Research Article

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Study on nanocrystalline coating prepared by electro-spraying 316L metal wire and its corrosion performance

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Abstract: In this work, we study the corrosion performance of coatings prepared by electrical explosion spraying of metal wires. 316L metal wire with a diameter of 1.5 mm is used as spray material, and the coating is prepared on the 45# steel substrate by electrical explosion spraying. The oil–water corrosion experiment of the coating is carried out in a constant temperature water bath of 60°C for 168 h. The scanning electron microscopy and energy-dispersive spectroscopy results of the experimental samples have shown that some metal oxides are found inside the coating, most of which are distributed at the grain boundaries with a size range of 30–50 nm. The corrosion rate of the coating is measured by weight loss method with a corrosion rate of 0.079 mm/annum. XRD results show that the corrosion generates $CaCO_3$, Fe_3O_4, and $MgFe_2O_4$. Coating corrosion is mainly caused by the formation of electrochemical corrosion between oxides and non-oxides in the coating, and pitting corrosion and intergranular corrosion in the presence of chloride ions.

Keywords: electrical explosion spraying, coating corrosion, nano-oxides, kinetic curve

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1 Introduction

Steel materials are widely used in automotive, aerospace, transportation, and equipment manufacturing industries due to their good toughness, easy processing, and high strength. Most steel materials, however, have poor corrosion resistance and are easily affected by environmental factors, leading to corrosion under actual working conditions. The metal and equipment materials scrapped globally due to corrosion account for about 1/3 of the annual production, giving rise to direct economic loss of about 700 billion US dollars, which is 6 times the total loss caused by natural disasters, such as earthquakes, floods, and typhoons [1,2]. Among the current methods to avoid the corrosion of steel materials, coating is one of the most economical, simplest, and most effective methods to improve the corrosion resistance of steel materials [3–5].

The current methods in preparing corrosion-resistant coatings on steel materials mainly include electroplating, electroless plating, and thermal spraying by coating the surfaces of steel materials with alloys containing nickel, zinc, and chromium as corrosion-resistant materials [6,7]. The methods of electroplating and electroless plating can, however, only use soluble metals as coating materials, and the generated liquid wastes in electroplating process can cause environmental pollution, which increases the cost in environmental protection. Moreover, traditional thermal spraying technology will heat the surfaces of steel materials during the coating process, which affects inevitably the surfaces of the substrates, resulting in decreases in both compactness and bonding force of the coatings [7,8].

Electric explosion spraying of metal wire is a process of rapid melting, vaporization, and plasmaization of the metal wire through which a current density of higher than 10^7 cm^{-2} passes within pretty short time. Meanwhile, the material states and parameters of the metal wire change sharply accompanied by strong light radiation and explosion sound, which is thereby called the electric explosion [9–11].

Electrical explosion spraying, as a kind of thermal spraying, uses metal wires as spraying materials on which large current with high energy density is applied to generate ohmic heating. The metal wires rapidly undergo phase transition of solid → liquid → vapor → plasma, resulting in an electrical explosion. With the help of explosion shock wave, metal droplets that are partially molten but not vaporized are sprayed onto surfaces of workpieces at high speeds to form high-temperature resistant and anti-ablative coatings [12,13]. Due to its unique spraying method, electrical explosion spraying has good adaptability to the spraying in pipes and holes. The spraying speed is fast, normally finished in about 1 ms, thus posing negligible thermal effects on workpieces. The generated metal grains are mostly microcrystalline and nanocrystalline, which can greatly improve the strength plasticity, abrasion resistance, corrosion resistance, and other properties of materials [14].

Currently, electrical explosion spraying is mainly used in the preparation of nano-powders and abrasion-resistant coatings [15–18]. Mizusako successfully coated Mo, W, and stainless steel on aluminum and steel plates by carrying out electrical explosion spraying on inner holes of pipe fittings in the atmospheric environments [19]. Padgurskas *et al.* used the same method to prepare low friction coatings [20]. Romanov *et al.* improved abrasion resistance and galvanic corrosion resistance of surfaces of copper alloys with ZnO–Ag coatings prepared by electrical explosion spraying [21]. In addition, researchers have reported anti-corrosion coatings prepared by electrical explosion spraying to improve the corrosion resistance of substrates, which, however, lacks in-depth research to clarify their corrosion performance under certain conditions [22,23].

