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Comparison of electrochemical properties of atmospheric pressure plasma coatings for Al₂O₃-3TiO₂ and CoNiCrAlY in sea water

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Abstract: To improve the durability of underwater rotating products, the corrosion characteristics in harsh marine environment were evaluated through various electrochemical experiments on the Al_2O_3 -3TiO₂ and CoNiCrAlY coating layers by atmospheric pressure plasma spray coating process. By evaluating the corrosion resistance of these materials, their applicability to environmentally friendly power generation equipment such as blades of tidal current turbines was examined. According to the Tafel analysis for micro-areas including the coating layer, the coating/metal interlayer and the base metal, the Al_2O_3 -3TiO₂ coating layer and the CoNiCrAlY coating layer show markedly lower corrosion current density than the base metal. The corrosion current density of the CoNiCrAlY coating layer (9.75316×10⁻⁸ A/cm²) is about 1.6 times more than that of the Al_2O_3 -3TiO₂ coating layer (6.13139×10⁻⁸ A/cm²).

Key words: Al₂O₃-3TiO₂ coating layer; CoNiCrAlY coating layer; underwater rotating products; marine environment; corrosion characteristics; atmospheric pressure plasma spray

1 Introduction

The atmospheric pressure plasma spray coating technology is used to improve the durability of products by forming a coating layer on metal surfaces with materials that have excellent qualities than substrate. In particular, it has a very extensive application scope because many different materials can be applied in line with the purpose such as abrasion resistance, cavitation resistance, and corrosion resistance [1,2]. On the other hand, thermal spray coating technology must select spray materials and condition in tune with the purpose because it is affected by various factors during the process. The spray materials used in this study were the ceramic powder Al₂O₃-3TiO₂ and the thermal barrier coating material CoNiCrAlY. Ceramic materials such as Al₂O₃-3TiO₂ have excellent chemical stability and mechanical characteristics such as high hardness and abrasion resistance. However, as they have high brittleness, the strength and bonding characteristics of the coating layer are low. However, CoNiCrAlY improves the surface characteristics such as corrosion resistance and abrasion resistance in a high temperature oxidation and corrosion environment [3,4]. This investigation intended to confirm the applicability of these high quality materials to harsh marine environment. Furthermore, to improve the durability of underwater rotating products, the corrosion characteristics in harsh marine environment were evaluated through various electrochemical experiments on the Al₂O₃–3TiO₂ and CoNiCrAlY coating layers.

2 Experimental

In this study, the ALBC3 alloy with excellent durability in seawater solution was used as the base metal, the ceramic powder Al_2O_3 -3TiO₂ and the thermal barrier coating material CoNiCrAlY were used as the coating materials. By evaluating the corrosion

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resistance of these materials, their applicability to environmentally friendly power generation equipment such as blades of tidal current turbines was examined. The chemical compositions of the used base metal and coating materials are listed in Tables 1, 2 and 3. To make identical roughness of the base metal surfaces, they were polished with emery paper No.600. For atmospheric plasma coating, the optimal conditions were applied to each material as shown in Table 4. Furthermore, the more detailed corrosion resistances of the Al₂O₃-3TiO₂ and CoNiCrAlY coatings will be analyzed more clearly later through potentiostatic and dynamic polarizations, EIS, and micro electrochemical experiments for micro-areas. The potential trends of ALBC3 alloy and coated specimens were measured over 86400 s in natural seawater. The anodic and cathodic polarization was made commencing from the open circuit potential to +3.0 and -2.0 V (vs SCE), respectively, at a scan rate of 2 mV/s at room temperature after immersion for 600 s.

