

Article

A Study on the High-Temperature Molten Salt Corrosion Resistance of Hot-Dip Aluminum/Carburizing Composite Coating on Ti65 Titanium Alloy

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Abstract: This article presents a new method for preparing a coating on Ti65 titanium alloy using a two-step procedure comprising hot-dipped aluminum and solid carburization. The effects of the carburization on the hot-dipped aluminum coating against the presence of a NaCl deposit at 810 °C were systematically studied. In this article, the microstructure, morphology, phase composition of the coating, and corrosion products were investigated using SEM (Scanning Electron Microscopy), EDS (Energy Dispersive Spectrometer), and X-ray diffraction. The results indicated that the corrosion resistance of the hot-dip aluminum/carburizing composite coating was not significantly enhanced with the hot-dip aluminum coating. This can be attributed to the formation of TiC and Ti₃AlC after carburization, which promoted the formation of loose and unprotected TiO₂ in the coating during molten salt corrosion. In addition, the oxidation of the carbon atom into CO₂ led to a high concentration of pores in the coating, creating channels for NaCl to penetrate the coating and accelerate the corrosion rate.

Keywords: titanium alloy; hot-dip aluminum/carburizing composite coating; hot-dip aluminum coating; molten salt corrosion



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1. Introduction

Molten salts possess many attractive properties for use as a coolant and storage medium in advanced nuclear reactors and concentrated solar power systems [1–3]. Despite these advantages, severe corrosion issues of structural materials in molten salt still remain a big challenge [4–6]. Titanium alloys are widely recognized as ideal materials for compressor blades due to their excellent properties, such as low density, high strength, and strong corrosion resistance [7–11]. However, when applied in marine environments, the titanium alloy surface combines salt sediment in the form of NaCl, high temperatures, Cl[−] ions, and oxygen, resulting in severe corrosion of compressor components [12–14]. As a result of the synergistic corrosion effects, severe corrosion failures occur with substantially higher corrosion rates than oxidation rates under typical atmospheric circumstances [12,13,15,16]. This rapid corrosion is often associated with the presence of molten salts on the surface or in the scales, and it also accelerates when solid or gaseous salts are present [17–19]. Therefore, it is essential to develop suitable protective coatings for titanium alloy to enhance resistance against oxidation and molten salt corrosion under high-temperature conditions.

According to previous research, the application of protective surface coatings is a preferred way to enhance corrosion resistance [20]. Naveen Kumar et al. [21] found that 86WC-10Co-4Cr-coated AISI316 stainless steel exhibited a much lower weight increase (1.1880 mg/cm²) than untreated specimens (2.6857 mg/cm²) in 88Fe₂(SO₄)₃-12%Na₂SO₄ corrosive environment for 50 cycles at 650 °C. Du et al. [22] prepared TiSiCN/Ag nanocomposite coatings on titanium alloy substrates and found that the mass gain in the titanium substrate was 2.750 mg/cm², while the mass gain in the coating was 1.937 mg/cm² after

hot etching at 550 °C for 72 h. These coatings provide additional protection by preventing contaminated salt from attacking the substrate [21–28]. One promising coating technique for improving the hot corrosion resistance of titanium alloys is the preparation of aluminate coatings [29–31]. Aluminum forms a stable Al_2O_3 phase to protect the metal matrix at high temperatures [19,32,33]. Among the Ti–Al intermetallic compounds (TiAl , TiAl_2 , and TiAl_3), TiAl_3 is the only one that can generate a dense Al_2O_3 oxide film in air, so TiAl_3 has the best oxidation resistance [34]. However, the theoretical thermal expansion coefficient of TiAl_3 is much higher than that of the titanium alloy matrix and is thus prone to thermal cracks [34,35]. In contrast, TiAl phase toughness is the best, but in a long-term high-temperature environment, the oxidation resistance of TiAl phase is insufficient [36]. Among the various methods available for preparing aluminum coatings, hot-dip aluminizing has garnered significant attention from researchers due to its advantages, such as a simple preparation process, low cost, and excellent coating adherence [37–39]. Hot-dip aluminizing is a method that involves immersing the plated object in molten metal liquid to generate a metal coating on its surface. In a study by Payank Pate et al. [40], the microstructure of the aluminized coating after diffusion heat treatments was found to consist of an outer layer of Al_2O_3 , followed by TiAl_3 and TiAl_2 phases. Yuansheng Wang et al. [41] discovered the formation of TiAl_3 , $\theta\text{-Al}_2\text{O}_3$, and compact $\alpha\text{-Al}_2\text{O}_3$ in the outer layer, which was believed to contribute to improved oxidation resistance at high temperatures. Shiang-Cheng Jeng [42] successfully prepared an aluminized Ti–6Al–4V alloy by hot-dipping aluminum under a protective argon atmosphere at 800 °C, and the results demonstrated that the aluminized coating provided significant protection against oxidation at this temperature.

Carburizing is a chemical heat treatment procedure that increases the carbon content of a steel surface and forms a specific carbon concentration gradient in order to improve steel strength, impact toughness, and wear resistance, as well as to extend the service life. The carburization process has been widely utilized in Ti-based alloys. Hong-Qiang DUAN [43] discovered that solid carburization on Ti–6Al–4V alloy and $(\text{TiB} + \text{La}_2\text{O}_3)/\text{Ti}$ composite can enhance surface hardness. Cuijiao Liao et al. [44] observed the formation of a stable passivation film on TiAl alloy using solid carburizing, which improved the corrosion resistance of porous TiAl alloy. Tianhang Yao et al. [45] demonstrated that the carburization process is an effective and feasible treatment for enhancing the oxidation resistance of high Nb-containing TiAl alloys. However, limited research has been conducted on carburizing after aluminized titanium alloy coating, and the mechanism protecting this coating against molten salt corrosion remains not fully understood.

In this article, a new method for preparing coatings using a two-step procedure comprising hot-dipped aluminum and solid carburization was used on Ti65 titanium alloy for the first time. The samples were buried in a crucible containing molten salt (NaCl) in a muffle furnace at 810 °C for high-temperature molten salt corrosion. More importantly, the experiment has good reproducibility. The effects of solid carburization on molten salt corrosion resistance were investigated in detail. Corrosion kinetics, SEM, EDS, and XRD results were used to discuss the mechanism protecting hot-dip aluminum/carburizing composite coating against molten salt corrosion [16,46]. Therefore, the objective of this study is to simulate corrosion of the hot-dip aluminum/carburizing composite coating on compressor blades in the marine environment, and the corrosion mechanism for the coating was analyzed to lay a good foundation for improving the marine environmental resistance of titanium alloys.

2. Materials and Methods

2.1. Material Preparation

Ti65 alloy was used as the substrate material. The composition of Ti65 alloy is shown in Table 1. Before the experiments, specimens were cut into 15 mm × 10 mm × 3 mm pieces with a sparkline saw. To facilitate hanging into molten aluminum, a hole with a 3 mm diameter was drilled near the edge of the samples. All the surfaces were ground with 400#,

600#, and 800# SiC paper and polished to achieve a mirror-like surface using aluminum oxide polishing.

Table 1. Chemical composition of the Ti65 alloy (wt.%).

Al	Sn	Ta	W	Zr	Mo	Nd	Si	C	Ti
5.9	4.0	2.0	1.0	3.5	0.3	0.3	0.35	0.05	Bal.