Unlike previous study, in this work, we prepare coatings of the spray material 316L metal wires on 45# steel substrates by electrical explosion spraying. A corrosion experiment is carried out on the coatings for 168 h. Based on the analyses of original structures, the diffusion of elements, the corrosion products, and the corrosion rate of the coating section after the corrosion test, we obtain the main factors affecting the corrosion performance of the coatings prepared by electrical explosion spraying.

2 Experiments

2.1 Preparation of the coatings

45# steel with a sample size of 10 mm × 10 mm × 20 mm was used as substrate with related composition shown in Table 1. 316L metal wires with a diameter of 1.5 mm were used as the spraying material with related composition shown in Table 2. Coatings were prepared by electrical explosion spraying with 20 times of spraying on the surfaces of the substrates under appropriate parameters of power supply. Figure 1 shows the working principle of electrical explosion spraying of metal wires.

Table 1: Chemical composition of 45# steel

Element	C	Si	Mn	Cr	Ni	Cu	Fe
Content (wt%)	0.45	0.21	0.6	0.18	0.25	0.1	Bal.

Table 2: Chemical composition of 316L steel

Element	C	Si	Mn	Cr	Ni	S	P	Fe
Content (wt%)	0.03	0.1	2.5	16	13	≤0.03	≤0.045	Bal.

Figure 1 shows the schematic diagram of working principle of electrical explosion spraying, where t1 is the charging module, d1 is the diode, r1 is the resistance value of the metal wire, c is the capacitance value of the electric quantity released during the electric explosion spraying of metal wires, S is the switch, L1 is the energy storage inductance, and E is the electrical explosion wire.

2.2 Characterization of the coatings

The phase of the coatings was detected by X-ray diffraction (XRD; smartlab, Rigaku) with a scanning angle of 20–90° and a speed of 2θ/min. The surfaces and cross-sectional morphologies of the coatings were characterized by scanning electron microscopy (SEM; Zeiss Sigma300, Germany) and energy-dispersive spectroscopy (EDS; OXFORD X-MAX^N). The bonding force between the coatings and the substrates

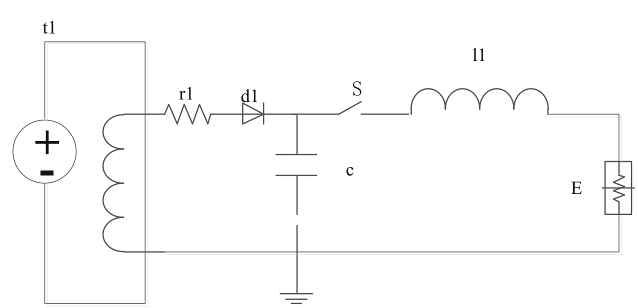

Figure 1: Schematic diagram of working principle of electrical explosion spraying.

was measured with a film FM1000 and a universal testing machine. The microhardness of coating was measured by automatic microhardness tester (Q10A+, Austria).

2.3 Corrosion test

The surfaces of the samples were polished smooth. The unsprayed surfaces were covered with epoxy resin and the boundary was sealed with silica gel. Then, the sample was immersed in the corrosion solution for 168 h at a constant temperature of 60°C in water bath. The components of the corrosion solution are shown in Table 3. The experiment was carried out according to NACE TM0284-2003 standard. The sample was weighed every 24 h, based on which the kinetic curve was plotted.

Table 3: Components of medium that mimics oilfield conditions (g/L)

Compound	Concentration	Ionic form	Content
$CaCl_2$	1.665	Ca^{2+}	0.6
$NaHCO_3$	0.826	HCO_3^-	0.6
NaCl	30.715	Cl^-	20
Na_2SO_4	1.775	SO_4^{2-}	1.2
$MgCl_2{\cdot}6H_2O$	0.846	Mg^{2+}	0.1

3 Results

3.1 Properties of the coatings prepared by electrical explosion spraying

The macro-morphology and micro cross-sectional morphology of the coatings can reflect quality of the prepared coatings. Figure 2a shows the macro-morphology of base material, and Figure 2b and c shows the macro-morphology and micro cross-sectional morphology of 316L metal wire coatings prepared by electrical explosion spraying, revealing that the metal coatings with white metallic luster are deposited densely and uniformly on the surfaces of the substrates with a thickness of about 125 μm. The coatings have been tested by thermal vibration (heating to 350°C