Table 1 Chemical composition of ALBC3 alloy (mass fraction,%)

Al	Fe	Ni	Zn
9.30	3.66	4.39	0.34
Sn	Pb	Si	Mn
0.01	0.013	0.17	0.55

Table 2 Chemical composition of Al2O3-3TiO2 (mass fraction,%)

Al_2O_3	TiO ₂	SiO_2	Fe ₂ O ₃
95.4-98.0	≤0.50	≤0.60	2.0-3.5

 Table 3 Chemical composition of CoNiCrAlY (mass fraction,

 %)

Со	Ni	Cr	Al	Y
35.35-	31.0-	20.0-	7.0-	0.35-
41.65	33.0	22.0	9.0	0.65

 Table 4 Spray conditions of atmospheric pressure plasma
 coating for Al₂O₃-3TiO₂ and CoNiCrAlY

Parameter	Al ₂ O ₃ -3TiO ₂	CoNiCrAlY	
Argon/($L \cdot min^{-1}$)	38	65	
Spray distance/mm	120	140	
Feed rate/($g \cdot min^{-1}$)	50	40	
Hydrogen/(L·min ⁻¹)	14	14	
Current/A	600	600	
Powder gas/($L \cdot min^{-1}$)	3.2	2.3	
Injector angle/(°)	90	90	
Traverse speed/(mm \cdot s ⁻¹)	8	8	

The polarization system consisted of a Pt coil, which acted as a counter electrode, and an Ag/AgCl saturated KCI as the reference electrode. The potentiostatic experiment was conducted for 3600 s under various applied potentials to compare changes of current density with time, and compare the degrees of corrosion. Additionally, the polarization resistance (R_p) was assessed by performing electrochemical impedance spectroscopy (EIS) to investigate the corrosion resistance for base metal and coating layers. Furthermore, micro-electrochemical experiments was conducted to evaluate the electrochemical characteristics for the coated layer, interlayer and substrate of local area on the cross-section using droplet cells, and the set-up of the droplet cells used in the experiment are shown in Fig. 1 [5-7]. The droplet cells use saturated calomel electrodes as reference electrodes and platinum conducting wires pass through the inside of the capillaries to be connected to the counter electrode. When the capillary tips come into contact with specimens, only the areas exposed to the internal electrolyte solution work as the areas of working electrodes to compose electrochemical cells. In order to expose the areas of working electrodes in micro units, the diameters of the capillary tips were made to be about 150 µm and exposed areas during the electrochemical experiment were measured down to six places of decimals. In the micro electrochemical experiment, the tendencies of polarization with the basic of the open-circuit potential (φ_{oc}) from -0.25 V to +2.5 V in the local area were analyzed. The average corrosion current densities and average corrosion potentials obtained through the Tafel analysis were compared.

3 Results and discussion

Figure 2 shows the natural potential measurement results over time for 86400 s for ALBC3 alloy and APS coated specimens in natural seawater solution. In general, both the Al₂O₃-3TiO₂ and CoNiCrAlY coatings exhibit nobler potential compared to the ALBC3 base metal. The ALBC3 alloy repeated rising and falling in a very narrow range of -0.218 V to -0.198 V in the early stage of immersion. They showed the potential of -0.219 V at around 20000 s, and maintained stable behavior until the end of the experiment. On the other hand, the potential of the Al₂O₃-3TiO₂ coating layer decreased from -0.1096 V to -0.154 V until about 13000 s at a constant slope in the early stage of immersion. Then it stabilized until 25500 s. After that, it rose to a little noble potential and maintained stabilized condition until about 77000 s, but it showed unstable variations again. The reason for this is that unlike the base metal, lamellar shaped volumes are laminated on the surface of the coating layer, and there



Fig. 1 Set-up of micro droplet-cell and micrograph of capillary tip (RE denotes reference electrode, CE denotes counter electrode, WE denotes electrode)



