

Article

Anticorrosion Properties of Zn–Al Composite Coating Prepared by Cold Spraying

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Received: 15 January 2019; Accepted: 21 March 2019; Published: 25 March 2019



Abstract: In order to slow down the corrosion and wear of offshore equipment, the Zn–Al composite coating was prepared on Q345 substrate by cold spray technique. The mass fraction of Zn and Al in the raw material was 2:3. The microstructure of the original coating was observed by scanning electron microscopy (SEM) and was characterized by energy dispersive spectrometer (EDS). From the composite alloy coating obtained by cold spraying, it was observed that the Zn and Al particles were uniformly distributed without oxidation product, and the powder particles were significantly plastically deformed. The microstructure of the composite coating is very dense and has strong adhesion to the substrate. Neutral salt spray test (NSS) and electrochemical accelerated corrosion test results showed that Zn–Al composite coating can effectively provide corrosion protection.

Keywords: cold spray; Zn–Al composite coating; corrosion; microscopic morphology

1. Introduction

In the context of large-scale development and utilization of marine resources, marine transportation, fisheries, and maritime military are inseparable from offshore equipment. However, a large amount of natural electrolytes forms a corrosive environment, which causes serious damage to the surface of offshore equipment. As a result, corrosion and fouling bring great losses to the Chinese. Therefore, it is of great significance to effectively protect metal materials in the marine environment. The research on marine metal materials has also received extensive attention from scholars [1–5].

At present, the surface treatment methods commonly used on the surface of marine equipment include thermal spraying, organic coating, hot dip plating, and the like [6–9]. In the preparation of thermal spray coating, it becomes semi-molten or molten during the thermal spraying process. The alloy coating will grow and oxidize to some extent, and the porosity of the coating will be high with large thermal stress. It will aggravate the corrosion of the entire surface of the entire offshore equipment, resulting in greatly shortened equipment life; organic materials are easily volatilized during the coating process, not only polluting the environment, but also aging the organic coating. Meanwhile, it is also easy to be washed away and scratched in the spray splash area. In recent years, cold spray technology has been used to prepare anti-biofouling and anti-corrosion coatings for offshore equipment, it is mainly used for anticorrosion protection of steel structures and reinforced concrete structures such as coastal facilities, wharves, trestles, lighthouses and buoys. Cold spray technology has shown good application prospects in surface treatment in this field.

Cold spray technology, also known as cold gas dynamic spray, is a new type of spray technology that has been rapidly developed in recent years. It uses compressed gas (nitrogen, helium and air, and etc.) as an accelerated gas stream to drive powder particles (size of 1–50 μm) to collide with the

substrate at a low temperature (room temperature to 600 °C) and a supersonic speed (300–1200 m/s) to complete solid state, so that the particles will undergo a strong plastic deformation and deposit to form a coating [10–16]. Compared with the traditional painting, thermal spraying and other processes of offshore equipment, the low-temperature characteristics of cold spraying has the following advantages: (1) The cold working environment has a low temperature, and it results in a fast deposition coating speed, so there is a wide range of substrate can be applied. It is portable to the entire surface of offshore equipment and is also suitable for the surface of equipment of various bases. (2) The thermal effect on the substrate and the powder particles is negligible, and the oxidation, burning loss, phase transformation and tissue change of the spray powder can be avoided. Meanwhile, the coating of the heat sensitive material such as the nanometer amorphous material is obvious. As a result, the advantage is that there are fewer oxidation products during the preparation process [17–21]. (3) Forming a coating has a low porosity and high density. Cold spraying in the working process, the subsequent particle can effectively combine with the front deposition coating, and the structure of the coating is also relatively dense. At the same time, the temperature of the coating prepared by cold spraying will not change significantly, so it will not have a thermal expansion and contraction of the coating volume, which can greatly reduce the void ratio of the coating. (4) The coating has a strong protection performance. Compared with the traditional organic coating, the cold spray coating not only is resistant to wear, and it can also can effectively carry out the electrochemical protection of the offshore equipment base in the case of wear of the coating [22].