2.2. Hot-Dip Aluminizing

Prior to hot-dip aluminizing, the surface of the samples was first ultrasonically degreased with acetone for 3–5 min followed by rinsing with water. Subsequently, the samples were cleaned and dried immediately. Under the protection of argon gas, an alumina ceramic crucible containing pure aluminum ingots was heated to 800 °C in a well-resistance furnace (Xiangtan Samsung Instrument Co., LTD, Xiangtan, China). After the aluminum ingots were completely melted, the temperature was reduced to 760 °C. Then, the pre-treated Ti65 samples were immersed in pure aluminum liquid and quickly extracted after 10 min. When the desired hot-dipping time was reached, the specimen was quickly extracted from the melt, the excess melt was shaken off its surface, and then it was air-cooled to room temperature. The derived specimen was denoted as the hot-dip aluminum coating.

2.3. Solid Carburization

The hot-dip aluminum alloy was buried in a corundum crucible. The composition of the used solid carburized was 10%Na₂CO₃-10%Ba₂CO₃-5%CaCO₃, and the charcoal had a particle size of 1.5–3 mm. After being covered with an alumina lid and sealed with silica sol binder, the alumina crucible was put into the center of the muffle furnace chamber (Xiangtan Samsung Instrument Co., LTD, Xiangtan, China). Then, tests were conducted at 1050 °C using a furnace for 4 h followed by furnace cooling. Finally, the samples with coatings were taken out from the crucible powder, cleaned, and dried for a corrosion test. This coating was named hot-dip aluminum/carburizing composite coating.

2.4. Corrosion Experiments

Prior to the corrosion test, the sample was placed in absolute ethanol for ultrasonic cleaning. After cleaning, the samples were dried for 2 h at 100 °C. The initial mass was subsequently measured using an electronic analytical balance with an accuracy of 0.0001 g, the mass was tested three times, and the average mass gain value is presented in this work. The corrosion weight change rate was calculated to assess the performance of molten salt corrosion resistance on the coated and uncoated specimens at high temperatures. First, sodium chloride particles were put into the alumina ceramic crucible, and then the samples were immersed completely in the sodium chloride particles. The corrosion tests were started by introducing the alumina crucible directly into a hot muffle furnace (Xiangtan Samsung Instrument Co., LTD, Xiangtan, China) at 810 °C in static air to ensure rapid heating. Uncoated Ti65 titanium was used as the reference sample for the NaCl molten salt corrosion experiment. After given intervals (5, 5, 5, 5, 5, 10, 10, 10, 31, 24 h), the samples were removed from the furnace and cooled in the air to room temperature. Subsequently, the corroded samples were washed in boiling deionized water for 30 min to clean out the remaining unreacted salt completely, and then they were dried completely. The mass of each sample was measured using an electronic balance. The mass gain per unit area after each cycle was calculated using Equation (1):

$$\Delta w = (m_1 - m_0)/S \quad (1)$$

where m_1 represents the weight (mg) after each interval, m_0 represents the weight (mg) at the beginning of each cycle, and S is the total sample surface area (cm²). In addition, for every experiment, three specimens were used in the same condition to obtain the average mass change.

2.5. Analytical Characterization

The coatings were analyzed using X-ray diffraction (XRD) and scanning electron microscopy with energy dispersive X-ray analysis (SEM/EDAX). An X-ray diffractometer (Rigaku D/max 2500, Tokyo, Japan) with Cu-K α ($\lambda = 1.542 \text{ \AA}$) was used to analyze the phase composition of the coating before and after molten salt corrosion (2θ : 10° – 90° , Scanning speed: 10.0000 deg./min , current: 40 mA , voltage: 40 kV). The microstructure and element distribution of the coating were analyzed using scanning electron microscopy (ZEISS EVO MA10, Zeiss, Jena, Germany). An energy dispersive spectrometer ((OXFORD X-MAXN, Zeiss, Jena, Germany) was used for the microstructure study and semi-quantitative chemical analysis, respectively.

3. Results

3.1. The Microstructures of the Hot-Dip Aluminum/Carburizing Composite Coating

Figure 1a exhibits cross-sectional morphologies of the hot-dip aluminum/carburizing composite-coated samples. The coating has clearly layered structures with a thickness of about $196 \text{ }\mu\text{m}$ and consecutive layers that differ in composition. Based on the enlarged SEM image (Figure 1b), some micro-cracks appear in the coating. According to the elemental mappings in Figure 2, there is sufficient Al and Ti in the coating, while the C atom is dispersed in the coating, and a thin and continuous oxide scale rich O forms at the surface, which indicates that the coating is partially oxidized during carburization.

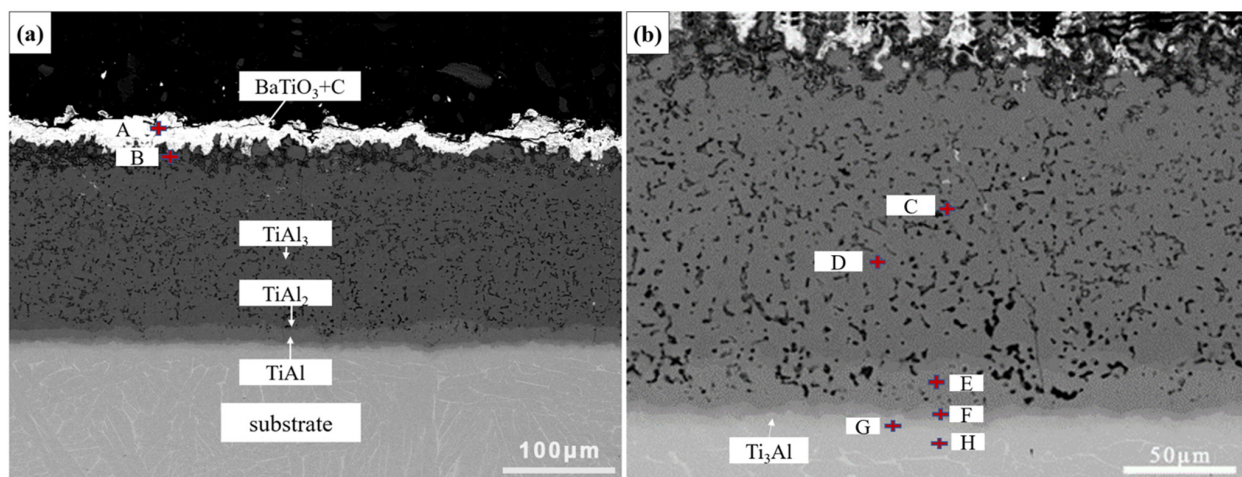


Figure 1. The cross-sectional morphologies of the hot-dip aluminum/carburizing composite coating. (a) is overall view of the microstructure; (b) is enlarged image.