Fig. 2 Curves of potential vs time for ALBC3 alloy and APS coating layers

are voids and open pores formed between the layers. These defects play the role of a penetration route for electrolytic solution at the electrochemical aspect [8]. Then, when the electrolytic solution penetrates into the base metal, micro-galvanic cells are formed between the coating and interlayer, and between the interlayer and the base metal. As a result, the coating layer decreases corrosion resistance effect and can be separated by causing the corrosion of base metal or lowering the adhesive strength of the interlayer. The defects formed inside the coating layer provide a penetration route for electrolytic solution. It seems to have caused somewhat unstable variations of potential [9]. On the other hand, the CoNiCrAlY coating layer exhibits nobler potential than the base metal and the $Al_2O_3-3TiO_2$ coating, and shows very stable tendency from the early stage of immersion until about 62000 s. After that, the potential tends to slowly move in the active direction, and the decrease of potential not great. Thus, it can be concluded that the corrosion resistance effects of the Al₂O₃-3TiO₂ and CoNiCrAIY coating layers would be excellent when compact coating layer is formed as the coating layers showed considerably nobler potential than the base metal. However, when plasma spray process is performed in the atmosphere with no vacuum environment, the impurities and gases from the outside are absorbed at high temperatures. In this case, when they exist in the coating layer as defects, it will cause unstable corrosion resistance characteristics [10].

Figure 3 compares the anodic polarization behavior of the ALBC3 alloy and APS-coated specimens in natural seawater solution. The open circuit potential (φ_{oc}) of the ALBC3 alloy is -0.207 V. As the anodic polarization progresses, the passivation characteristic of decreasing current density appears at 0.137 V ($\varphi_{\rm p}$). However, as the measured passive critical current density (J_p) is very high at 0.01403 A/cm², it cannot be applied as anodic corrosion protection method. The reason that passivation formation is observed in the anodic polarization behavior of the ALBC3 alloy is that the matters form by oxidation reaction on the surface. The Al and Ni elements added to the base metal have the characteristic for forming and restoring of sound passivation. For the passivation section of the ALBC3 alloy, oxidation matters are removed by the oxygen generated with continuous dissolution reaction and the de-lamination of oxidation matters or pitting corrosion occurs by the penetration of chlorine ions. This pitting corrosion (φ_t) is measured at 1.182V, and the current density rapidly increases larger than this potential. On the other hand, for the APS-coated specimen, the variation of current density with potential is very low compared to the ALBC3 base metal. In particular, current density steadily increases with potential larger than the open circuit potential (φ_{oc} =-0.037 V) of the Al₂O₃-3TiO₂ coating, showing no characteristic of passivation for formation and restoration. For the passivation section of the ALBC3 alloy, the oxidation matters are removed by the oxygen generated with continuous dissolution reaction and the de-lamination of oxidation matters or pitting corrosion occurs by the penetration of chlorine ions. This pitting corrosion (φ_t) is measured at 1.182V, and the current density rapidly increases after this potential. On the other hand, for the APS-coated specimen, the variation of current density with potential is very low compared to the ALBC3 base metal. In particular, the current density steadily increases with potential larger than the open circuit potential $(\varphi_{oc}=-0.037 \text{ V})$ of the Al₂O₃-3TiO₂ coating, and no passivation characteristics are observed. However, it shows considerably lower current density than the base metal, because the main chemical Al₂O₃ of the Al₂O₃-3TiO₂ coating layer is an insulator. Chemicals with such insulation characteristics have very good

corrosion resistance because it interferes with the flow of current generated by voltage in the electrochemical cell [11]. The increase of current density of the CoNiCrAlY coating layer according to potential variation is a little lower than that of the base metal. However, it shows very high current density compared to the $Al_2O_3-3TiO_2$ coating, and passivation is not observed at all. Consequently, the CoNiCrAlY coating layer shows weaker corrosion resistance than Al₂O₃-3TiO₂ during anodic polarization. These defects are the penetration route by the inclusion of impurities during the flight of melting splats in the APS spraying, the pores between the laminated layers and the defects due to the thermal history during cooling at high temperature [10,13]. In other words, as the Al₂O₃-3TiO₂ coating layer has relatively many pores and defects, the penetration of electrolytic solution is easy during long-term immersion. So, it shows the combined potential of the coating layer and the base metal at the interface. During the anodic polarization, however, the CoNiCrAlY coating layer causes more dissolution reaction under the reversible environment, and shows relatively higher current density.