The initial results show that zinc layer provides the best electrochemical protection for steel [23–25], but its long-term protection against high-chlorine or offshore steel structures is not good. Aluminum is well-stabilized [26], and thermal sprayed Al coatings have higher hardness, longer durability and higher erosion resistance [27], but aluminum coatings are less electrochemically protective than zinc coatings. The Zn–Al coating [28–31] is a promising alloy coating. Studies have shown that the Zn–Al coating prepared by cold spraying has good thermal stability, corrosion resistance, compactness, and the bonding strength is greater than the bonding strength of the substrate. Although there are many studies on Zn–Al coatings, the working environment and experimental conditions are different, and the application direction is different. On the one hand, the cold sprayed Zn–Al composite coating described in the article is applied to offshore equipment, and the coating has the advantages of the above cold spraying. On the other hand, this study focuses on the marine working environment, and the advantages of the composite coating are also shown in the experiment. Therefore, the purpose of this study was to investigate the corrosion resistance properties of Zn–Al composite coatings in order to facilitate their application in offshore equipment.

2. Experimental Methods

The raw materials used in this study were spherical Zn and Al powder particles, whose size was 1–50 µm, and the mass fraction of Zn and Al powder was 2:3 and the mixture was stirred for 1 h in a ball mill to obtain the mixed powder of sprayed raw materials. The base material is made of 20 mm × 60 mm × 2 mm Q345 steel (a low alloy steel widely used in bridges, vehicles, ships, construction, pressure vessels, special equipment, etc.) plate, and the surface is sandblasted, scaled and cleaned.

The coating was prepared using a cold spray technique (DyMET423, Beijing Chuangxuan Bide Tech&Trade Ltd, Beijing, China). At the center of the cold spray equipment is gun mechanism, which is equipped with a high-pressure gas pipeline, a heater, a powder feeding pipe and a de Laval nozzle (2 mm in diameter). The overall gun mechanism is mounted on a six-degree-of-freedom robot arm and the compressed power gas is nitrogen. The pressure is 1.8 MPa and the working temperature is 400 °C. The spray gun is 20 mm away from the spray substrate and the powder particles are sprayed at 800 m/s.

The apreo scanning electron microscopy (SEM, FEI, Brno, Czech Republic) was used to study the microscopic morphology of the composite coating. The surface morphology of the coating, the plastic

deformation of the Zn–Al powder particles, the pores of the coating, and the mixing of the spray of Zn–Al can be observed by monitoring the microstructure of the coating by SEM. In addition, the cross section of the coating can also be intuitive and the state of bonding of the coating to the substrate can be observed. The energy dispersive spectrometer (EDS, FEI, Brno, Czech Republic) is used to measure the different features of the coating and the distribution of elements.

The scratch function of the multi-function material surface performance tester (MFT-4000, Lanzhou Huahui Instrument Technology Co., Ltd, Lanzhou, China) was used to determine the adhesion of the composite coating. In the scratch test, a 120° stylus probe was selected, and the stylus loading speed was set to 100 N/min, the scratch length was 10 mm, and the termination load was 100 N.

The composite coating was investigated by neutral salt spray (NSS, LRHS-108-RY, Shanghai Linpin Instruments Co., Ltd, Shanghai, China) and electrochemical accelerated corrosion tests method to evaluate the corrosion resistance of the composite coating. A sample having a surface area of $10 \times 10 \text{ mm}^2$ is prepared before the test. Neutral salt spray test is performed in a static environment, where the ambient temperature of the salt spray is stable at $35 \pm 1^\circ\text{C}$, and 100 mL of $3.5 \pm 0.5\%$ NaCl solution at 100% relative humidity is prepared, and its pH is between 6.5 and 7.2. The neutral salt spray test equipment used is model LRHS-108-RY and it includes an atomizing nozzle to atomize the salt solution in the chamber, the salt solution pool; the sample supports the vertical direction of 15° to 20° ; and the heating element, the controller, and the humidification tower are connected to the reservoir. In the electrochemical accelerated corrosion experiment, sealing the uncoated surface for the accuracy of the test, and the polarization curve is obtained by scanning. The non-coated base steel Q345 sample was first immersed in NaCl solution for 24 h to test Tafel curve. Taking the coating samples and putting them into NaCl solution for 24, 48, 72, 96 h, and then the Tafel curve test was carried out with a scan frequency of 0.5 mV/s. The I_{corr} was obtained by using Butler Volmer analysis (ThalesXT5.1.4 software), and test data is recorded each time.