To further confirm the phase constitutions of the hot-dip aluminum/carburizing composite coatings, the chemical compositions of layers were detected using EDS, as listed in Table 2, and the XRD patterns are given in Figure 3. The EDS results indicate that the chemical composition of the top scale (Point A in Figure 1a and Table 2) is $22.20\text{C}-51.20\text{O}-2.62\text{Al}-12.38\text{Ti}-11.51\text{Ba}$ (at.%). Combined with the XRD results, it is reasonable to assume that is BaTiO_3 produced by the reaction between the carburizing agent (BaCO_3) and Ti atom and some C atoms without reaction. The chemical composition of Point B in Figure 1a consists of a mixture of Al_2O_3 , TiO_2 , TiC , and Al_4C_3 ($15.31\text{C}-53.12\text{O}-24.72\text{Al}-6.85\text{Ti}$ at%). In addition, some worm-like structures (Point C in Figure 1b and Table 2) are found in the coating. According to the EDS data in Table 2, it contains an O element, but the content of the Ti element is very low, which is presumed to be composed of Al_2O_3 and Ti-Al. The reason for this structure may be that the molten aluminum spread rapidly and the Ti contacting the molten aluminum began to dissolve and diffuse to the molten aluminum at the same time during the hot-dip aluminum process. TiAl_3 is the first compound formed during the reaction between Ti and Al since the diffusion flux through TiAl_3 is

several orders of magnitude higher than that through the other intermetallic compounds in Ti-Al (TiAl_2 , TiAl , Ti_3Al) [47], and the excess Al atoms are expelled from the TiAl_3 crystal. With the nucleation and growth of TiAl_3 particles, the amount of Al in the liquid phase rapidly grew until, at the freezing point, all the Al converted to pure Al. Therefore, granular TiAl_3 and pure Al filled between the particles were formed after hot-dipped aluminum [41,42,48,49]. During the carburizing process, the original pure Al was oxidized, and Al_2O_3 grains were formed as nucleating particles in the coating. With the extension of time, O atoms continued to enter the coating, and Al_2O_3 grew until all the pure Al had become Al_2O_3 . Additionally, it can be seen that C atoms are dispersed at the liquid phase interface. It is interesting to note that at Point D (Figure 1c and Table 2), the composition of Al to Ti is nearly 3:1, demonstrating that the gray block was comprised of TiAl_3 , which is consistent with the XRD patterns (Figure 3). It was reported that the TiAl_3 phase is brittle in the aluminized coating because of cracks [40,50]. Moreover, the coating near the substrate is still composed of titanium and aluminum in an atomic ratio of about 3:1, 1:1, and 2:1, which would correspond to Ti_3Al , TiAl , and TiAl_2 , respectively. The Ti_3Al layer is compact and uniform and has good adhesion to the substrate. The presence of TiAl_3 , Al_2O_3 , and TiAl_2 phases was confirmed with the XRD pattern, and a tiny TiAl_2 peak was identified due to the oxidation of TiAl_3 to TiAl_2 during carburizing. Because the outer phase coating is so thick ($\sim 158\mu\text{m}$), X-rays cannot reach the inner layer to detect TiAl and Ti_3Al , which agrees well with the XRD characterization.

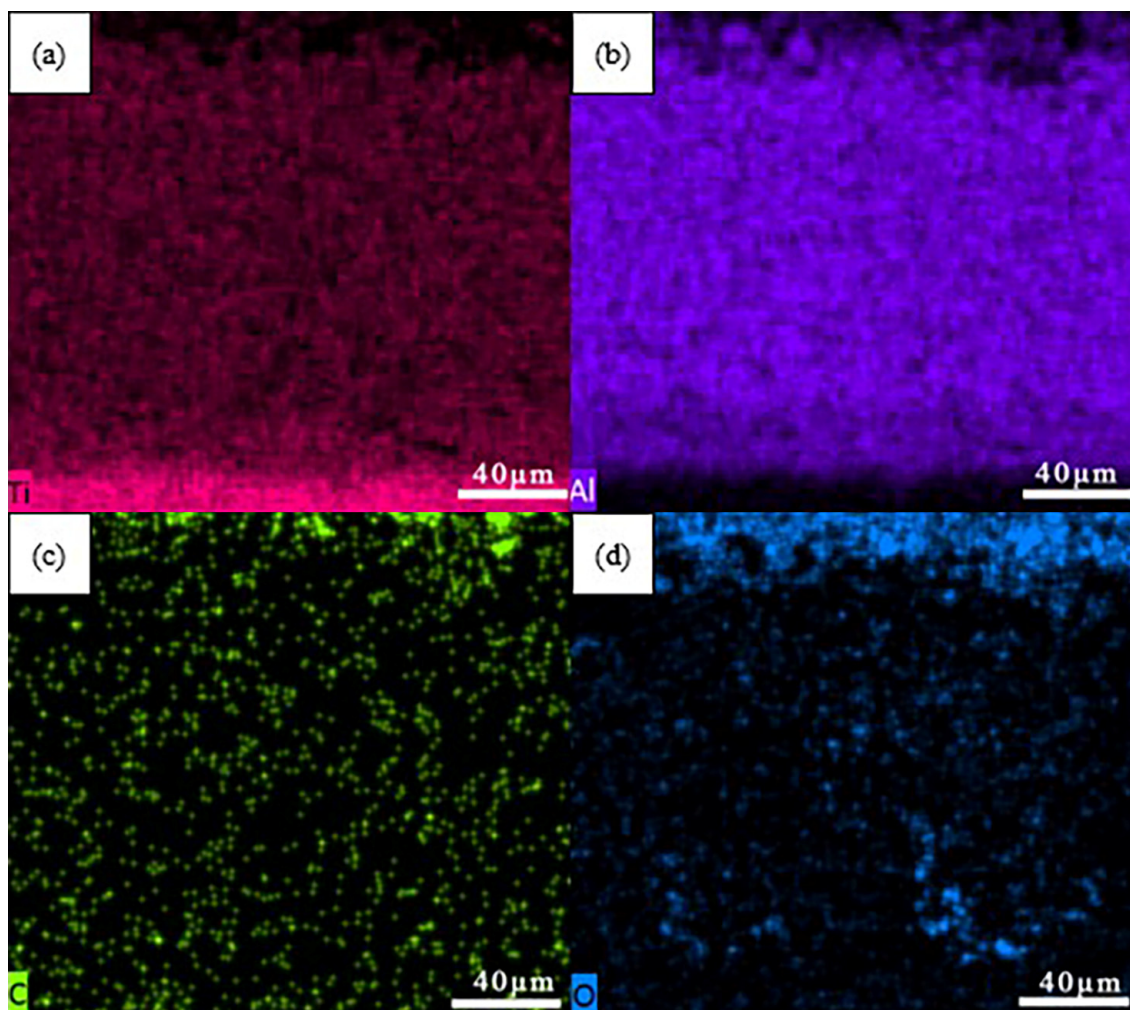
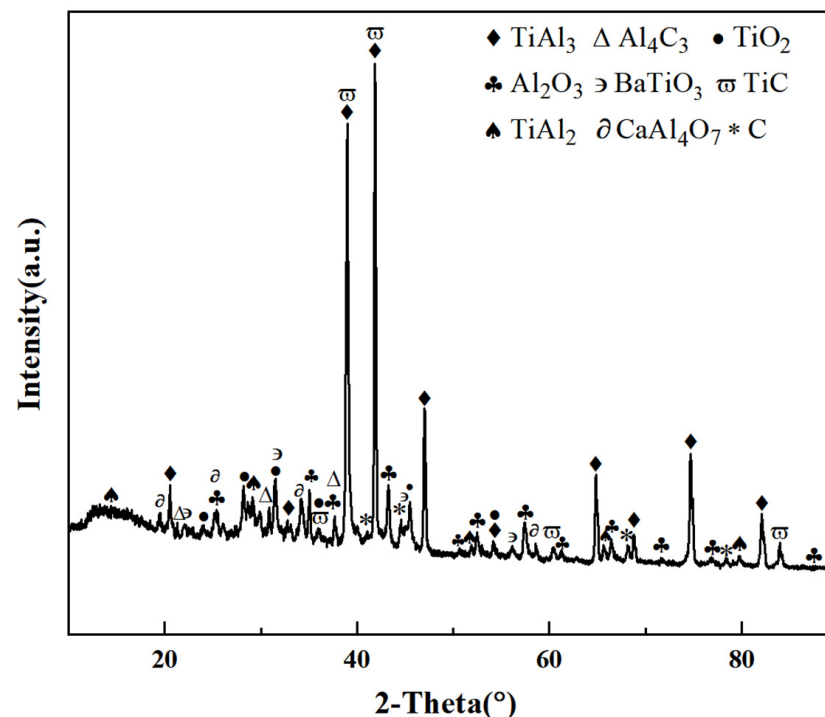