Fig. 3 Comparison of anodic polarization trends for ALBC3 alloy and APS coating layers

Figure 4 presents the cathodic polarization behavior of the ALBC3 alloy and APS-coated specimens in natural seawater solution. The ALBC3 alloy shows a rapidly increasing trend of the current density with the decrease of potential as the ions uniformly distribute in areas close to the surface of the working electrode due to slow reduction rate after the open-circuit potential (-0.208 V). At around -0.45 V, however, the reduction rate of oxygen increases on the working electrode surface and the current density slows down as the oxygen ions are quickly consumed in the area close to the surface of ALBC3 alloy. This is the concentration polarization due to the dissolved oxygen reduction reaction and an electrochemical reaction that is



Fig. 4 Comparison of cathodic polarization trends for ALBC3 alloy and APS coating layers

dominated by the diffusion of oxygen in seawater solution. This polarization is caused by the concentration difference between the working electrode surface and in the electrolytic solution. Concentration polarization is dominated by the diffusion rate of ions toward the metal surface as the concentration of oxygen ions in the solution is limited. When the reduction rate is fast, the current density reaches and stays at the limiting diffusion current density (J_1) . This limiting diffusion current density is affected by the diffusion coefficient, such as the concentration of reacting ions in solution, and the thickness of the diffusion layer. This is a very important behavior in the reduction process [12]. The concentration polarization range of the ALBC3 alloy is from -0.45 V to -1.0 V. The J_1 in this case is approximately in the range from 3.35×10^{-5} to 5.86×10^{-5} A/cm². Thus, with the concentration polarization, the concentration of oxygen ions on the surface rapidly decreases by fast reduction rate and slows down the corrosion reaction. This potential range allows cathodic protection method in seawater environment, and it is used as a corrosion resistance system for marine structures as impressed current cathodic protection (ICCP) system. After the concentration polarization the activation polarization

occurs at the potential below -1.0 V and the current density sharply increases again. The APS-coated specimens show a little different behavior compared to the base metal. The CoNiCrAlY coating shows a higher current density than the base metal from the open circuit potential to -1.15 V. This range corresponds to the concentration polarization section and seems to present higher corrosion reaction than the base metal. After that in the activation polarization area, it show relatively lower values. On the other hand, the $Al_2O_3-3TiO_2$ coating layer only shows concentration polarization after the open circuit potential and the current density gradually decrease along with the decreasing potential. Although activation polarization must be more conspicuous below -1.68 V, the current density decreases further and stays at 1.149×10^{-4} A/cm². Consequently, the application of the $Al_2O_3-3TiO_2$ and CoNiCrAlY coating technology will show better corrosion resistance than the base metal.

Figure 5 compares the Bode plot and Nyquist plot which are the EIS analysis results for the ALBC3 alloy and APS-coated specimens in natural seawater solution. The surface of metals exposed to corrosive environment consists of oxides, chlorides, and hydroxides. In the case of ALBC3 alloy, its corrosion resistance is improved by forming corrosion products on surface such as cuprous oxide (Cu₂O). As shown in Fig. 5(a), the specimen coated with Al_2O_3 -3TiO₂ shows the highest polarization resistance in the Bode plot and the CoNiCrAlY coating shows a similar polarization resistance as the ALBC3 alloy. This result brings out in striking difference in the electro-kinetic potential polarization result that presents lower current density than substrate. The reason for relatively low corrosion resistance of the CoNiCrAlY coating layer appears to be that the electrolytic solution could easily penetrate as there are many pores and capillary cracks in the coating layer [13]. The EIS analysis find very weak corrosion resistance of the CoNiCrAlY coating compared to the $Al_2O_3-3TiO_2$ coating. The reason for this seems to be that when corrosion occurs on the coating surface in various ways, the movement of electric charge is interfered by the oxides that are formed on the surface. It seems that the Al₂O₃-3TiO₂ coating shows high corrosion resistance because it consists of Al₂O₃ compounds and the movement of electric charge is limited on the surface that has no corrosion damages. Furthermore, the Nyquist plot in Fig. 5(b) also shows the same tendency, but CoNiCrAlY shows a little higher corrosion resistance than the ALBC3 alloy due to the Warburg parameter response resulting from diffusion.

