

# Characteristics analysis of the developed surface modification technologies to improve the anti-corrosion performances for offshore equipments<sup>†</sup>

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## Abstract

In this study, we carried out the characteristics analysis and experiment research of the developed technologies based on surface modifications to enhance the anti-corrosion performance of offshore equipments. The developed technologies are the dipping method to generate super-hydrophilic surface which is then treated to be super-hydrophobic using chemical deposition and lubricant impregnation, and the spray coating method to generate super-hydrophobic surface. It is well-known that the super-hydrophilic and super-hydrophobic surfaces have good anti-corrosion performance. However, the mechanical properties should be maintained. We have verified the anti-corrosion performance through the salt spray test and the measuring contact angle. And, we have evaluated the mechanical characteristics such as hardness. As the results, the dipping and the spray coating methods improved the anti-corrosion performance maintaining its mechanical properties at the same coating thickness of about 80 nm.

**Keywords:** Offshore equipment; Anti-corrosion; Surface modification technology; Mechanical properties evaluation; Corrosion resistance evaluation

## 1. Introduction

The equipments used in offshore industries are mostly composed of metal materials such as aluminum, steel, titanium and etc. The offshore equipments always are exposed to the risk of corrosion and fouling because they are working at seawater and under marine atmospheric environment. The corrosion and fouling cause severe damages to offshore equipments such as fatigue destruction of equipment due to the material durability degradation. In addition, it can cause various problems including large accident such as plumbing explosion and increase of maintenance cost [1-3]. Therefore, it is very important to identify the causes of corrosion and to prevent it in the design and maintenance steps of offshore equipments.

In general, many studies have identified that the main causes of marine corrosion are salt and fouling organisms [4-11]. Baek [5] proved that the corrosion level depends on NaCl concentration, temperature, dissolved oxygen and dissolved gas in seawater. Especially, his results show that the higher the salinity of the marine atmosphere, the greater the corrosion

rate. Park et al. [7] found that structure stability problems of ships and offshore equipments are caused by marine biofouling on their surfaces.

In the shipbuilding and offshore industries, Tri-Butyl-Tin Self-Polishing Copolymer (TBT-SPC) anti-fouling coating was widely used to prevent corrosion of offshore equipments because of its excellent anti-fouling performance and easy re-coating. However, several studies found that a toxic effect of TBT compounds causes ecosystem disturbance and severe environmental destruction [12, 13]. So, the International Maritime Organization (IMO) banned the use of TBT compounds through the international convention on the control of harmful anti-fouling system on ships in 2001. For this reason, researches on environment-friendly anti-fouling paints and anti-fouling technologies have been actively conducted recently [14]. Especially, there is a log of ongoing research on technology to block causes of corrosion using by a material surface modification [15, 16].

There is an active method and a passive method as modification technologies of the material surface. The active method is that an air layer or an air droplet is injected between the surface and seawater to reduce the direct contact area with water to block the cause of corrosion [17]. The passive method is that the surface is modified to a hydrophobic surface

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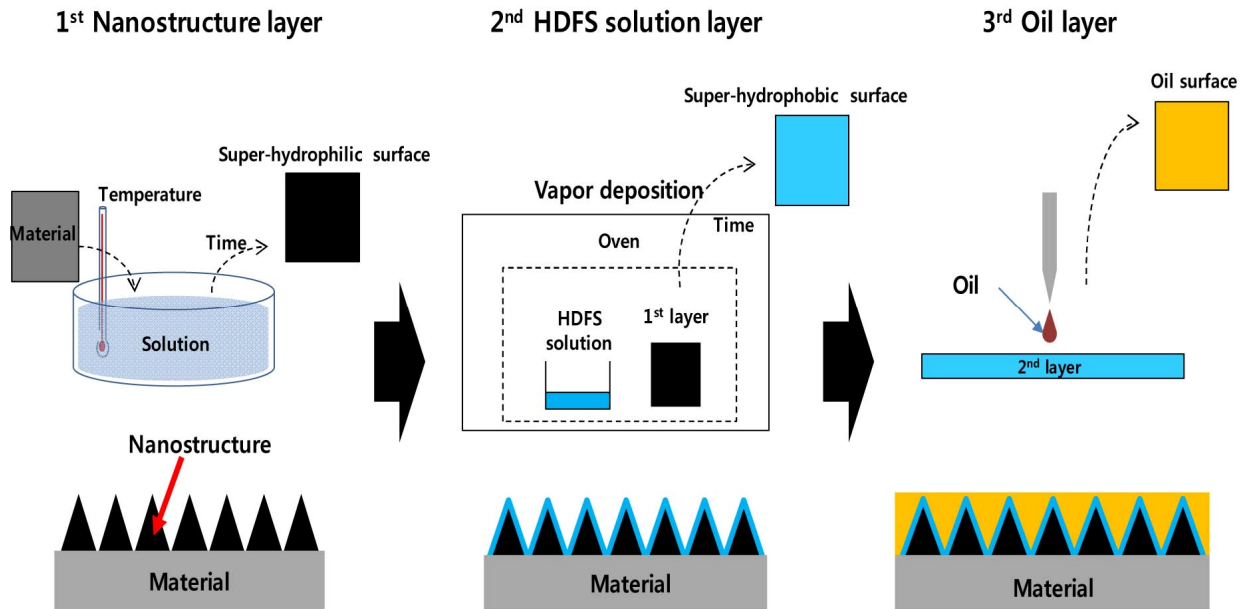