The hardness of the coating was measured by using an automatic turret digital microhardness tester (HXD-1000TM, Shanghai Precision Instruments Co., Ltd, Shanghai, China). The indenter was selected from Vickers diamond, and the program was self-controlled to maintain the load mode. The load was 0.4903 N and the time was 20 s. Each sample in different time periods was randomly selected to test five points on the surface of the coating, and the microhardness of the coating was replaced by the average value.

3. Results and Discussion

3.1. Microstructure of Zn–Al Composite Coating

Figure 1 shows a typical surface topography of a Zn–Al composite coating seen from the SEM micrograph. As shown in Figure 1a, the composite coating is magnified $50\times$, and it can be visually observed that the distribution of the two metal powders in the layer is uniform. It indicates that the mixing of the Zn and Al powder particles is uniform; as shown in Figure 1b, the composite coating is enlarged by 1000 times, and two kinds of bright and dark elements can be clearly observed; as shown in Figure 1c, upper part is a composite coating, and the lower part is the base. As a whole, the composite coating is densely structured without obvious porosity. Zooming in on the $500\times$ section, it was found that the bright and dark powders were in the shape of a ribbon, distributed evenly and with few voids, and closely adhered to the substrate. According to the two angles of Figure 1b,c, it was found in the coating, the two metal powder particles showed a flat shape, and the Zn and Al powder particles were severely plastically deformed when sprayed onto the substrate, thereby obtaining a dense lamellar structure.

The mass ratio of zinc to aluminum in the composite coating is 2:3, but the density of zinc is greater than aluminum, so the volume of aluminum is larger than the zinc. We guess that the main component of the bright region in the coating is zinc, and the main component of the dark region is aluminum. The EDS map analysis was performed by dot to verify the guess.

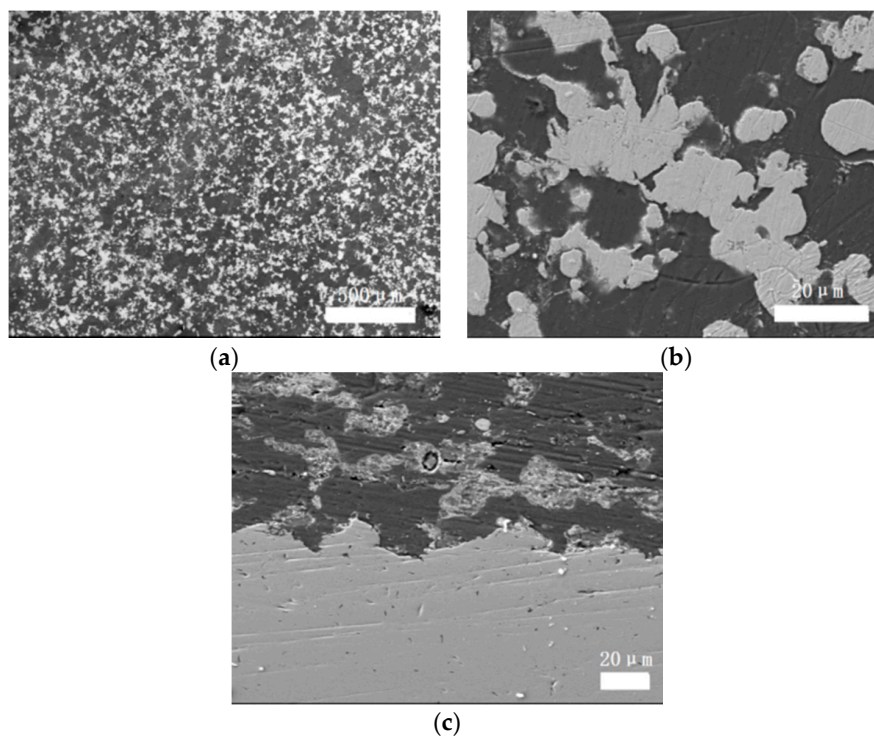


Figure 1. (a) Enlarged 50 $\times$ view of the coating, (b) enlarged 1000 $\times$ view of the coating, bright region is zinc and dark region is aluminum, (c) enlarged 500 $\times$ view of the interface, upper part is a composite coating and the lower part is the base.

Figure 2 is a spectrogram measured by an energy dispersive spectrometer (EDS), Table 1 chemical compositions of the Zn–Al coating according to EDS analyses. According to Points 1 and 2, the zinc content in the coating at Point 1 is 85.9%, and the aluminum content is only 1.6%; at Point 2, the zinc content in the coating is only 0.1%, and the aluminum content is 90.6%. Therefore, it can be concluded that the bright metal portion is a zinc-rich group and the dark metal portion is an aluminum-rich group. It is also shown from the energy spectra at Points 1 and 2 that there is almost no oxidation of the composite coating sprayed by the cold spray.