Figure 6 shows the variation of current density over time after a potentiostatic experiment during 3600 s for the Al_2O_3 -3TiO₂ and CoNiCrAlY coating layers in



Fig. 5 Comparison of EIS results for ALBC3 alloy and HVOF coating layers in sea water: (a), (a') Bode plot; (b), (b') Nyquist plot



Fig. 6 Potentiostatic experiments for Al₂O₃-3TiO₂ (a, a') and CoNiCrAlY coatings (b, b') in sea water

natural seawater solution. The potentiostatic experiment is good for evaluation of corrosion resistance as it evaluates detailed corrosion characteristics and compares the current density at constant potential within the same duration. Thus, it is needed to evaluate more detailed corrosion characteristics for the Al₂O₃-3TiO₂ and CoNiCrAlY coating layers which are applied to the APS coating technology. In this study the same potential condition was selected and the corrosion resistance characteristics were compared by comparing the current density under each potential condition. Both coating materials show similar overall tendency for the applied potential. The Al₂O₃-3TiO₂ coating in Fig. 6(a) increases from 200 s to 400 s in the early stage of the experiment and then tends to keep decreasing until the end of the experiment at 0.3 V and 1.0 V. The current density at the end of the experiment is 4.69×10^{-4} A/cm² and 8.54×10^{-4} A/cm^2 , respectively, and the higher the potential is, the greater the current density becomes. In the potential range of -0.25 V and -1.0 V, it shows a stable tendency until the end of the experiment. At 3600 s, the current densities are 2.15×10^{-3} A/cm² and 3.97×10^{-5} A/cm², respectively. At the applied potential of -0.2 V, however, the variations of current density are very large and it repeatedly rises and falls. This phenomenon is that the

oxidation and reduction reactions occur simultaneously in local areas near the equilibrium potential. The current density at this potential is very low at 1.24×10^{-6} A/cm². Meanwhile the CoNiCrAIY coating layer in Fig. 6(b) quickly moves in the noble direction at 0.3 V and 1.0 V in the early stage of experiment and shows stable behavior at a very high current density until the end of experiment. At the potential of -2.5 V, the current density is very high due to the activation polarization. As the applied potential is close to φ_{oc} , the current density tends to decrease. At the potential of -0.2 V, the current density becomes very low at 1.53×10^{-6} A/cm², and it repeatedly rises and falls. The current density values at the end of experiment for each potential condition are detailed in Table 5.

Figure 7 shows the tendency of current density at each applied potential at the end of the potentiostatic experiment during 3600 s for the Al_2O_3 -3TiO₂ and CoNiCrAlY coating layers. At all the potentials applied, the CoNiCrAlY coating shows higher current density than the Al_2O_3 -3TiO₂ coating. In particular, the difference in current density rapidly increases in the activation polarization (-2.5 V) and activation dissolution reaction (1.0 V) parts. Consequently, the CoNiCrAlY coating appears to have lower corrosion

Table 5 Current densities at end of potentiostatic experiments for Al_2O_3 -3TiO₂ and CoNiCrAlY coatings

Detential (v. $A = (A = C1)/V$	Current density/($A \cdot cm^{-2}$)		
Potentiai (vs Ag/AgCI)/v	Al_2O_3 -3 TiO_2	CoNiCrAlY	
-2.5	2.15×10^{-3}	2.19×10 ⁻²	
-1.0	3.97×10^{-5}	9.03×10 ⁻⁵	
-0.2	1.24×10^{-6}	1.53×10 ⁻⁶	
0.3	4.69×10^{-4}	7.25×10 ⁻³	
1.0	8.54×10^{-4}	2.30×10^{-2}	



Fig. 7 Comparison of current density at end of potentiostatic experiments

resistance due to activation polarization and dissolution reaction, which corresponds to the results of the EIS experiment described above.