Fig. 1. Process of developed dipping method.

have a property to dislike water [18].

In Korea, the research team of KRISO and Kyung Hee University have developed two surface modification technologies as eco-friendly surface control technology to improve anti-corrosion performance of offshore equipments. One is a dipping method that applies super-hydrophobic coating and oil to reduce surface energy after producing nano-metal oxide on the surface of material [18]. Another is a spray coating method that directly injects nanoparticles onto the surface to make it super-hydrophobic [19]. Several previous studies have shown that the nano-metal oxides have low toxicity. Therefore, these technologies are available in the ocean industries, because it is eco-friendly compared to other anti-corrosion technologies [20, 21].

These surface modification technologies should improve the anti-corrosion performance without changing the mechanical properties of the material. Therefore, it is necessary to verify whether the developed dipping method and spray coating method meet the requirements of surface modification technologies. In this study, test evaluations were conducted to verify the developed surface modification. The material used in the test is 6061-T6 aluminum alloy which is widely used in offshore equipments. The aluminum alloy is used for core equipments such as HPU and control panel among offshore equipments because it is resistant to external impacts and has good thermal conductivity to transfer heat internally.

The test evaluation methods are as follows. First, the dipping and spray coating methods were applied to aluminum specimens. Second, the thickness of the coating layer, the hardness of the surface, and the thermal conductivity were measured to analyze the changes of mechanical properties. Third, the neutral salt spray test and measurement of the contact angle were performed to evaluate anti-corrosion perform-

ance. This paper concerns the evaluation of mechanical properties, anti-corrosion performance of 6061-T6 aluminum alloy with surface modification technologies.

## 2. Development of surface modification technologies

### 2.1 Dipping method

Surface modification is the act of modifying the surface of material by physically or chemically treatment. Surface modification by dipping method can apply to easy various orientation and size of substrates. It was carried out by exposing the sample to reagents including chemical solution. In this paper, wet chemically oxidation scheme and oil infusing by dipping was applied to aluminum surface as shown in Fig. 1 [18, 22, 23].

The investigated aluminum surfaces were prepared as follows. We use an available aluminum substrate (15 mm × 15 mm × 0.1 mm).

Each surface was rinsed with acetone, ethanol, isopropanol and de-ionized (DI) water to wash. After then, the substrate was dipped into HCl solution (4M) about 30 seconds to remove native oxides on the surface then triple-rinsed with DI water.

For nanostructuring, boiling water was applied and oxidation scheme was applied as follows [24, 25]. The substrates were dipped into boiling DI water for 30 min and then rinsed with DI water followed by N<sub>2</sub> blow. After the oxidation processes, oxide structures with super-hydrophilic characteristic were formed on aluminum surfaces.