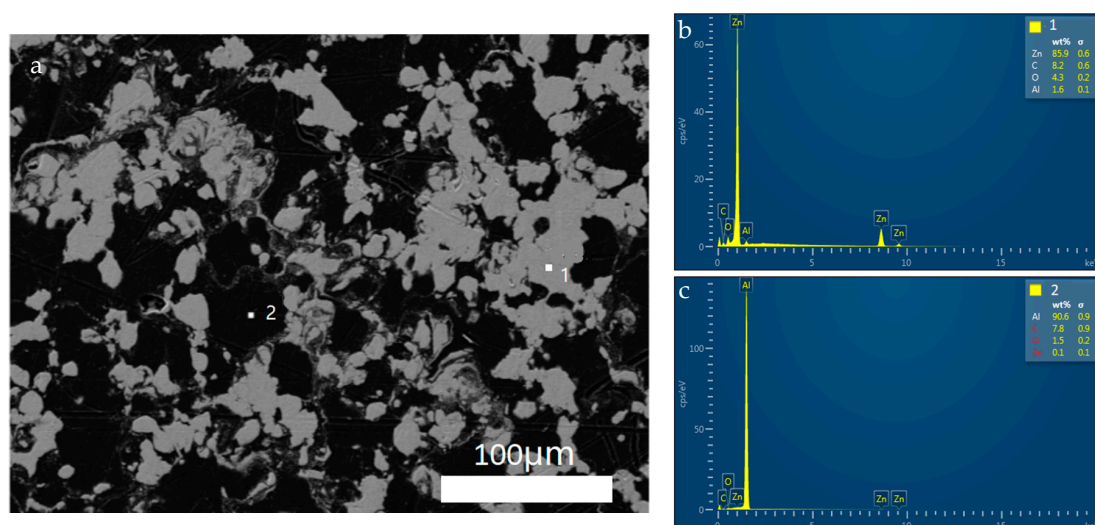
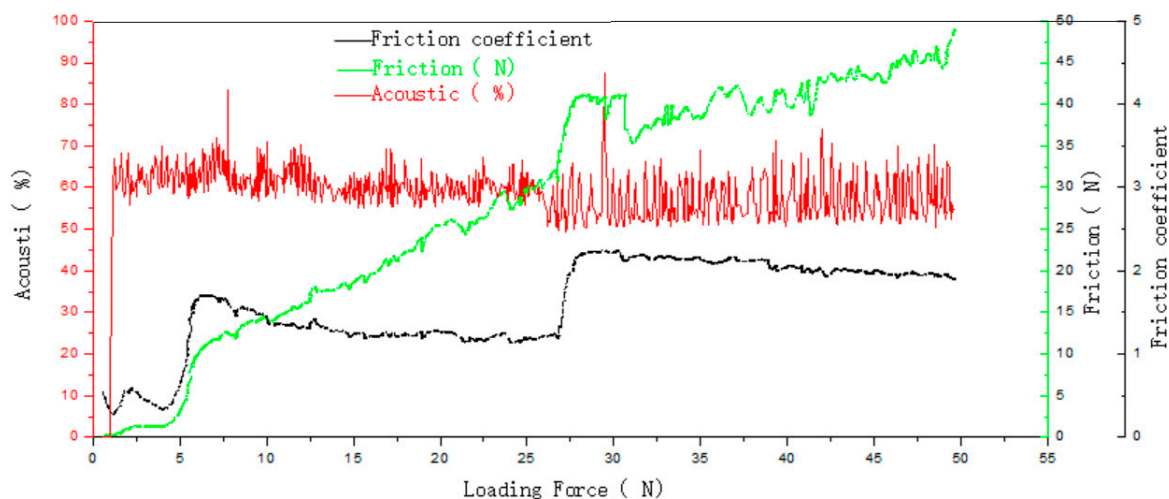


Figure 2. Energy dispersive spectrometer (EDS) dot spectrogram: (a) enlarged 1000 $\times$ view of the coating; (b) 1 dot spectrogram; (c) 2 dot spectrogram.