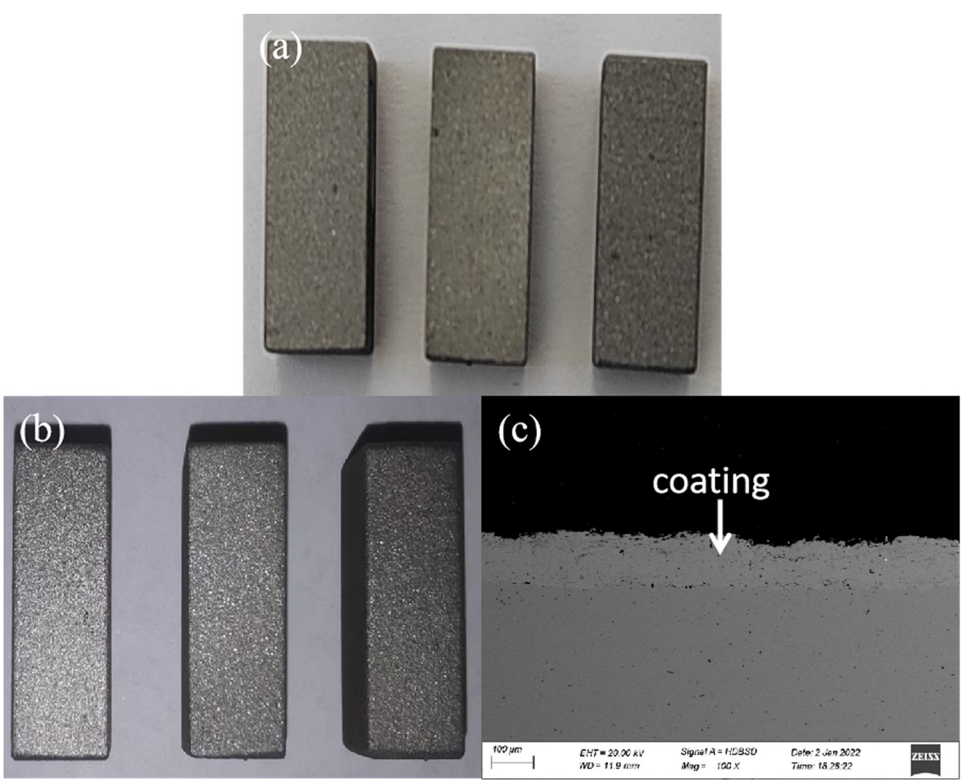

Figure 2: (a) The macro-morphology of base material. (b) Photos and (c) cross-section scanning electron microscope images of 316L wire coatings by explosive spraying.

Table 4: Bonding strength of the coatings

Coating	Bonding strength (MPa)
Sample 1	45.532
Sample 2	38.496
Sample 3	59.986
Average	48.005

Figure 3: Macro-morphology of the coatings in bonding strength test.

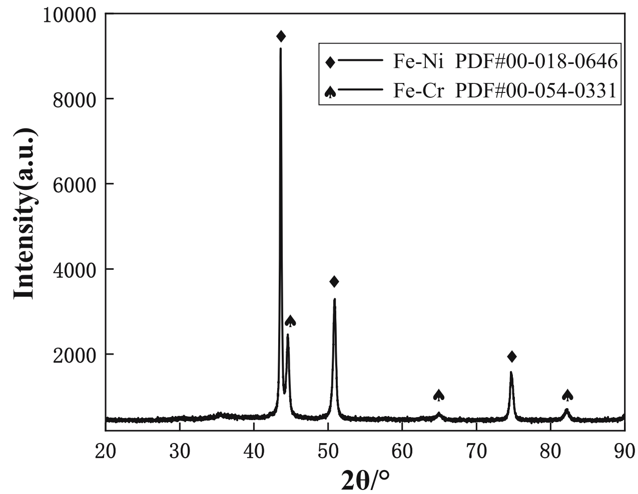

Figure 5: XRD spectrum of the coating surface.

for 30 min and then cooling to room temperature with water) 50 times without detachment or peeling. Table 4 shows the results of bonding strength between the coatings and the substrates. All samples in the experiment show detachment between the coatings and the substrates, and no detachment is found within the coatings (Figure 3). The average value of the measured bonding strength is about 48 MPa, as is shown in Table 4. The microhardness of the coating is 246HV.