To turn this surface into super-hydrophobic surface, HDFS solution was prepared by mixing 1H,1H,2H,2H-Perfluorodecyltrimethoxy silane (CAS No.101947-16-4, Sigma-Aldrich) of 0.1 ml with toluene of 2 ml [26, 27]. Then HDFS solution was put in a small dish and placed within a sealed

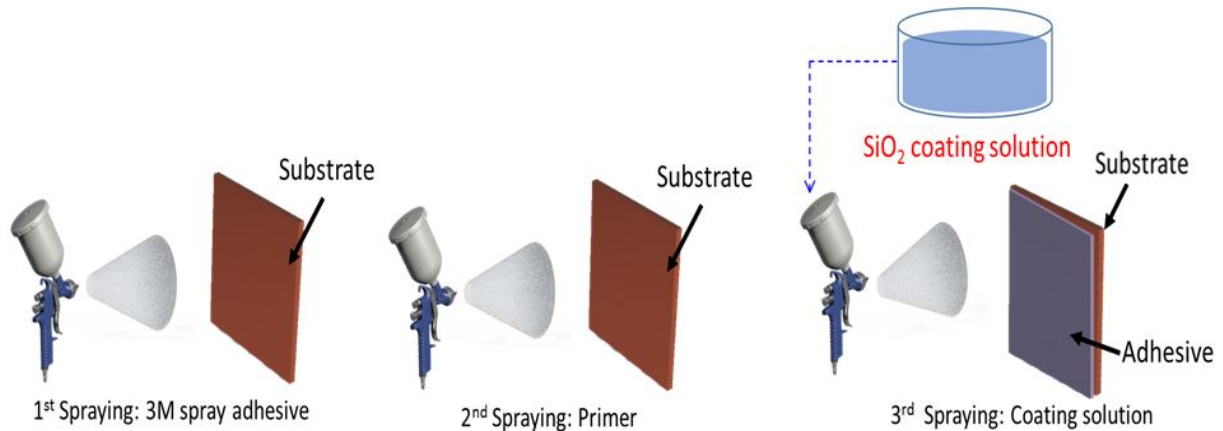


Fig. 2. Process of developed spray coating method.

container. The prepared nanostructured aluminum sample was placed into the same container with the HDFS solution to induce HDFS vapor deposition on the samples. The container was heated at 80°C for 3 hours, then the samples were taken out from the oven and cooled down to room temperature in a fume hood. The sample was gently rinsed with ethanol followed by N<sub>2</sub> blow.

For lubricant impregnated surface, HDFS coated super-hydrophobic surface was dipped into silicone oil with a viscosity of 100cSt [28]. Then, the oil layer was uniformed by N<sub>2</sub> blow and holding the sample vertically overnight.

## 2.2 Spray coating method

Spray coating is a deposition technique where spraying equipment sprays particles or solution through the air onto the substrate [29]. The equipment of spray coating is composed of nozzle, solution container and compressor, etc. In spraying process, the droplet of coating solution is atomized by nozzle and compressed air. The atomized droplets are attached at the substrate. The characteristics of coating are determined by properties (size of droplet, velocity of droplet, etc.).

Spray coating procedure to the super-hydrophobic surface is as in the following [19, 30]. The coating solutions were prepared by mixing solution A and solution B. The solution A is made by 1g of Poly (vinylidene fluoride) (CAS No.24937-79-9, Sigma-Aldrich) in 19 g of N, N-Dimethylformamide (CAS No.68-12-2, Sigma-Aldrich). To completely dissolve Poly (vinylidene fluoride), the solution A is heated at 50 °C for about 3 hours. The solution B was prepared by sonicating 2.5 g of SiO<sub>2</sub> nanoparticles (CAS No.68909-20-6, ABCR GmbH & Co.) in 19 g of Toluene (CAS No.108-88-3, Sigma-Aldrich) for about 15 minutes. After mixing solution A with B, the coating solutions were stirred by magnetic bar for minimum 1 hour. The prepared surfaces were sprayed by adhesive, primer and coating solution with a distance of 20 cm away from the nozzle and substrate and a pressure ~20 kPa as shown in Fig. 2. Then, the fabricated surface was completely dried at room temperature for about 3 hours.

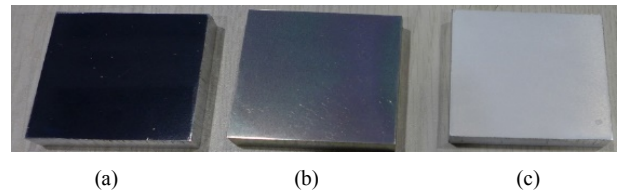


Fig. 3. Aluminum specimens: (a) Pure specimen; (b) dipping specimen using dipping technology; (c) spray specimen using spray coating technology.