Table 1. Chemical compositions of the Zn–Al coating according to EDS analyses.

HEACs	Regions	Elements (at.%)			
		Zn	Al	O	C
Zn–Al coating	1 (bright)	85.9	1.6	4.6	8.2
	2 (gray)	90.6	0.1	7.8	1.5

The scratch test data of the MFT 4000 multi-functional surface performance tester is shown in Figure 3, where the red line indicates the acoustic signal, the green line indicates the friction, and the black line indicates the friction factor. When the applied load exceeds the adhesion strength between the coating and the matrix, the coating is scratched, and the acoustic signal, friction factor and the frictional force fluctuate violently at the same time. As shown in the experimental data in Figure 3, the acoustic signal has two mutated regions. Each mutation represents that the needle is drawn into the material of different components, and it results in an increase in friction factor and friction. The first mutation loading force is about 10 N, and it is observed that the scratcher breaks the coating oxide film, the second mutation loading force was about 55 N, and the scratcher can be determined to break the composite coating. The adhesion of the composite coating obtained during the test is 55 N, so that the composite coating could be firmly attached to the surface of the substrate.

**Figure 3.** Scratch test of composite coating.

In general, the Zn–Al composite coating prepared by the cold spray technique has uniform distribution of Zn, Al powder particles and it has a compact structure with no oxidation.

3.2. Corrosion Behavior of the Coating

The electrochemical accelerated corrosion test measure the open circuit voltage and the polarization curves at different times, as shown in Figure 4 below. The corrosion potential (E_{corr}) characterizes the thermodynamic stability of the tested samples under the electrochemical corrosive condition [32]. The corrosion current density (I_{corr}) implies that the corrosion rate and breakdown potential are the lowest potential values at which pitting occurred [33]. The corresponding electrochemical parameters of tested samples are all listed in Table 2. Figure 4 and Table 2 show that the polarization potential of the base steel Q345 is -1.992 V, and the corrosion current density is $6.589 \times 10^{-5} \text{ A}\cdot\text{cm}^{-2}$. Figure 4 and Table 2 also show the accelerated corrosion plan of 24, 48, 72, and 96 h. As the coating's corrosion potential (E_{corr}) gradually increases from -1.396 to -1.325 V, the current density gradually decreases from 1.737×10^{-4} to $1.154 \times 10^{-4} \text{ A}\cdot\text{cm}^{-2}$, and the corrosion rate can be gradually slowed down. The lower I_{pass} illustrated that passivation behavior could occur more easily, and the passivation film was formed rapidly. By comparing the relevant corrosion parameters of

the base steel Q345 and the coating, it was found that the polarization potential of Q345 was -1.192 V, much smaller than the corrosion potential of the coating (-1.396 V), so the coating and the base steel Q345 together corrode the coating first. It has the function of protecting the base steel. The corrosion current density of the base steel Q345 was also much smaller than the corrosion current density of the composite coating, so the Q345 corrosion rate is slower than the corrosion rate of the composite coating. Besides, the passivation films formed on the coating surface prevented the inner layer from being exposed to corrosive liquid, which could slow down the corrosion reaction. Compared with the tested samples, the 96-hour corrosion-resistant coating had a lower pass rate of $1.154 \times 10^{-4} \text{ A}\cdot\text{cm}^{-2}$ than the other samples, showing good passivation. The reason might be that the coating began to experience a vigorous electrochemical reaction in the NaCl solution, which generated an oxidation product that hindered the rate of corrosion. To probe deeper into the corrosion resistance of the coating, the NSS test was conducted.