Figure 4 shows the structures of 316L metal wire before and after electrical explosion spraying. It can be seen that the grain size of the 316L metal wire is 15–20 μm. After electrical explosion spraying, the grain size of the coating is 150–300 nm. White granular substances with inhomogeneous size of 30–50 nm can be observed in the coatings, most of them near the grain boundaries.

Figure 5 shows the XRD spectrum of the coating surface. As can be seen from the XRD results, the phase composition of the coatings is Fe–Cr and Fe–Ni. Figure 6 shows the SEM images and EDS spectrum of the surfaces of the coatings. The EDS result shows that oxygen element that is not from the original metal appears in the coatings. Analysis of point energy spectrum reveals that the white granular materials are oxides mostly distributed in the grain boundaries, which cannot be detected by XRD due to its low content and small grain size. The formation of oxides can be attributed to the reaction of the high-temperature metal droplets generated during electrical explosion spraying process and oxygen in the air. The molten

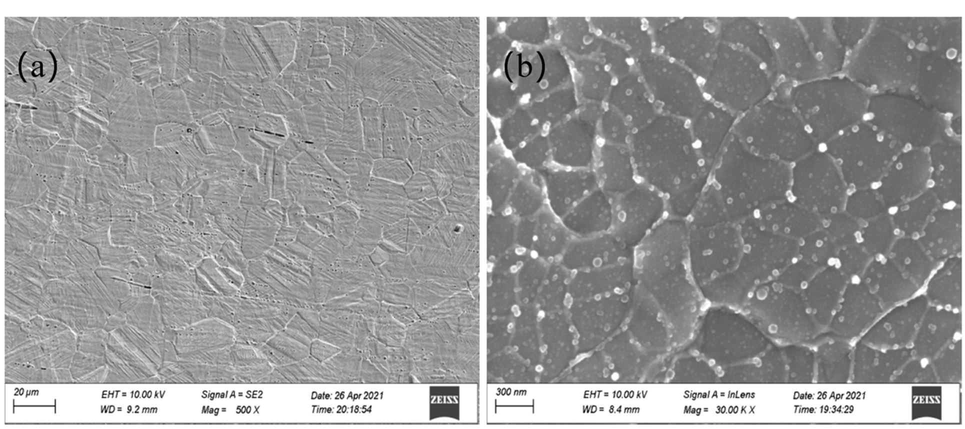

Figure 4: The structures of 316L metal wire (a) before and (b) after electrical explosion spraying.