## 3. Mechanical properties evaluation

The surface modification technologies should not only change the mechanical properties of the material but also affect anti-corrosion performance. So, the influence of the developed surface modification technologies on the mechanical properties of the material was verified by several mechanical properties measurement tests.

Specimens used in the tests are shown in Fig. 3. The prepared specimens were pure 6061-T6 aluminum alloy specimens without the surface modification technologies, dipping specimens applying the dipping method, and spray specimens applying the spray coating method. After the specimens were manufactured, the mechanical properties variation by the surface modification technologies were observed by measuring the thickness of the surface coating layer, the hardness of the surface layer and the thermal conductivity of the material, which are advantages of the aluminum material.

The specimens for each test were prepared according to the size and test specifications of the test equipment as shown in Table 1.

### 3.1 Thickness variation by coating layer

Thin film thickness meter (SR100 made by AST) which can measure from 2 nm to 5 μm (error rate: 0.25 %) was used to observe thickness variations by coating layers. The thickness variations were measured based on the pure specimen thickness measured by the offset and reinforcement for each wave-

Table 1. Al6061 specimen size for mechanical properties measurement.

Measurement items	Al6061 specimen size		
	Width [mm]	Height [mm]	Thickness [mm]
Film thickness	100.0	100.0	1.0
Hardness	50.0	50.0	10.0
Thermal conductivity	10.0 (2 ea)	10.0 (2 ea)	1.0 (2 ea)
	3.0 (1 ea)	3.0 (1 ea)	1.0 (1 ea)

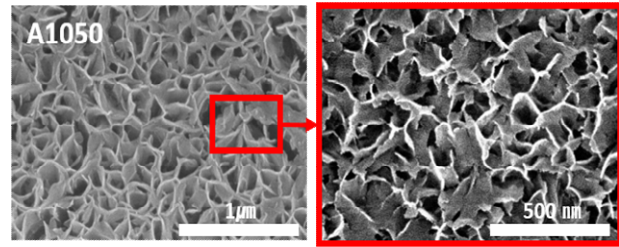
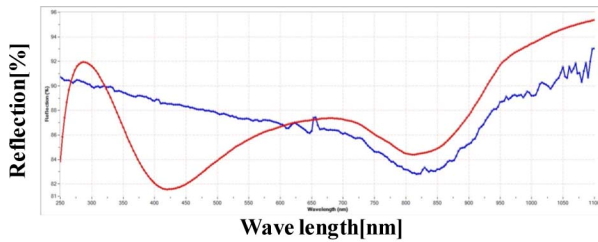
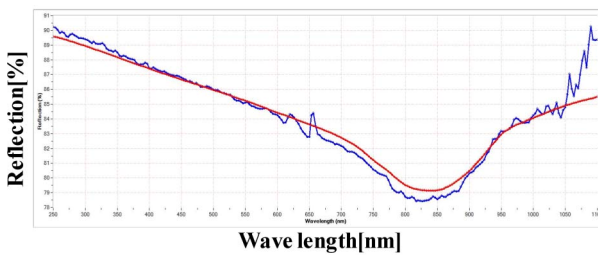


Fig. 5. FE-SEM images of the oxidation scheme applying aluminum alloy [18].



(a)



(b)

Fig. 4. Reflection data by wave length: (a) Dipping specimen; (b) spray specimen.

length using the interference of incident light and reflected light on the surface of the coating layer [31].

Fig. 4 shows the reflection data by each wavelength as the thickness measurement results of the dipping and spray specimens. In Fig. 4, the blue lines are the surface reflections of the base material as a reference data and the red lines are the surface reflections of the dipping and spray specimens. Fig. 4(a) shows that the reflection patterns of the dipping specimen are not constant depending on the wavelength. However, Fig. 4(b) shows that the spray specimen has similar reflection patterns by wavelength on the surface of the pure material and coating layer.

The reason why the reflection patterns are not constant in the dipping specimen is that the material surface of the dipping specimen was modified to multi-layers with nanostructure surface (shown in Fig. 5), HDFS solution, and oil. Since the surface was not uniform, the thickness of the dipping specimen was measured at five points on the surface of the specimen and the mean value was derived as the coating layer thickness.