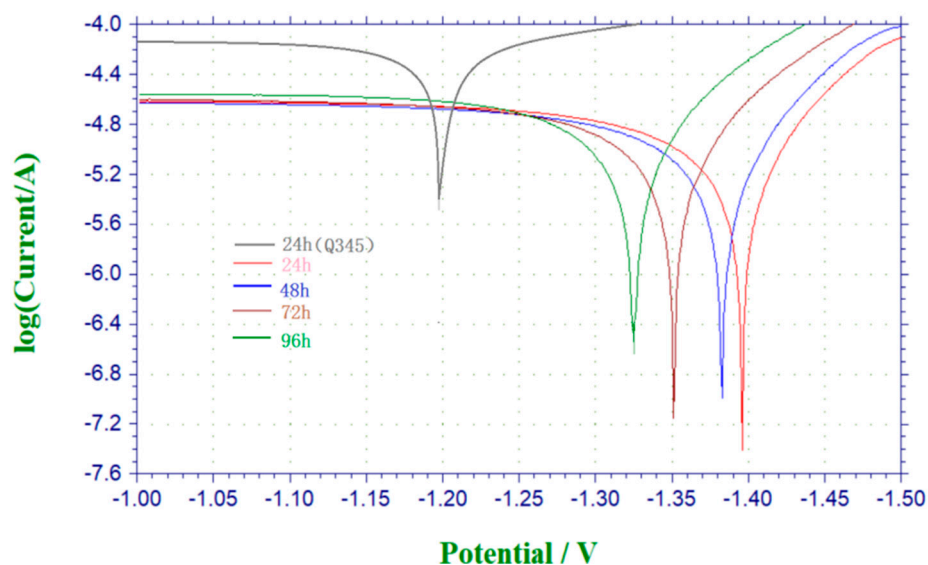


Figure 4. Potentiodynamic polarization curves of the Q345 steel substrate, Zn–Al coating in 3.5 wt.% NaCl solution.

Table 2. Fitted parameters of polarization curves for the Q345 after immersion of 24 h and the Zn–Al coatings after immersion of 24, 48, 72 and 96 h in 3.5 wt.% NaCl solution.

Times/h	E_{corr} (VSCE)	I_{corr} ($\text{A}\cdot\text{cm}^{-2}$)	I_{pass} ($\text{A}\cdot\text{cm}^{-2}$)	B_a (mVdec^{-1})	B_c (mVdec^{-1})
24 (Q345)	-1.192 ± 0.03	6.589×10^{-5}	8.37×10^{-4}	3.392	3.947
24	-1.396 ± 0.03	1.737×10^{-4}	2.39×10^{-3}	2.743	8.729
48	-1.384 ± 0.04	1.456×10^{-4}	1.63×10^{-3}	2.571	8.134
72	-1.351 ± 0.01	1.248×10^{-4}	0.96×10^{-3}	2.397	8.222
96	-1.325 ± 0.02	1.154×10^{-4}	0.622×10^{-3}	2.375	7.915

Figure 5 is a typical corrosion morphology after corrosion in a 3000x neutral salt spray (NSS) test. According Figure 5a, which is corroded for 24 h, and the surface layer of the coating has a large number of agglomerate structures, and the voids have a dense needle-like and lamellar structure; Figure 5b is corroded for 48 h. A large number of needle-like and lamellar structures appear in the lumps; in Figure 5c, the flaky material on the surface of the coating gradually decreases, and the needles gradually increase; in Figure 5d, the surface of the coating is etched for 96 h. Cracks have occurred, and there are also a large number of needle-like, lamellar structures in the cracks.