Figure 8 shows the polarization behavior for Tafel analysis for the micro-areas of the cross-sections of the Al₂O₃-3TiO₂ and CoNiCrAlY coatings including the coating layer, coating/base metal interface and the metal using micro-droplet cells. For the experiment areas, after polishing the cross-sections of the coated specimens, the capillary tip contacts with the coating layer (A), the coating/base metal interlayer (B), and the base metal (C). For the coating/base metal interlayer, they are exposed at the equal ratio of area by positioning the capillary of the micro-droplet cells at the center. Furthermore, as the electrochemical behavior at the micro-areas using the micro-droplet cells appears in various ways depending on the distributions of pores, oxides, and various elements, three or more experiments were performed for each area to improve reliability. Firstly, in the case of Al_2O_3 -3TiO₂ in Fig. 8(a), the corrosion current density

of the coating layer is 6.13139×10^{-8} A/cm² which is 1/45 of that of the base metal. On the other hand, the corrosion current density of the coating/base metal interlayer measured at 3.6061×10⁻⁷ A/cm² which is approximately 6 times as that of the coating layer and 1/8of that of the base metal. For CoNiCrAlY in Fig. 8(b), the corrosion current density of the coating layer is 9.75316×10^{-8} A/cm², which is 1/28 of that of the base metal, and the value of the coating/base metal interlayer is 1/7 of that of the base metal. Thus, the corrosion current density at each interlayer appears to be the mixed potential of the coating layer and the base metal. The corrosion current density of the coating layers are 1/45 and 1/28 of that of the base metal, respectively. It is predicted that CoNiCrAlY will have a corrosion current density that is about 1.6 times as that of Al₂O₃-3TiO₂. The details of the Tafel analysis results for the Al₂O₃-3TiO₂ and CoNiCrAlY coating layers, interlayers, and the base metal are shown in Table 6.



Fig. 8 Comparison of Tafel analysis curves using micro-droplet cell at local area: (a) Al₂O₃-3TiO₂; (b) CoNiCrAlY

 Table 6 Tafel analysis for local area of cross-section on coating layers using micro-droplet cell

Area —	Corrosion potential/V		Corrosion current density/(A·cm ⁻²)			
	Al ₂ O ₃ -3TiO ₂	CoNiCrAlY	Substrate	Al ₂ O ₃ -3TiO ₂	CoNiCrAlY	Substrate
Coating layer	-0.319	-0.217	-0.295	6.13139×10 ⁻⁸	9.75316×10 ⁻⁸	2.71121×10^{-6}
Interlayer	-0.307	-0.258		3.6061×10^{-7}	3.85313×10^{-7}	2./1131×10

4 Conclusions

1) The natural potential shows that the $Al_2O_3-3TiO_2$ and CoNiCrAlY coating layers have very noble potentials compared to the ALBC3 alloy.

2) The anodic and cathodic experiments show that the CoNiCrAIY coating layer shows a higher current density than the Al_2O_3 -3TiO₂ coating layer during polarization, and no passivation is observed. In contrast, the Al_2O_3 -3TiO₂ coating layer shows very good corrosion resistance as it contains insulation compounds.

3) The potentiostatic experiment found that the $Al_2O_3-3TiO_2$ coating layer has lower corrosion current density at all potentials than the CoNiCrAlY coating layer. As a result, the $Al_2O_3-3TiO_2$ spray material is found to be more appropriate for improvement of corrosion resistance of the ALBC3 alloy when using the atmospheric plasma technology for coating.

4) At the Tafel analysis for micro-areas including the coating layer, the coating/metal interlayer and the base metal, the Al_2O_3 -3TiO₂ coating layer and the CoNiCrAlY coating layer shows markedly lower corrosion current density than the base metal: their corrosion current density values are only 1/45 and 1/28 of that of the base metal. Furthermore, the coating/metal interlayer shows a corrosion current density that is in the middle between the coating layer and the base metal. The corrosion current density of the CoNiCrAlY coating layer is about 1.6 times as that of the Al₂O₃-3TiO₂ coating layer.

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