The measured thicknesses of the coating layer were 77.1 nm for the dipping specimen and 79.4 nm for the spray specimen. As the measured results, the coating thicknesses of the

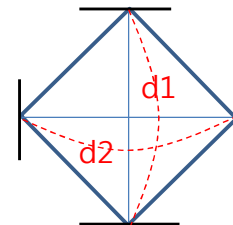


Fig. 6. Normal pressure mark on specimen surface by Vicker's hardness measurement.

dipping and spray specimens were almost the same. Both specimens were about 80 nm thickness and were very thinly coated on the base material. The thickness of the coating layer does not affect the analysis of other mechanical characteristics because it is coated with similar thickness.

### 3.2 Hardness variation by coating layer

A Vickers hardness measurement was performed in accordance with KS B 0811 [32] to determine the hardness variation of the material by the coating layer. The Vickers hardness tester made by i-Solution (error rate: 7.0 %) calculates the hardness through measuring the indentation strength of the pure, dipping and spray specimens. When a load is applied to a pyramid-shaped diamond indentation rod attached to a hardness measuring instrument, indentation marks are created on the specimen surface as shown in Fig. 6. The hardness of the material is calculated using the lengths of d1 and d2 in the indentation marks and the indentation load. The formula is as follows.

$$Hv = \frac{1.854 \times P}{d1 \times d2} \tag{1}$$

where Hv is a hardness of material, P is a pressure load, and d1 and d2 are cross length

The original hardness of aluminum material was 107 Hv and the hardness of the pure specimen was measured as 106.2 Hv by Vickers hardness tester. The measurement results are reasonable because the error rate of the Vickers hardness tester is 7.0 %. The hardness of the dipping specimen was measured as 108.6 Hv and the hardness of the spray specimen was 114.0 Hv. The surface modified specimens had slightly higher hardness value than the hardness of the pure specimen.



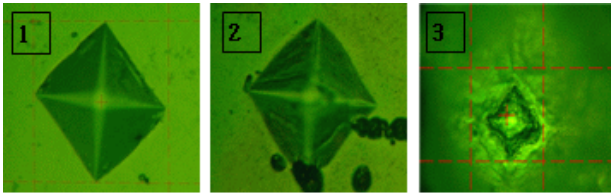


Fig. 7. Measured pressure marks of test specimens: (1) Pure specimen; (2) dipping specimen; (3) spray specimen.

Fig. 7 shows the pressure marks created on the specimen surface by the indentation rod of the Vickers hardness tester. The pure specimen had a normal pressure mark. However, the dipping and spray specimens had abnormal pressure marks. Although hardness could be measured using the abnormal pressure mark, the measured hardness would have errors. The reason why the dipping and spray specimens have higher hardness than the pure specimen is the error by this abnormal pressure marks. But, it is almost the same hardness value considering the equipment error rate because the difference between the measured results is within 7.0 %.

It was confirmed that hardness of pure, dipping and spray specimens were not significantly different through this experiment. This result indicates that the hardness of the material was not changed by the surface modification technologies.

### 3.3 Thermal conductivity variation by coating layer

A thermal conductivity is calculated by the measured density, specific heat and thermal diffusivity of the material [33]. The calculation formula is as follows. The thermal conductivity is a mechanical property which is changed by temperature as Eq. (2). Therefore, the test temperature was defined as room temperature of 25 °C.

$$\lambda(T) = \rho(T) \times C_p(T) \times \alpha(T) \tag{2}$$

where  $\lambda$  is a thermal conductivity,  $\rho$  is a density of material,  $C_p$  is a specific heat, and  $\alpha$  is a thermal diffusivity.

Densities, specific heats, and thermal diffusivities of pure, dipping and spray specimens were measured to evaluate the thermal conductivity variation of the material by the coating layer. The density was measured according to ASTM D 792-13 [34]. The specific heat was measured using a flash method [35]. The thermal diffusivity was measured according to ASTM E1461-13 [36].

The calculated results of the thermal conductivity are shown in Table 2. The pure specimen had a thermal conductivity value of 148 W/m·k, and the dipping and spray specimens had similar thermal conductivity values of about 140 W/m·k.