Figure 2. Element mappings of hot-dip aluminum/carburizing composite coating (Ti, Al, C, and O are represented by red, purple, green, and blue, respectively). (a) is Ti; (b) is Al; (c) is C; (d) is O.

Table 2. EDS results for the regions marked in Figure 1b (at.%).

Point	Ti	Al	C	O	Ba	Possible Phase
A	12.38	2.62	22.20	51.20	11.51	C + BaTiO ₃
B	6.85	24.72	15.31	53.12	/	Al ₂ O ₃ + TiO ₂ + TiC + Al ₄ C ₃
C	15.94	60.62	/	25.45	/	Al ₂ O ₃ + Ti-Al
D	29.87	70.13	/	/	/	TiAl ₃
E	33.19	66.81	/	/	/	TiAl ₂
F	49.39	50.61	/	/	/	TiAl
G	70.52	29.48	/	/	/	Ti ₃ Al
H	83.71	16.29	/	/	/	Ti ₃ Al + Ti

**Figure 3.** The XRD patterns of the hot-dip aluminum/carburizing composite coating.

3.2. Molten Salt Corrosion Kinetics and Optical Images

The evolution with time of the weight loss per unit area of the Ti65 alloy, hot-dip aluminum coating, and hot-dip aluminum/carburizing composite coating after the 120 h molten salt corrosion test are presented in Figure 4. The data for the uncoated samples and hot-dip aluminum coating are also given for comparison. Clearly, the weight loss of uncoated samples increased rapidly with increasing corrosion time due to the formation of the non-protective corrosion product at the surface. In contrast, the hot-dip aluminum coating and hot-dip aluminum/carburizing composite coating alloy followed with a steady and smaller mass change. Additionally, after 120 h of hot salt corrosion, the total mass loss of the hot-dip aluminum coating and hot-dip aluminum/carburizing composite alloy was near 0.67 mg/cm² and 5.06 mg/cm², respectively, while the total mass gains of uncoated alloys dramatically reduced to 76.57 mg/cm², which is approximately 114.3 and 15.1 times that of the hot-dip aluminum coating and hot-dip aluminum/carburizing composite coating. Therefore, the results indicate that the molten salt corrosion resistance of the hot-dip aluminized/carburized composite coating and the hot-dip aluminized coating was similar, and both were much higher than that of Ti65 alloy.

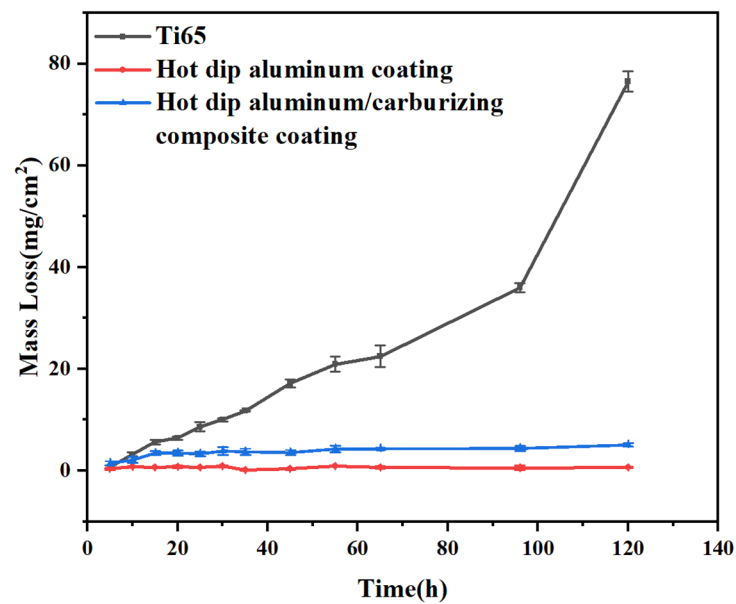


Figure 4. Kinetic curves of Ti65, hot-dip aluminum coating, and hot-dip aluminum/carburizing composite coating.

After the molten salt corrosion experiments, optical images of these specimens were also obtained, as shown in Figure 5. The color of the hot-dip aluminum/carburizing composite coating sample turned into the surface, which was composed of black and dark gray. The corrosion products were severely spalled after molten salt corrosion for 45 h, and the corrosion attack proceeded rapidly with an increase in exposure time.

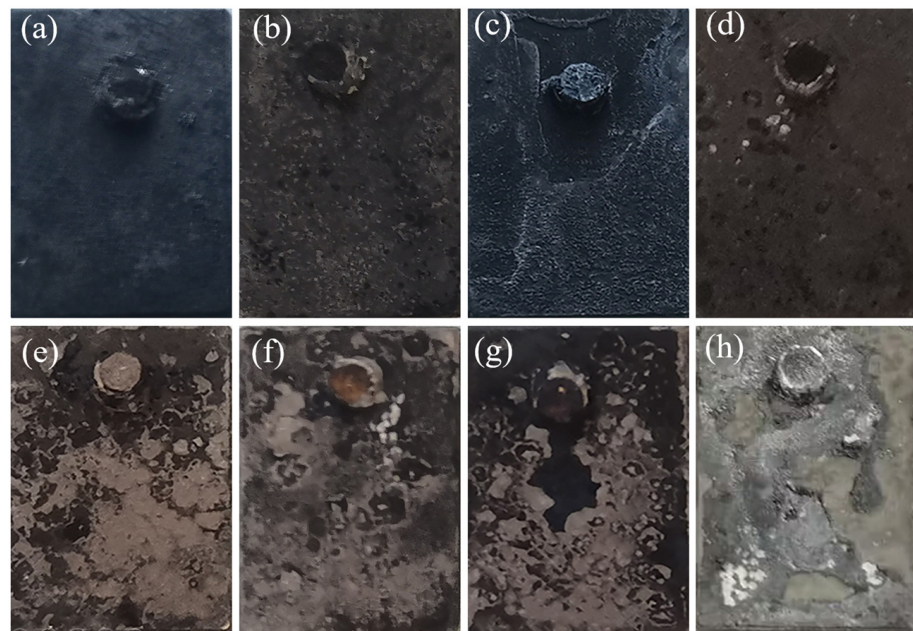


Figure 5. Optical images showing macro-morphologies of the hot-dip aluminum/carburizing composite coating after molten salt corrosion for (a) 0 h, (b) 5 h, (c) 10 h, (d) 35 h, (e) 45 h, (f) 65 h, (g) 96 h, and (h) 120 h.