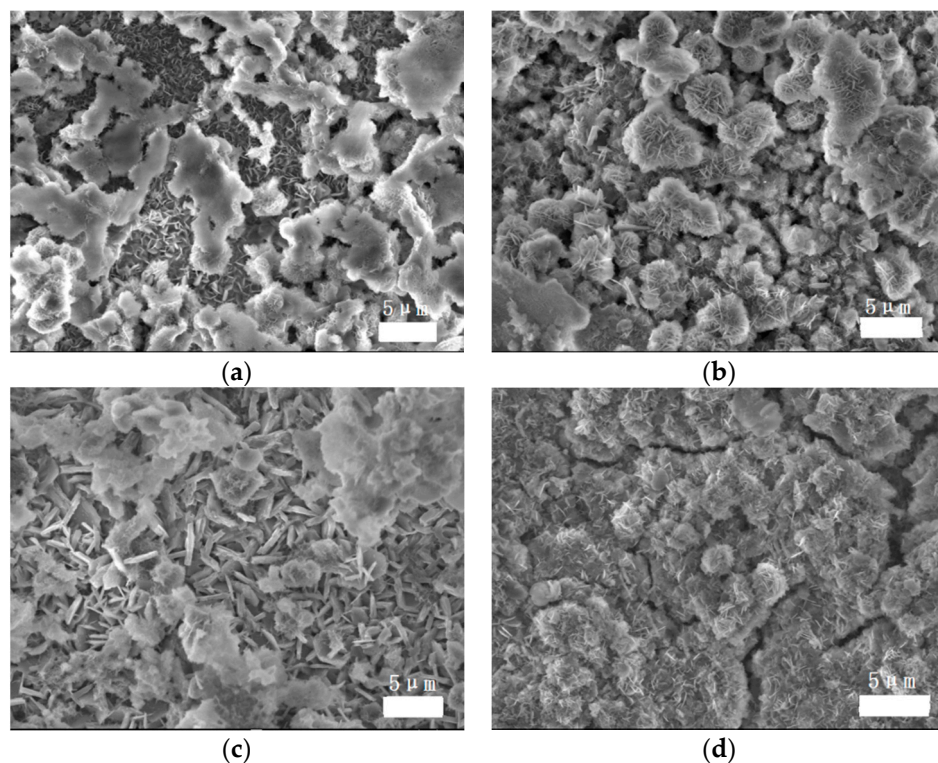


Figure 5. The surface of the coating is magnified $3000\times$ in different corrosion times: (a) 24 h; (b) 48 h; (c) 72 h; and (d) 96 h.

The EDS linear scanning corrosion coating is shown in Figure 6. It contains a large amount of Zn, Al, and Cl elements on the white agglomerates, and the needle-like morphology contains a large amount of Al elements, and the content of Zn elements is small. In Figure 6, Al is not corroded and remains in its original state. But the Zn is severely corroded, the resulting corrosion products are mainly basic zinc chloride hydrate and basic zinc carbonate hydrate. The crack appearing in Figure 5d is because the crack is a Zn-rich group, and Zn is preferentially corroded, so cracks are generated.

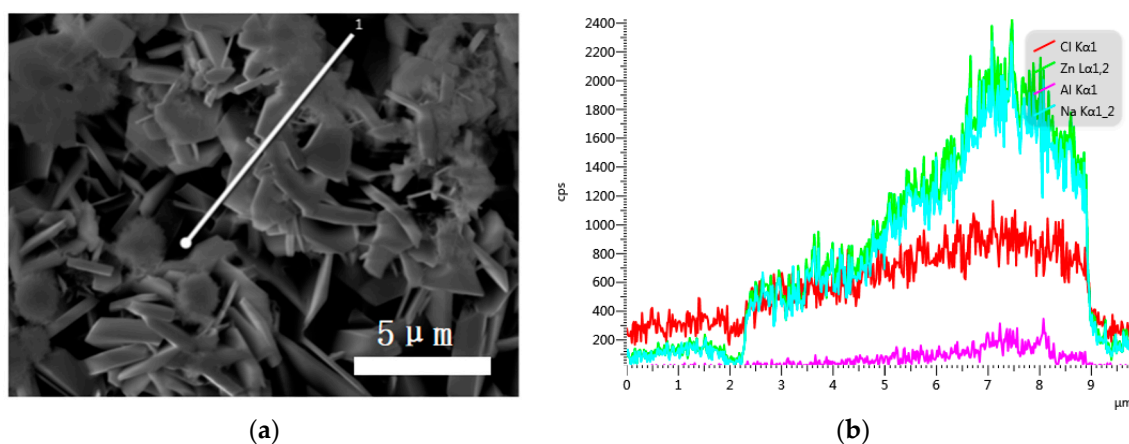


Figure 6. Coating EDS line scan after NSS test. (a) enlarged $1000\times$ view of the coating; (b) Coating EDS line scan element content.

Not only does the high hardness coating can improve its own hardness, but also it better resist the scratching of the surface by foreign objects. Table 3 shows that microhardness of coatings on different corrosion time, calculating the average microhardness of five points at the same time, and plotting the Figure 7. Figure 7 the line graph shows the surface hardness of the coating at different corrosion time.

The hardness of the uncorroded coating is about 78.14 HV, and it decreases slowly in the first 24 h. But corrosion increases with the corrosion time, the hardness of the coating also gradually increased. When the corrosion was 96 h, the hardness of the coating is 142.86 HV. The results show that the initial oxidation of the coating surface is accompanied by a decrease in the hardness of the coating. When the corrosion rate is slow and the oxide film was formed by corrosion passivation, the hardness of the coating is gradually increased. The greater the hardness of the coating, the stronger the ability to resist the pressing of hard objects into the surface.

Table 3. Microhardness of coatings on different corrosion time.