There were little difference in the density of specimens, but there were some difference in specific heat and thermal diffusivity of specimens. As a result, differences in thermal conductivity were occurred due to differences in specific heat and thermal diffusivity. However, the differences of thermal con-

Table 2. Thermal conductivity measurement results.

Specimen items	Pure	Dipping	Spray
Density [g/cm <sup>3</sup> ]	2.682	2.655	2.625
Specific heat [J/g·K]	0.840	0.816	0.879
Thermal diffusivity [mm <sup>2</sup> /s]	66.183	63.65	60.835
Thermal conductivity [W/m·K]	148	140	141

Table 3. Al6061 specimen size for anti-corrosion performance tests.

Test items	Al6061 specimen size		
	Width [mm]	Height [mm]	Thickness [mm]
Salt spray test	60.0	80.0	1.0
Contact angle measurement	60.0	80.0	1.0

ductivity exist within the error because the error rate of the measuring equipment was more than 4 %. Therefore, the thermal conductivities of pure, dipping and spray specimens are not significantly different. This result indicates that the thermal conductivity of the material was not changed by the surface modification technologies.

## 4. Anti-corrosion performance evaluation




We have confirmed that the mechanical properties of the base material were not changed by the developed surface modification technologies. Even if the mechanical properties of the base material are not changed, the developed technique is meaningless if the anti-corrosion performance of the base material is not improved. Therefore, the anti-corrosion performance of the developed technologies was evaluated to verify the value of the developed surface modification technologies.

The specimens for each test were prepared according to the size of the test equipment and test specifications as shown in Table 3.

### 4.1 Anti-corrosion performance by salt spray test

An anti-corrosion performance by salty can be evaluated by salt spray test. The salt spray test which is a kind of acceleration test was used to evaluate the anti-corrosion performance of the surface modification technologies. The salt spray test accelerates corrosion by directly spraying a high concentration of salt water on the material. The salt spray test method used in this experiment is a neutral salt spray test according to KS D 9502 [37]. The neutral salt spray test is a common test

Table 4. Salt spray test results of pure, dipping, and spray specimens.

Specimen items	Corrosion area rate [%]	Rating number	
Pure	10	3	
Dipping	0.25	8	
Spray	0.05	9.5	


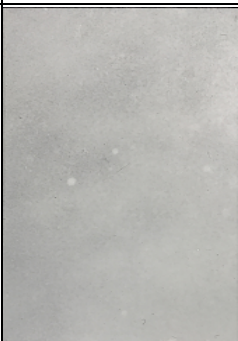
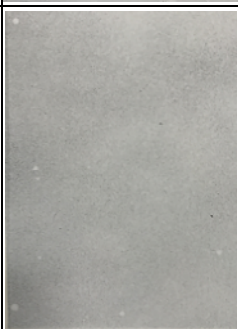
method to evaluate the anti-corrosion of equipments in the offshore industries.

The degree of corrosion is evaluated by visually inspecting specimens that have been corroded by salt spray test. The evaluation method is to derive a rating number that represents the degree of corrosion. The rating number is defined from 1 to 10, and the number of 10 means non-corrosion.

The salt spray test was conducted continuously for 72 hours using sodium chloride solution having hydrogen exponent (pH) of 6.5. After the salt spray test, a sample area of 50 mm by 100 mm on the specimen was selected to determine the degree of corrosion and the rating number was derived from the comparison with the standard figures in KS D 9502.

The rating numbers of each specimen are shown in Table 4. The rating number of the pure specimen was 3 with corrosion area rate of 10.0 %. The rating number of the dipping specimen was 8 with corrosion area rate of 0.25 %, and the rating number of the spray specimen was 9.5 with little corrosion.

Table 5. Salt spray test results of spray, commercial paint, and mixture specimens.

Specimen items	Corrosion area rate [%]	Rating number	
Spray	0.05	9.5	
Commercial paint	0.05	9.5	
Mixture (Commercial paint + spray coating)	0.02	9.8	

As the results of the salt spray test, it was confirmed that the corrosion rates of the material were drastically reduced due to the surface modification technologies. This means that the developed surface modification technologies have excellent anti-corrosion performance. Among the developed technologies, the spray coating method had the best corrosion resistance.