3.3. Cross-Sectional Morphologies and Surface Analysis of the Hot-Dip Aluminum Coating after Molten Salt Corrosion

The surface observation results and XRD patterns of the hot-dip aluminum coating after 120 h of molten salt corrosion at 810 °C are shown in Figure 6. The surface of the

hot-dip aluminum coating was torn off, and some irregular massive particles were strewn on it. The corrosion products discovered on the surface of the hot-salt-corroded hot-dip aluminum coating mainly consist of TiAl_3 followed by TiO_2 with only a few diffraction peaks in the XRD patterns, confirming that the coating was stable under this condition.

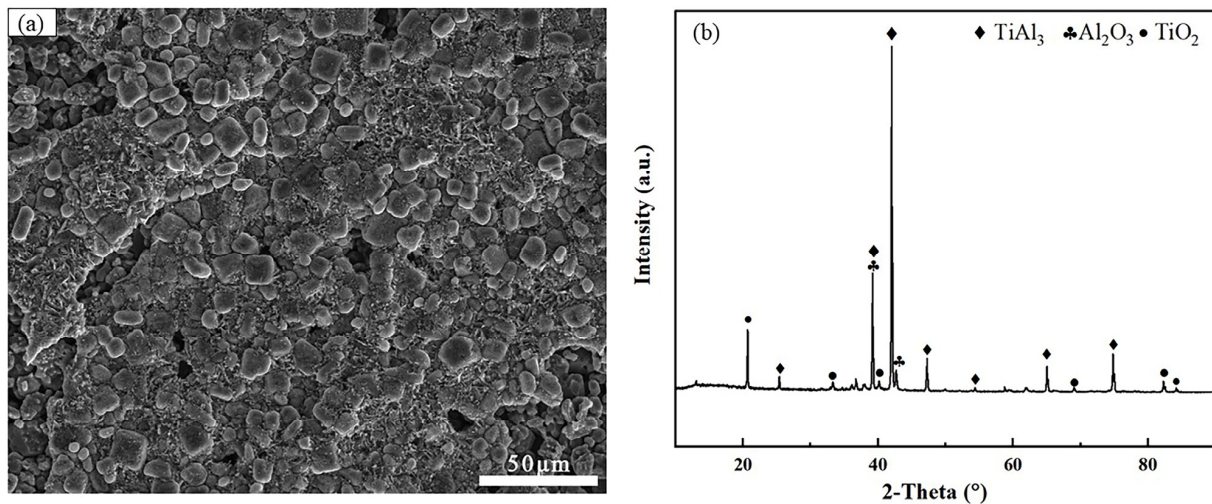


Figure 6. Surface morphology (a) and XRD patterns (b) of the hot-dip aluminum coatings after corrosion in NaCl salt at 810 °C for 120 h.

Figure 7 depicts the cross-sectional morphologies of the aluminate coating. The data in Table 3 are the atomic content ratios corresponding to those indicated in Figure 7b. It is clear that there was a transverse crack throughout the coating due to the effect of thermal stress and molten salt oxidation. The worm-like structure of the corroded coating is still apparent (Point A in Figure 7b). The EDS results show that the element content at this point was 7.58Ti-38.41Al-54.01O (at. bal), suggesting that the phase composition was still Al_2O_3 and hot-dip aluminum intermetallic compounds. The chemical composition of Point B (Point A in Figure 7b and Table 3) was 24.64Ti-55.54Al-31.34O phase, which may be composed of TiAl_3 , Al_2O_3 , and TiO_2 phases. The composition of Points C, D, and E in Figure 7b and Table 3 was 35.67Ti-64.33Al, 51.78 Ti-48.22Al (at. bal), and 78.81Ti-21.19Al (at. bal), respectively, so it is reasonable to deduce that the phase was TiAl_2 , TiAl , and Ti_3Al , respectively. The reason for the appearance of these three hot-dip aluminum phases is the interdiffusion of the Al and Ti atoms between the composite substrate and TiAl_3 coating with the extent of corrosion time. The formation of the transition diffusion layer of TiAl_2 , TiAl , and Ti_3Al can reasonably reduce crack propagation or development, so the crack does not extend to the substrate [51].

3.4. Cross-Sectional Morphologies and Surface Analysis of Hot-Dip Aluminum/Carburizing Composite Coatings after Molten Salt Corrosion

Figure 8 demonstrates the surface morphology and XRD patterns in the hot-dip aluminum/carburizing composite coating after 120 h of molten salt corrosion. The surface was rough with irregular-shaped clusters distributed throughout, as shown in Figure 8a, and some regions were formed of blocks separated by fissures. The EDS results (Table 4) indicated the chemical compositions of the white scale (Point A in Figure 8a) was 11.89Ti-3.22Al-8.46C-65.37O-1.64Ca-9.42Ba, and the content of O, Ti, and Ba elements were high, which is presumed to be composed of BaTiO_3 . The greyscale had a high content of Ca elements, and combined with the XRD results, this may be composed of CaAl_4O_7 . Furthermore, the content of the Al element is high in the black area. According to the XRD pattern (Figure 8b), it was found that the main phase was the diffraction peak of the Al_2O_3 and TiAl_3 phases, and the diffraction intensities of TiAl_2 were enhanced. In addition, Al_4C_3 disappeared, but NaAlO_4 , Na_2TiO_3 , and TiAl newly emerged. The XRD patterns inside the

coating after 5 h and 120 h of molten salt corrosion were conducted to further shed light on the microstructures of the hot-dip aluminum/carburizing composite coatings (Figure 9). The black line refers to the XRD diffraction picture after grinding off a layer of the surface, and the red line is the XRD diffraction map after grinding off a layer based on the red line. Ti_3AlC and Al_4C_3 X-ray diffraction peaks were found in the coating after 5 h of corrosion. However, the Al_4C_3 X-ray diffraction peaks disappeared, and only one Ti_3AlC diffraction peak was found, which could not confirm the existence of Ti_3AlC in 120 h, indicating that Ti_3AlC and Al_4C_3 were consumed by the reaction with the increment of exposure time. In addition, the TiAl phase was found in the samples corroded for 5 h and 120 h.

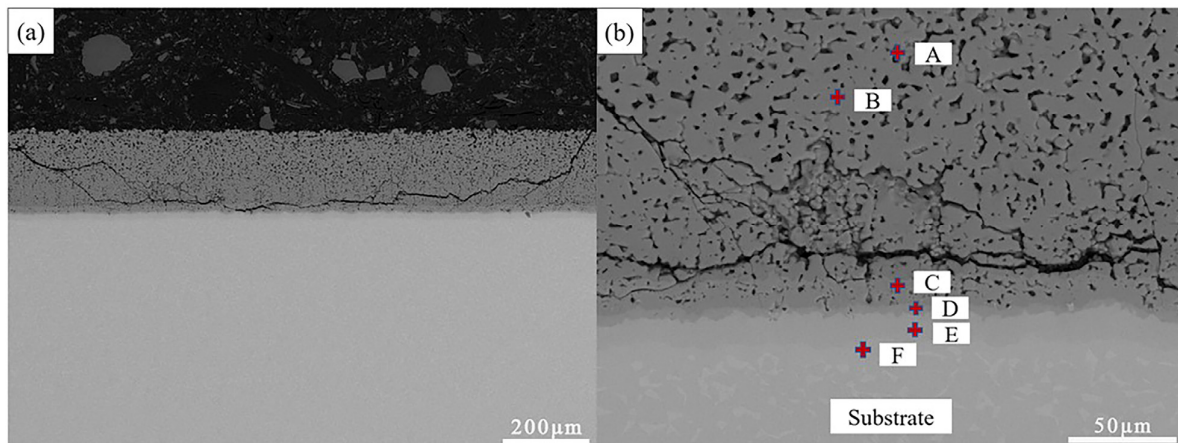


Figure 7. (a) The cross-sectional morphologies of the hot-dip aluminum coating in NaCl salt at 810 °C for 120 h. (b) Microstructure details. The “capitalized letters/red +” is the location of EDS point scanning.