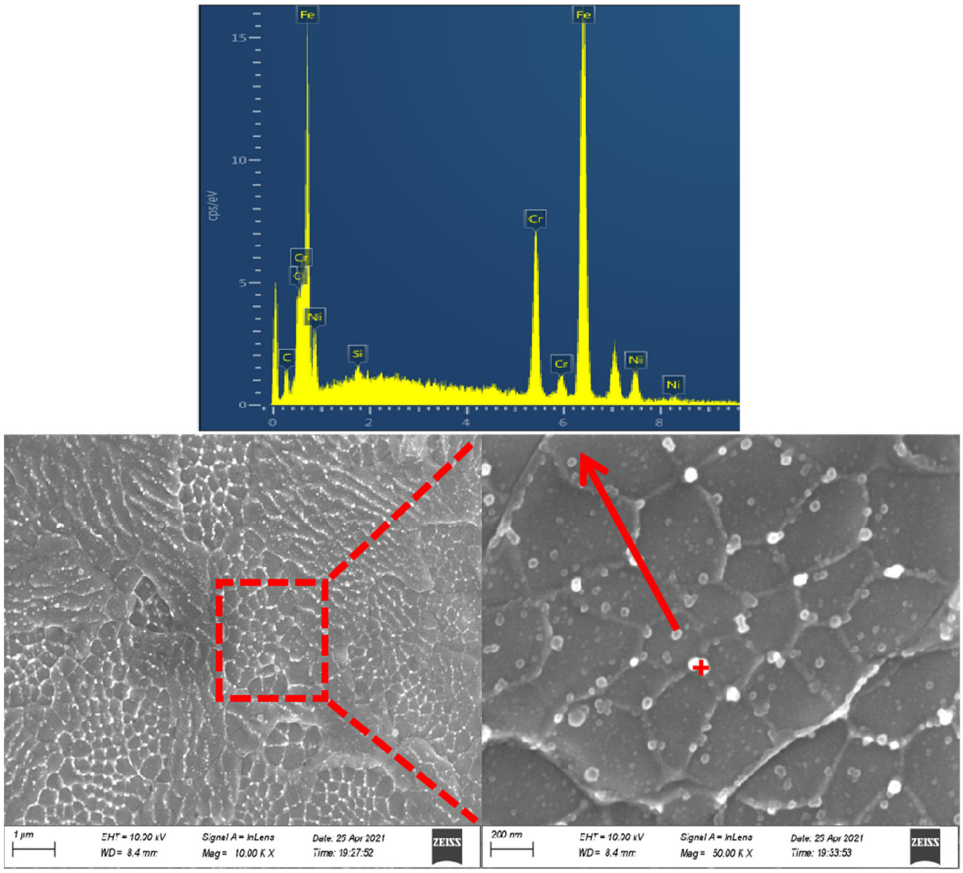

Figure 6: SEM images and EDS spectrum of the coating prepared by electrical explosion spraying.

metal droplets form some oxides, which hit the substrate at high speed and spread out on the substrate. Most of the oxides are distributed near the grain boundaries because of higher solubility of oxides at the grain boundaries than in the grains [24–27].

3.2 Corrosion performance of the coatings prepared by electrical explosion spraying

Figure 7 shows the kinetic curve of the coatings prepared by electrical explosion spraying with the treatment of corrosion solution at a constant temperature of 60°C in the medium shown in Table 3 for 168 h. It can be seen that the overall curve is in parabolic shape, and the corrosion rate increases linearly before 120 h and gradually tends to be stable after 120 h, indicating a decrease of corrosion rate after 120 h. Based on formula (1), the average corrosion rate within 7 days is calculated to be 0.079 mm/annum.

$$R = \frac{0.76 \times 10^7 \times (m_0 - m_1)}{STD}, \tag{1}$$

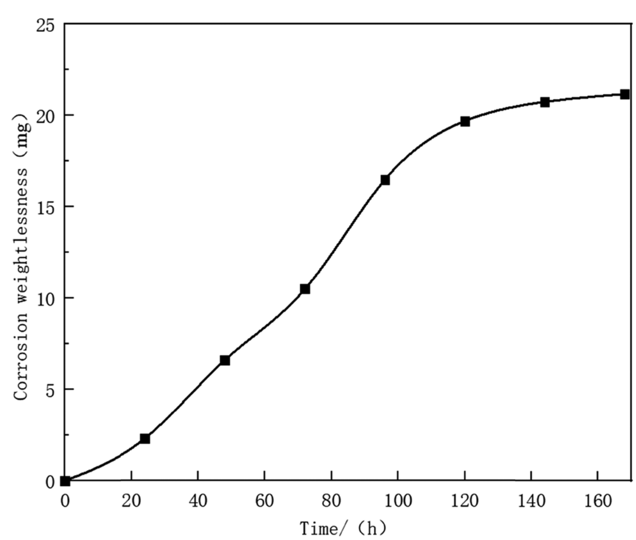

Figure 7: Kinetic curve of the coatings in corrosion test.

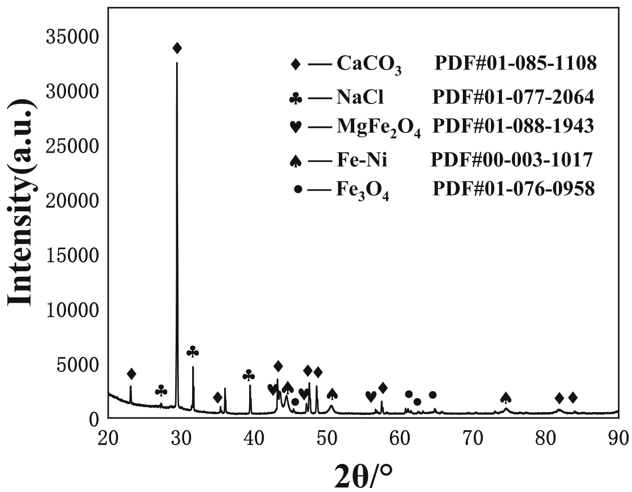

Figure 8: XRD spectrum of the coatings prepared by electrical explosion spraying after corrosion test at 60°C for 168 h in oil–water system.