Also, we confirmed that the spray coating method had similar performance with commercial anti-corrosion paint. Table 5 shows the results of the salt spray test of the specimens with the spray coating, the commercial paint, and mixture the commercial paint and the spray coating. The rating numbers of the spray and commercial paint specimens were 9.5. The specimen mixed with commercial paint and spray coating technique had the rating number of 9.8.

The developed spray coating method has proven to be as successful technique comparable to anti-corrosion technique

Table 6. Contact angle measurement results before salt spray test.

Specimen items	Static contact angle [degree]	Advancing contact angle [degree]	Receding contact angle [degree]
Pure	114.1	123.4	70.0
Dipping	150.3	158.1	148.1
Spray	159.3	161.0	158.1

Table 7. Contact angle measurement results after salt spray test.

Specimen items	Static contact angle [degree]	Advancing contact angle [degree]	Receding contact angle [degree]
Pure	-	-	-
Dipping	81.9	78.4	52.7
Spray	136.3	147.1	42.3

using commercial paint. In addition, we have found that a combination of commercial paint and the developed spray coating method has better corrosion resistance.

**4.2 Anti-corrosion performance by measurement of contact angle on surfaces**

An anti-corrosion performance by organic matter can be evaluated by measuring the degree of bonding between the organic matter and the material by cultivating the organic matter on the surface, or by measuring the contact angle of the surface which can confirm whether the surface is super-hydrophilic or super-hydrophobic that the organic matter cannot adhere to Refs. [38, 39]. In this study, the contact angle measurement method was used to evaluate the anti-corrosion performance of the surface modification technologies.

In general, the super-hydrophilic surface has static contact angle of 90° or less, and the hydrophobic surface has static contact angle of 90° or more. The super-hydrophobic surface has characteristics of a static contact angle of more than 150°, and a difference between an advancing contact angle and a receding contact angle of less than 10° [40]. In this experiment, the contact angles before and after the salt spray test were measured to verify the anti-corrosion performance and confirm whether the modified surface remains intact or not.

Table 6 shows the contact angle of the surface before salty-based corrosion by the surface modification technologies. The pure specimen had hydrophobic surface because the static contact angle is 114°. The dipping and spray specimens had super-hydrophobic surface because the static contact angles are more than 150° and the differences between the advancing contact angle and the receding contact angle are less than 10°. This result implies that the developed surface modification technologies had normally modified the surface of the base material to the super-hydrophobic surface which has the capacity for corrosion resistance.

Table 7 shows the contact angle of the surface after salty-

based corrosion by the surface modification technologies. The static contact angles of the dipping and spray specimens were about 80° and 140°. The surface of the dipping specimen did not maintain super-hydrophobic because the oil and HDFS layer on the surface were damaged. However, the spray specimen surface maintained excellent hydrophobic even though it was not super-hydrophobic.

Through this experiment, it was confirmed that the developed surface modification technologies has the good anti-corrosion performance. However, the dipping technique had a durability problem.

**5. Conclusions**

In this study, we developed the surface modification technologies. One is the dipping method that creates super-hydrophilic nanostructures on the material surface and applies HDFS & oil on the nanostructures. Another is the spray coating method that injects nanoparticles on the material surface. And we carried out the characteristics analysis and experiment research of the developed technologies.

These surface modification technologies must maintain the mechanical properties of the material and improve only the anti-corrosion performance. We applied the developed surface modification technology to 6061-T6 aluminum alloy to evaluate whether the developed technologies have these characteristics. Mechanical properties and anti-corrosion performance tests were conducted to evaluate the developed technologies.

As the results of the tests, the dipping method and the spray coating method maintained mechanical properties of the base material at the same coating thickness of about 80 nm. The anti-corrosion performance was improved compared to the base material. Especially, the spray coating method had performances comparable to the commercial anti-corrosion paint. However, the dipping method was not maintained the performances because the super-hydrophobic coating layer had the partial damage by the salt spray test.

The coating layer generated by dipping method was bonded by the bond strength between the nanostructure and the HDFS solution. This bond strength is lower than the bond strength of the adhesive used spray coating method. The coating layer of the dipping specimen was damaged by the injection water of the salt spray test instrument due to the weak bond strength. Therefore, the currently developed dipping method is required further research to enhance its bond strength.

In the future, we will improve the durability of the dipping method which is a problem found in this study, and verify the performance of the developed technologies through real-sea test.

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