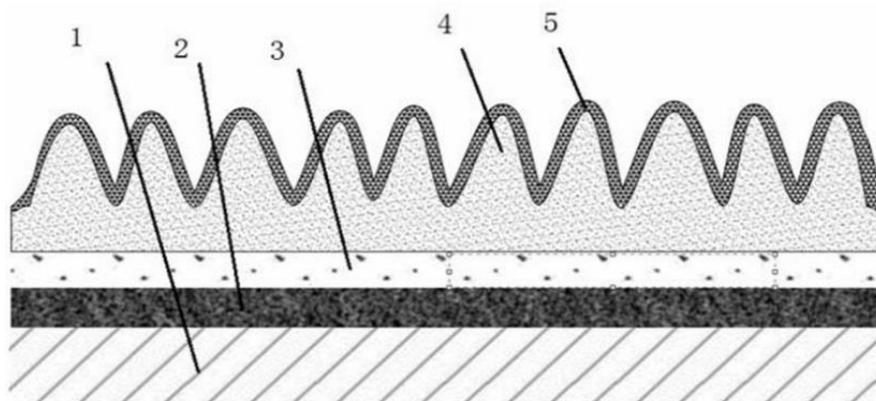


Figure 1. Diagram of section structure. (1 -Base material; 2-Chromium plating layer; 3-Ni plating layer; 4 - silver coating; 5-water vapor barrier layer)

Among the above coatings, the nickel coating [17] is the main anticorrosive coating of the substrate, and the chromium coating is the transition layer between the substrate and the nickel coating. The chromium coating can make the nickel coating combine well with the non-metallic substrate, prevent the falling off due to the poor adhesion between the nickel coating and the non-metallic substrate [18], and make the material have a lasting anti-corrosion performance. In addition, the chromium coating can also adjust the difference in the thermal expansion coefficient between the substrate and the nickel coating [19], which greatly expands the material selection types of the substrate. The nickel coating as the anticorrosive coating can be firmly plated on the surface of the metal substrate, and the nickel coating can be firmly plated on the surface of the non-metallic substrate, especially on the surface of the polymer material [20]. The silver coating outside the nickel coating can make the material have good anti-fouling performance. The silver coating can be controlled through the silver salt plating process [21]. The super-hydrophobic material is provided with anti-corrosion protection by nickel coating and anti-fouling protection by silver coating.

The surface atomic vapor deposition [22] (ALD) on the silver coating obtained by the above method has a thickness of 10-100 nm of water vapor barrier layer. Water vapor barrier layer can be  $\text{TiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{HfO}_2$ ,  $\text{ZrO}_2$ ,  $\text{ZnO}$ ,  $\text{V}_2\text{O}_5$  in one or more than two metal oxide deposition layer [22], priority for aluminum oxide deposition layer, as the preferred aluminum oxide is preferred for a crystal form of aluminum oxide [23], the thickness of the water vapor barrier layer is preferred for  $30 \pm 5$ nm.

Finally, ALD was used to rapidly deposit a layer of water and vapor barrier layer, which completely covered the surface structure of the silver coating without damaging the

nanostructure of the silver coating. In the process of water and vapor barrier layer deposition, a smaller nano-scale bump structure will be formed. The diameter of the bump is 3-5 nm, the spacing is 8-10 nm, and the height is 3-5 nm, which makes the marine fouling organisms more difficult to adhere to the protective layer of the sensor or the adhesion is not strong and easy to be washed by water flow to achieve the effect of self-cleaning [24], so as to achieve the purpose of preventing marine biological pollution and achieve complete environmental friendliness. Aluminium oxide atomic layer can also provide airtight protection for the material [25] to isolate air and water and further improve the corrosion resistance of the material.

**2.4. Performance and characterization**

Due to the addition of P salt complexing agent in the process of silver plating and proper process control, the silver plating layer with 50-80 nm in diameter, 80-100 nm in spacing and 20-30 nm in height can be obtained. The static contact angle between the material surface and water is more than 150 °, which makes the material highly hydrophobic and the rolling angle of water can be 4 ± 1.2 °. Therefore, if it is plated on the surface of the substrate such as the sensor, the surface of the substrate such as the sensor will have strong anti-fouling ability and self-cleaning effect. In addition, the super-hydrophobic material obtained by this method will form a nano-small bump structure in the process of water-blocking and vapor-blocking layer deposition. The diameter of the small bump is 3 – 5 nm, the spacing is 8 – 10 nm, and the height is 3 – 5 nm, so that the material surface has a barrier effect on water, and the water flows on the material surface. The effect is shown in Figure 2.