where R is the corrosion rate (mm/annum), m_0 is the mass before the test (g), m_1 is the mass after the test (g), S is the total area of the sample (cm^2), T is the test time (h), and D is the density of the material (kg/m^3).

Figure 8 shows XRD spectrum of the coatings prepared by electrical explosion spraying after the corrosion test at 60°C for 168 h in oil–water system. It can be seen that the corrosion of the coatings produces $CaCO_3$, Fe_3O_4 and $MgFe_2O_4$. The Fe–Ni phase in the map is derived from the coatings, and NaCl is the residue of the solute in the corrosion solution.

Figure 9 shows the SEM image and EDS spectra of the cross-section of the coatings prepared by electrical explosion spraying after corrosion test at 60°C for 168 h in oil–water system. The results show the presence of oxygen and chloride elements on the surfaces of the coatings after corrosion, and the formation of oxides between the coatings and the substrates. The presence of oxygen and chlorine elements on the surfaces is consistent with the results in the XRD spectrum in Figure 8, which indicates possible micro-crevices in the coatings, allowing the corrosion elements like chloride in the solution to penetrate between the coatings and the substrates and forming a "corrosion zone" between the coatings and the substrates.

4 Discussion

The corrosion of coatings prepared by electrical explosion spraying is mainly attributed to the presence of oxides near the grain boundaries. These oxides can form protrusions, depressions, or holes on the surfaces and inside of

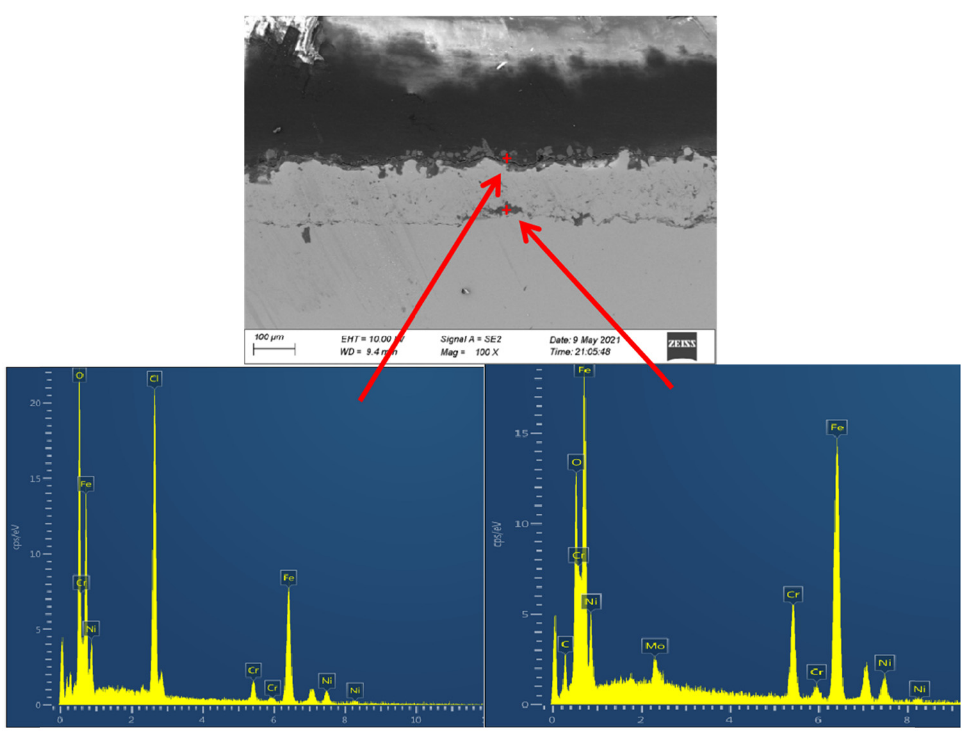

Figure 9: SEM image and EDS spectra of the cross-section of the coatings prepared by electrical explosion spraying after corrosion test at 60°C for 168 h in oil–water system.

the coating and react with solutes in the corrosion solution to cause detachment and dissolution, resulting in the formation of tiny pores, and further the formation of microgalvanic corrosion inside and outside the pores of the coatings and near the oxides. The following reactions then occur [28].