Table 3. EDS results for the regions marked in Figure 7b (at.%).

Point	Ti	Al	O	Possible Phase
A	7.58	38.41	54.01	$\text{Al}_2\text{O}_3 + \text{Ti-Al}$
B	24.64	55.54	31.34	$\text{TiAl}_3 + \text{Al}_2\text{O}_3 + \text{TiO}_2$
C	35.67	64.33	/	TiAl_2
D	51.78	48.22	/	TiAl
E	78.81	21.19	/	Ti_3Al

Figure 10 depicts the cross-sectional morphology of the hot-dip aluminum/carburizing composite coating in molten NaCl at 810 °C for 120 h. It can be observed that the aluminum/carburizing composite coating suffered severe corrosion attack forming a thick, porous, and loose scale, whose characteristics are completely different from those of the non-corroded coating. Notably, the corrosion extent of the hot-dip aluminum/carburizing composite coating suffered a more severe corrosion attack than the hot-dip aluminum coating after exposure for the same period at the same temperature. Combining the EDS results (Table 5) and elemental mapping, the flocculent structure formed should be TiO_2 and Na_2TiO_3 . TiO_2 mostly exists in loose island structures at high temperature, which is easy to flake and leads to the reduction of Ti content. The chemical composition of the dark part at the edge of the flocculent material was 9.5Ti-65.88Al-60.20O-0.64Cl-0.66Na, and the phase composition was TiAl_3 and Al_2O_3 . The gray block scale with the average content of Al and Ti 63.81 at% and 34.33 at%, respectively, was composed of TiAl_2 based on the EDS result (Table 5). The coating near the base was still composed of TiAl and Ti_3Al . According to the element mappings (Figure 10), Cl and Na atoms were mainly distributed in the grain boundary of the carburizing layer, the pure aluminum layer, and the loose TiAl_3 layer, accelerating the grain boundary corrosion. Particularly, there were cracks throughout the coating after molten salt corrosion for 120 h, as shown in Figure 11. The main chemical

composition of the cracks was C and O elements (Table 6), and the oxidation of the carbon atom into CO_2 led to a high concentration of pores in the coating, creating channels for NaCl to penetrate the coating and accelerate the corrosion rate.

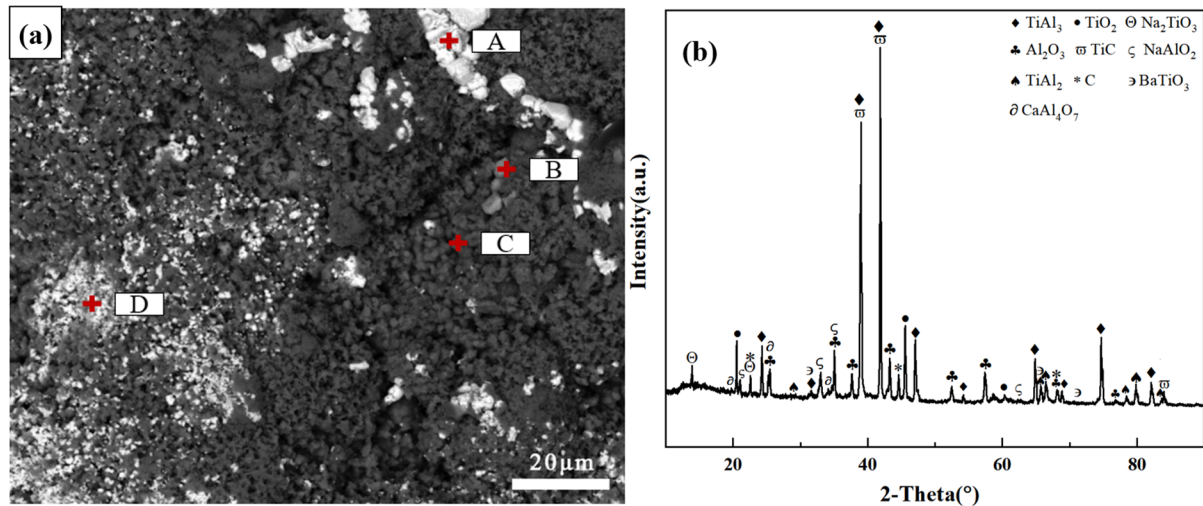


Figure 8. Surface morphologies (a) and XRD patterns (b) of the hot-dip aluminum coatings after corrosion in NaCl salt at 810 °C for 120 h. The “capitalized letters/red +” is the location of EDS point scanning.

Table 4. EDS results for the regions marked in Figure 8a (at.%).

Point	Ti	Al	C	O	Cl	Na	Ca	Ba
A	11.89	3.22	8.46	65.37	/	/	1.64	9.42
B	24.68	17.94	/	36.70	/	1.19	19.49	/
C	0.64	29.58	7.36	61.37	0.30	0.61	/	0.14
D	4.63	25.58	4.07	60.90	/	0.94	/	3.88

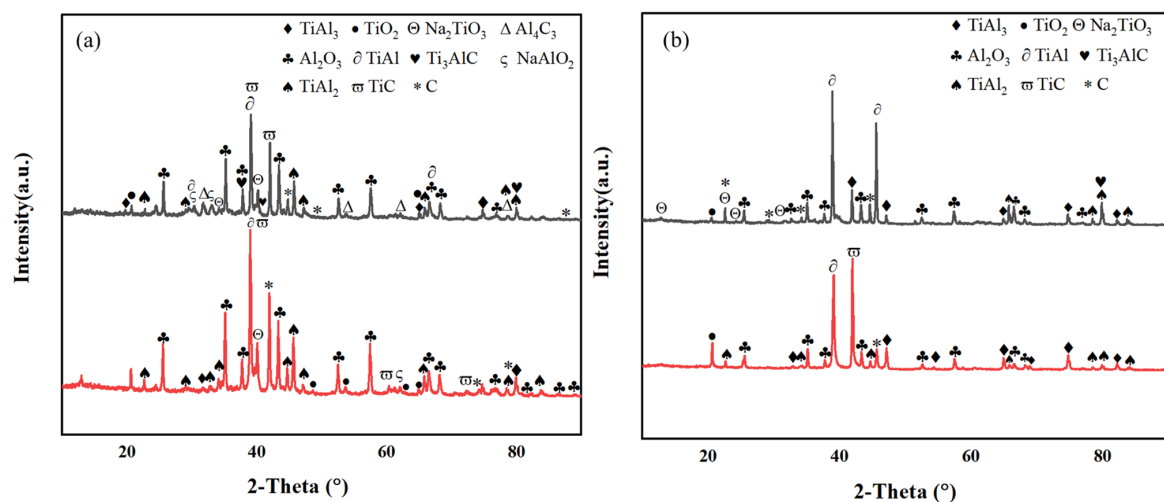


Figure 9. XRD patterns (after the removal of a certain surface layer) of the hot-dip aluminum/carburizing composite internal coatings after corrosion in NaCl salt at 810 °C for 5 h (a) and 120 h (b), respectively. The black line refers to the XRD diffraction picture after grinding off a layer of the surface, and the red line is the XRD diffraction map after grinding off a layer based on the red line.