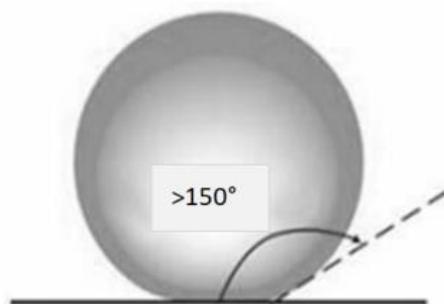


Figure 2. Hydrophobic principle of materials

Table 1. mastoid size under different conditions

Item parameters	Number of examples				
	1	2	3	4	5
Thickness of Chromium Coating	200±50	220±50	230±50	210±30	220±10
Nickel plate thickness	200±20	200±20	200±20	200±20	200±50
Thickness of silver plating	300±50	400±50	300±50	300±50	200-300
Aluminium oxide	40±30	50±30	60±30	60±30	50
The concentration of silver nitrate solution (mol/L)	0.25	0.30	0.35	0.40	0.25
Sodium phosphate( NaH <sub>2</sub> PO <sub>2</sub> ) concentration (mol/L)	0.05	0.07	0.08	0.09	0.06
Citric acid concentration (mol/L)	0.18	0.18	0.12	0.18	0.12
Sodium dodecyl sulfate concentration (g/L)	2.0	2.6	3.0	2.6	2.5
1,4 - Butynediol concentration (g/L)	0.18	0.18	0.20	0.18	0.15
Sodium sulfate concentration (mol/L)	0.58	0.60	0.58	0.58	0.52
Electrolyte PH	5.2	5.5	5.3	5.2	5.2
Plating time (min)	100	30	20	30	20
Current density (mA/cm <sup>2</sup> )	12	12	15	15	10
Electrolysis temperature (°C)	60	62	60	63	65
Mastoid diameter(nm)	30-80	50-70	50-80	30-80	30-80

### 3. Results and their applications

#### 3.1. Effect of different experimental conditions on mastoid size

The papilla size has a great influence on the hydrophobicity of the substrate, and the papilla sizes obtained under different reaction conditions and process conditions are also different. Therefore, in order to achieve better hydrophobic effect and antifouling self-cleaning ability, it is necessary to verify the size of papilla formed under different conditions and the influence of these sizes on hydrophobicity. Detailed experimental data are shown in table 1.

#### 3.2. Self-cleaning ability of super-hydrophobic coating materials

The diameter of the mastoid on the surface of silver-plated material is 30-80nm. The water and vapor barrier layer is deposited on the surface of silver-plated layer. The water and vapor barrier layer will form nano-convex structure during the deposition process, and the diameter is 3-5nm. In this case, the liquid cannot infiltrate into the surface of the coating [26], and flow or sliding will occur on the surface of the coating. Under the water drop angle tester, the water drop angle of the liquid dropped on the prepared coating material was greater than 150 degrees, and the overall shape was round, which proved the super-hydrophobicity of the coating material [27]. The self-cleaning ability of the material is directly related to the surface roughness and hydrophobicity of the material. The adhesion of impurities or microorganisms in the external environment requires sufficient roughness and wettability on the surface of the object. The coating material can prevent the immersion of liquid [28], and the surface of the coating remains dry. At the same time, the material also has a very low roughness, which ensures the anti-biological adhesion of the coating [29].

#### 3.3. Application of Super-hydrophobic Materials in Ocean

The corrosion damage of marine environment to marine monitoring equipment is very serious [30], including marine atmospheric environment, seawater, marine organisms and so on, which will make the marine equipment aging failure rapidly. The key to anticorrosion is hydrophobicity [31]. It has been proved by experiments that this super-hydrophobic material can be used for underwater anti-corrosion and anti-pollution of various instruments and instruments, such as marine sensors, marine detectors, and water flow detectors. The preparation method of the coating can process the shell of various instruments and instruments to obtain anti-corrosion and anti-pollution instruments, and can also process the components of the instrument to make the components have super-hydrophobic properties. The processed marine equipment makes it more difficult for marine organisms to attach to the protective layer of the sensor or not firmly attached to it, which is easy to be washed by water, so as to achieve the role of self-cleaning, so as to achieve the purpose of preventing marine biological pollution and achieve complete environmental friendliness. Aluminium oxide atomic layer can also provide airtight protection for the material [33] to isolate air and water and further improve the corrosion resistance of the material.

### 4. Conclusion

The super-hydrophobic material is excellent on the surface of metal substrate and has good anti-fouling effect for metal materials. Due to the existence of chromium coating, the thermal expansion coefficients of substrate and nickel coating can be adjusted, which greatly expands the selection types of substrate materials on the surface of marine equipment. Therefore, the coating material is also feasible for anti-fouling of non-metallic materials such as polymer [34] substrate. The coating material is used to treat the shell of the marine sensor. Under the condition of meeting the mechanical properties and sealing requirements of the marine sensor, the problems of seawater corrosion and marine biological pollution of the sensor in the ocean

are solved, the material selection range of the sensor substrate is widened and the economic cost of the sensor is reduced. At the same time, the preparation process of the coating material is controllable, and the corresponding process parameters can be adjusted to adapt to different requirements of the use environment, without any pollution to the marine environment. The static contact angle between the whole coating and water is  $> 150^\circ$ , which has high hydrophobicity and self-cleaning effect, and solves the pollution problem of marine organisms on the sensor. The method has good sealing property and has nothing to do with the size and shape of the substrate. The wear resistance of the coating is excellent.

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