Corrosion Times/h	Microhardness (HV)				
0	79.6	75.2	77.6	80.5	77.8
24	80.6	60.1	70.6	68.3	73.4
48	81.0	91.7	77.5	87.3	85.8
96	132.0	122.3	163.8	150.5	145.7

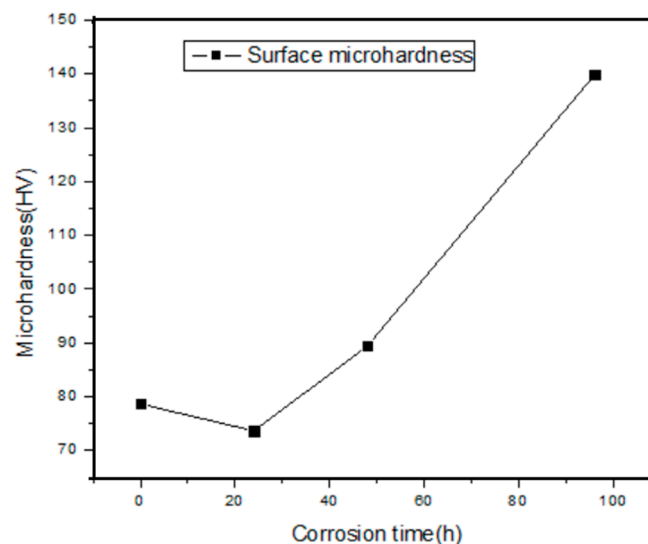


Figure 7. Microhardness of coatings on different corrosion time.

In summary, Figures 5 and 6 conclude that in the salt spray test, the Zn–Al composite coating will corrode Zn first, and a large number of clusters gradually corrode, then obtaining dense needle-like, lamellar oxidation. The product the rate of corrosion gradually slows over time, resulting in a passivation film that further reduces corrosion and thereby protects the coating matrix. Even if the coating are scratched, the composite coating has cathodic protection to the substrate. Therefore, the Zn–Al composite coating has good corrosion resistance.

4. Conclusions

In this experimental study, a Zn–Al composite coating was prepared on the Q345 substrate by cold spray technique. The corrosion resistance and other properties of the composite coating were studied by salt spray test, electrochemical test and hardness test. The following conclusions can be drawn:

- The Zn–Al composite coating prepared by cold spray technology can be firmly attached to the Q345 substrate. The energy dispersive spectrometer (EDS) analyzed the coating without significant oxidation during the preparation process. The SEM microstructure shows that the coating material, Zn, Al powder particles are uniformly mixed and good plastic deformation occurs.
- After the salt spray test and electrochemical test of the sample, the microstructure of the sample at different corrosion time periods showed that the sample had a severe anodization reaction at the

beginning of the accelerated corrosion, and a large amount of agglomerated oxidation products were formed. With the accelerated corrosion test, the oxidation products on the microscopic surface of the sample are gradually fine and dense, and the corrosion rate gradually slowed down. The oxidation product can effectively slow the corrosion of the sample surface. The corrosion surface hardness data measured by the hardness tester shows that the longer was the corrosion time in a certain range along the greater is the hardness of the surface, so the better is the wear resistance.

- The more positive E_{corr} values and lower I_{corr} values of the base material indicate improved corrosion resistance properties of the uncoated material. On the one hand, the composite coating isolates the substrate from contact with the marine environment, therefore, corrosion of the substrate in direct contact with seawater is avoided. On the other hand, even if the surface of the composite coating is scratched, the coating and the substrate are anodized in a closed loop in seawater, thereby protecting the substrate from corrosion. In summary, the composite coating increases the corrosion behavior of the sample compared to the base material alone.

Author Contributions: Conceptualization, S.W.; Methodology, X.L. and S.W.; Validation, X.L. and G.W.; Data Curation, S.W. and X.L.; Writing—Original Draft Preparation, X.L.; Writing—Review and Editing, S.W. and D.W.; Supervision, T.X. and H.D.; Project Administration, S.W.

Funding: This research was funded by the National Natural Science Foundation of China (Nos. 51872122 and 51705199), Shandong Key Research and Development Plan, China (No. 2016JMRH0218) Shandong Provincial Natural Science Foundation, China (No. ZR2017BEE055), Distinguished Middle-Aged and Young Scientist Encourage and Reward Foundation of Shandong Province (No. ZR2016EMB01) and Taishan Scholar Engineering Special Funding (2016–2020).

Conflicts of Interest: The authors declare no conflict of interest.

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