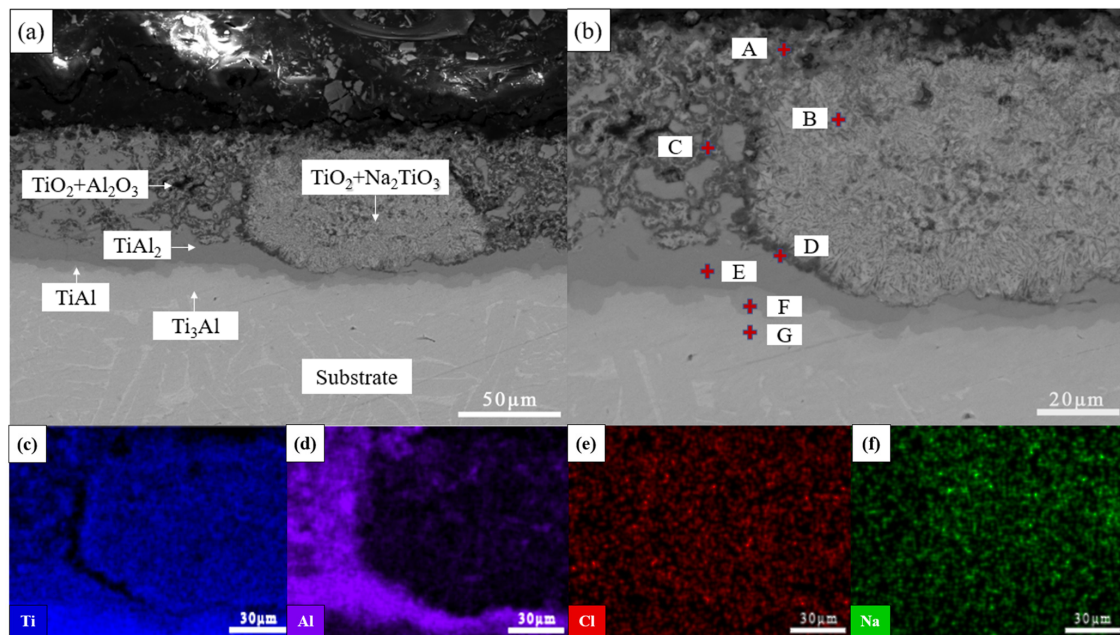


Figure 10. The cross-sectional morphologies and element mappings of the hot-dip aluminum/carburizing composite coatings in NaCl salt at 810 °C for 120 h. (a) is overall view of the microstructure; (b) is locally enlarged image; (b) is Ti; (d) is Al; (e) is Cl; (f) is Na. The “capitalized letters/red +” is the location of EDS point scanning.

Table 5. EDS results for the regions marked in Figure 10b (at.%).

Point	Ti	Al	O	Cl	Na	Possible Phase
A	21.96	15.58	60.71	/	1.75	$\text{Al}_2\text{O}_3 + \text{TiO}_2$
B	19.47	10.39	67.10	/	3.05	$\text{TiO}_2 + \text{Na}_2\text{TiO}_3$
C	18.21	30.96	50.83	/	/	$\text{Al}_2\text{O}_3 + \text{TiO}_2$
D	9.50	29.00	60.20	0.64	0.66	$\text{Al}_2\text{O}_3 + \text{TiAl}_2$
E	34.33	65.57	/	/	/	TiAl_2
F	46.85	53.15	/	/	/	TiAl
G	80.36	19.64	/	/	/	Ti_3Al

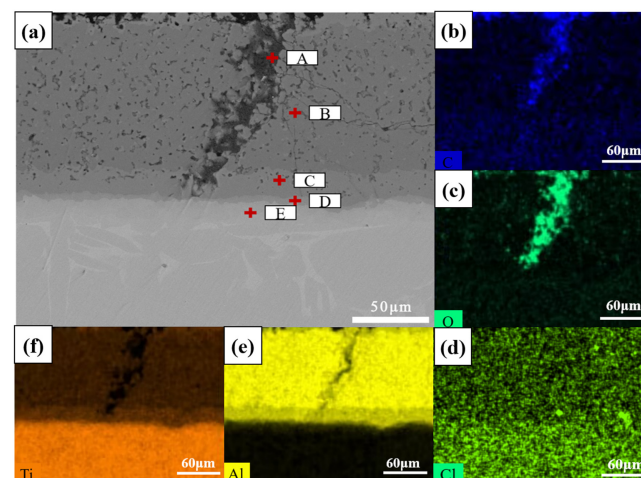


Figure 11. The cross-sectional morphologies and element mappings of the hot-dip aluminum/carburizing composite coatings in NaCl salt at 810 °C for 120 h. (a) is microstructure image; (b) is C; (c) is O; (d) is Cl; (e) is Al; (f) is Ti. The “capitalized letters/red +” is the location of EDS point scanning.

Table 6. EDS results for the regions marked in Figure 11b (at.%).

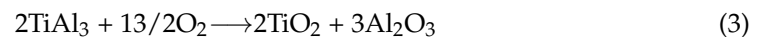
Point	Ti	Al	C	O	Possible Phase
A	4.87	32.24	15.27	47.62	Al ₂ O ₃ + C
B	24.54	65.42	10.02	/	TiAl ₃
C	37.77	62.23	/	/	TiAl ₂
D	49.96	43.40	6.64	/	TiAl + C
E	77.82	22.18	/	/	Ti ₃ Al

4. Discussion

From the above-mentioned results, it can be clearly concluded that both the hot-dip aluminum composite coating and the hot-dip aluminum/carburizing composite coating can protect Ti65 titanium alloys when exposed to a molten salt environment. The molten salt corrosion resistance of the hot-dip aluminized/carburized composite coating and the hot-dip aluminized coating were similar, and both were much higher than that of Ti65 alloy.

4.1. The Microstructures of the Hot-Dip Aluminum Coating

The hot-dip aluminum coating consists of a TiAl₃ phase near the substrate and a mixed layer containing TiAl₃ and pure Al [42,45]. During the corrosion experiment, the Al and TiAl₃ were first oxidized generating TiO₂ and Al₂O₃ as follows:



A layer of Ti-Al (TiAl₂, TiAl, Ti₃Al) could also be formed at the interface between the coating and Ti65 substrate, which was mainly due to the outward diffusion of Ti atoms from the substrate and the inward diffusion of Al atoms during the corrosion process. The interfacial reaction is illustrated below:



The Ti-Al (TiAl₂, TiAl, Ti₃Al) layers were compact, uniform structures, which effectively blocked the penetration of the atoms/particles [51].

Given that the melting point of NaCl(s) is 801 °C, NaCl salt maintained a melted state on the coating at the test temperature of 810 °C, leading to the dissolution of oxide scales as follows:



However, no diffraction peaks of corrosion products NaAlO₂ and Na₂TiO₃ were detected in the XRD results, indicating the content of NaAlO₂ and Na₂TiO₃ was relatively low. Furthermore, the corrosion products detected using XRD were mainly composed of TiAl₃, showing that the coating still presents excellent corrosion resistance. More importantly, the reaction between TiAl₃ and O₂ can be considered as the main mechanism of hot-dip aluminum coating in hot corrosion environments.