Fe element in the pores works as the anode:

$$Fe \rightarrow Fe^{2+} + 2e^{-} \tag{2}$$

Dissolved oxygen and water molecules in the pores work as the cathode:

$$O_2 + 2H_2O + 4e^{-} \rightarrow 4OH^{-} \tag{3}$$

OH^- diffuses as the reaction progresses, and pH value at the opening of the holes gradually increases:

$$Fe^{2+} + 2OH^{-} \rightarrow Fe(OH)_2\downarrow \tag{4}$$

The unstable $Fe(OH)_2$ will have a further reaction:

$$4Fe(OH)_2 + 2H_2O + O_2 \rightarrow 4Fe(OH)_3\downarrow \tag{5}$$

$Fe(OH)_3$ forms iron oxides in the presence of oxygen, which adhere to the surfaces of the coatings [29,30].

Meanwhile, with the increase in pH, the HCO_3^- ions in the corrosive medium gradually turn into CO_3^{2-}, which react with Ca^{2+} to form insoluble $CaCO_3$ attaching to the surfaces of the coatings.

$$CO_3^{2-} + Ca^{2+} \rightarrow CaCO_3\downarrow \tag{6}$$

At the same time, Mg^{2+} in the solution reacts with oxygen to form MgO, which then combines with Fe_2O_3 to form $MgFe_2O_4$ with a spinel structure.

$$MgO + Fe_2O_3 \rightarrow MgFe_2O_4 \tag{7}$$

The back-and-forth cycles of the above reactions form an autocatalytic process of the occluded corrosion cell, and the progress of such corrosion is the result of a combination of chemical and electrochemical interactions.

$MgO + Fe_2O_3 \rightarrow MgFe_2O_4$. Therefore, the gradually formed oxides and corrosion product $MgFe_2O_4$ in the spinel structure adhere to the surfaces of the coatings to form relatively dense protective films, which prevent the inward diffusion of oxygen and reduce the corrosion rate after 120 h. In addition, Cl^- in the solution can easily penetrate the tiny gaps between the oxide films and establish channels between the substrates and the corrosive environments, enabling ion exchange with the substrate materials [31,32]. Fe atoms are oxidized to form Fe^{2+} and gradually turn into iron oxides in the presence of oxygen. Once damages of these oxide films occur at certain points, the metal substrates under the damaged areas and the oxides in the undamaged areas form activation–passivation corrosion cells. The passivation surfaces act as the cathodes, which are much larger than the activation areas, leading to deep development of the corrosion and the formation of a "corrosion zone" between the coatings and the substrates [33–35], which has been confirmed by the XRD spectrum in Figure 8 and the SEM image and EDS spectra in Figure 9. The results show that the oxides generated in the process of melting and solidification of the metal wires during electrical explosion spraying have a significant impact on corrosion performance of the coatings.

5 Conclusion

In this work, we have studied the corrosion properties of coatings prepared by electrical explosion spraying of 316L metal wires in oil–water system and obtained the following results:

1) Electrical explosion spraying method can refine the grains in the obtained coatings. The grain size of the coating structure is 150–300 nm after electrical explosion spraying, while the grain size of the original structure is 15–20 µm.
2) The bonding force between the coatings and the substrates can reach 48 MPa by electrical explosion spraying of 316L metal wire.
3) $CaCO_3$, Fe_3O_4, and $MgFe_2O_4$ are generated in an oil–water corrosion test of coatings prepared by electrical explosion spraying of 316L metal wire at a temperature of 60°C for 168 h, with the corrosion rate being about 0.079 mm/annum.
4) The preparation of coatings by electrical explosion spraying can generate oxides, which are mostly distributed near the grain boundaries. The presence of these oxides is the main factor accounting for the corrosion of coatings.

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