4.2. The Microstructures of the Hot-Dip Aluminum/Carburizing Composite Coating

For the hot-dip aluminum/carburizing composite coating, the molten salt corrosion products detected using XRD were mainly composed of Al₂O₃ and TiAl₃. During the initial stage of the hot corrosion test, the free carbon atoms in the outermost layer were oxidized, causing the formation of pores through the scale. These defects provided a short-circuit

diffusion channel for the inward diffusion of the corrosive medium and thus contributed to accelerating the whole corrosion process. The reactions can be written as follows:



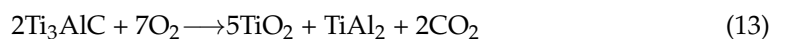
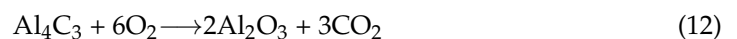
Subsequently, the oxygen and NaCl particles would gradually penetrate the coating. However, previous work performed by Ciszak reported that the initially formed TiO_2 would be promptly destroyed in the early stage of its emergence because the attack of chloride ions created a porous structure, which could not protect the underlying coatings from corrosion [52]. The oxides generated on the coating surface could react with NaCl as follows Equations (6) and (7).

The occurrence of reaction (6) coating covered with NaCl salts leads to the dissolution of oxide scales. Na_2TiO_3 and NaAlO_2 were evidently detected in the hot-dip aluminum/carburizing composite coating based on the XRD pattern (Figure 7b) and the EDS results (Table 4). Furthermore, $\text{Na}_x\text{Ti}_y\text{O}_z$ corrosion products have been reported to exhibit a porous structure and fail to protect the underlying coating from corrosion [53,54]. On the other hand, growth stress and thermal stress were easily released for the porous structure [55].

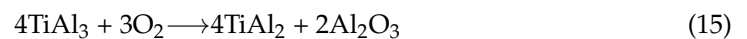
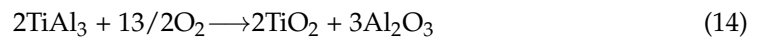
With the dissolution of oxide scales with NaCl, the oxygen and the Cl_2 seeped further through the porous structure leading to severe internal oxidation. Some TiC reacted with oxygen to form titanium dioxide and carbon dioxide, while other TiC reacted with chlorine to form TiCl_4 and carbon. The carbon was oxidized into carbon dioxide. The reaction is illustrated below [56,57]:



Meanwhile, Al_4C_3 and Ti_3AlC quickly oxidized with oxygen to form mixed oxides, such as TiO_2 , CO_2 , and TiO_2 .



The pores formed by the loss of the C atoms provide a fast path for the further penetration of corrosives, leading to oxidation inside the matrix. At the late corrosion stage, the new Al_2O_3 oxide scales would continue to form because of the high contents of Al atoms in coating.

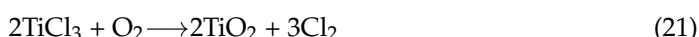


However, the presence of TiO_2 particles interrupted Al_2O_3 continuity, consequently resulting in the formation of loose and porous corrosion regions of TiO_2 with clear voids and micro-channels. Moreover, the chloride passed through the vacancy and micro-channel dissolving the oxide scales, as in Equations (6) and (7).

In addition, with an increase in the molten salt corrosion time, the oxide scales were continuously consumed so that the Al content in the coating was insufficient to form Al_2O_3 scales. Once the reactions in Equations (6) and (7) occurs, some of the resulting Cl_2 diffused through the porous oxide scales and molten salt, corroded the alloy, and then reacted with Al and Ti to form volatile metal chlorides at the oxide scale/metal interface. The accumulation of these volatile metal chlorides with high saturated vapor pressure at the corrosion scale/aluminizing coating interface resulted in the generation of cracks. The reaction can be written as:



The volatile chloride produced by the reaction would be diffused outward with the inward diffused oxygen as their thermodynamic stability is not as good as the corresponding oxides [58], which can be expressed as follows:



In this way, gaseous Cl_2 is produced again, so the reaction process occurring in NaCl-covered hot-dip aluminum/carburizing composite coatings can be regarded as the self-sustainable ox chlorination, which is consistent with previous research [16,59,60]. However, during the carburization process, the existence of C atoms, TiC, Al_4C_3 , and Ti_3AlC can promote the formation of pores on the coating. Moreover, it can be deduced that a large amount of CO_2 was also generated during molten salt corrosion. Meanwhile, TiC and Ti_3AlC were oxidized to porous and non-protective TiO_2 , leading to a poor hot corrosion resistance compared to hot-dip aluminum coating.

5. Conclusions

The composite coating of hot-dip aluminum/carburizing composite coating was successfully prepared on Ti65 titanium alloy using a two-step method for hot dipping aluminum and solid carburizing. The intermetallic compound layer can be divided into two layers after aluminizing: the inner layer, consisting mainly of Ti_3Al , and the outer layer, composed of granular TiAl_3 and a mixed layer of pure Al filled between the particles. During the carburization process, aluminum elements continue to diffuse to the substrate, and the titanium elements in the substrate continue to diffuse to the coating during carburization, forming a series of hot-dip aluminum alloy layers (TiAl_2 , TiAl , Ti_3Al) from the outside to the inside. The coating consists of compounds such as TiC, Al_4C_3 , Ti_3AlC , and free carbon atoms. This results in the formation of a composite coating of hot-dip aluminum /carburizing. According to the corrosion behavior of Ti65 titanium alloy, hot-dip aluminum coating, and hot-dip aluminum/carburizing coating in NaCl salt, the following conclusions can be drawn:

- (1) Both hot-dip aluminum coating and hot-dip aluminum/carburizing coating can effectively improve the corrosion resistance of sodium chloride molten salt. However, the hot-dip aluminum coating exhibits superior corrosion resistance in NaCl salt at 810 °C compared with the hot-dip aluminum/carburizing coating.
- (2) For the hot-dip aluminum coating, after molten salt corrosion for 120h, the outer layer mainly forms a mixture of TiAl_3 , Al_2O_3 , and TiO_2 , while the inner layer forms a layered structure of $\text{TiAl}_2/\text{TiAl}/\text{Ti}_3\text{Al}$. The mixture of TiAl_3 and Al_2O_3 in the outer layer plays a good protective role. In addition, the inner $\text{TiAl}_2/\text{TiAl}/\text{Ti}_3\text{Al}$ was well bonded to the substrate and was smooth without cracking.
- (3) For the hot dip-aluminum/carburizing composite coating, carbon exists in the coating as free carbon atoms, TiC, and Ti_3AlC . During the process of molten salt corrosion, C is oxidized into CO_2 gas, leading to the formation of high-concentration pores in

the coating, which becomes a channel for molten salt ions and oxygen penetration. In addition, TiC and Ti₃AlC promote the formation of non-protective oxide TiO₂, resulting in a deterioration in the corrosion resistance and a self-catalytic corrosion mechanism.

- (4) The results provide a basic understanding on the corrosion behavior of hot-dip aluminum/carburizing composite coatings in molten salt environments. In addition, the identified corrosion mechanism of the coating lays a good foundation for improving the marine environmental resistance of titanium alloys.

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