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Geometric Influence of Hard Phase on Corrosion Performance between WC-Reinforced Coatings Prepared by High-Velocity Oxygen-Fuel Spray and Electric Contact Strengthening

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Copyright: © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). **Abstract:** Several kinds of WC-reinforced coatings were prepared by high-velocity oxygen-fuel spray (HVOF) and electric contact strengthening (ECS), respectively, and their corrosion behaviors in 3.5% NaCl solution were investigated. The microstructure, element distribution, phase and corrosion resistance of these coatings were compared. The results showed that, compared with HVOF-sprayed coatings, the ECS-prepared coatings were denser and with lower porosity. Simultaneously, the ECS coatings that used raw powder commercial WC-12Co retained the original spherical geometry of the hard phase. In open-circuit potential measurements, these ECS coatings gave higher stable potentials (E_{ocp}). In potentiodynamic polarization tests, although the corrosion currents (i_{corr}) of the homogeneous coatings were approximately the same, these ECS coatings still exhibited higher corrosion potentials (E_{corr}). The spherical geometric distribution of the hard phase led to a bounded diffusion model caused by the diffusion change of corrosion products.

Keywords: WC-reinforced coating; high-velocity oxygen-fuel spray; electric contact strengthening; heating-pressing action; corrosion resistance

1. Introduction

WC-reinforced coatings are widely used in mining, oil drilling and other industries due to their excellent performance [1,2]. One of the most common WC-reinforced coatings is the WC-12Co coating. At present, commercial WC-12Co powder is widely applied by synchronous powder feeding to deposit this coating. This commercial powder was formed by the agglomeration and sintering of WC and Co. It can be observed that fine-grained WC particles are uniformly combined with Co at high magnification. Thus, this powder can achieve enough kinetic energy in jet flight, and coatings can be obtained with sufficiently fine particle size in the hard phase.

Although the mechanical properties of cobalt binders are relatively better than those of nickel binders, nickel binders can provide higher corrosion resistance. In complex working environments, many salt-containing solutions (such as petrochemical products, mine water, lubricants, coolants, cutting fluid, and chemically aggressive water) are highly corrosive, which makes WC-Co materials more vulnerable to corrosion [3,4]. Therefore, many studies have attempted to add bonding phase Ni to the WC-Co coating system. This can effectively improve the density, wear resistance, oxidation resistance and corrosion resistance, with only a slight decrease in hardness [5–7].

The corrosion behavior of coatings is a very complex process, and is dependent on corrosion cells induced by various factors, such as surface and internal microstructure changes, pore distribution, impurity inclusion and electrolyte permeability [8–10]. To date,

the effects of different preparation parameters on the corrosion resistance of WC-based coatings have been studied in many articles. Picas et al. [11] found that the corrosion resistance of WC-Co-Cr coating prepared by gas-fueled HVOF is better than that by liquid-fueled HVOF in acid medium. This was due to the gas-fueled HVOF-sprayed coating having a higher porosity and promoting electrolyte penetration, which contributed to microcrack corrosion along the deposition boundary. Wang et al. [12] reported that WC-10Co-4Cr coating sprayed with pure oxygen contained more decarburized phase W₂C than that sprayed atmospherically due to its high deposition temperature and oxygen-enriched environment. Therefore, the corrosion resistance of former was weaker in 3.5% NaCl solution. Hong et al. [13] found that spraying distance was a more significant factor with respect to coating porosity than either oxygen flow or kerosene flow. Therefore, WC-10Co-4Cr coating deposited at a shorter spraying distance exhibited better corrosion resistance in 3.5% NaCl solution.

The corrosion resistance can even be influenced by artificially changing the surface morphology. Pishva et al. [14] reported the porosity and microcracks of HVOF-sprayed WC-10Co-4Cr coating were increased by mechanical grinding. This made it possible for corrosive solution to penetrate into the coating, leading to a decrease in the corrosion resistance of the coating. Wei et al. [15] reported the effect of surface roughness on the corrosion resistance of (FeCoCrNi)/WC alloy in 3.5 wt.% NaCl solution. It was found that a double-layer passive film was formed on the surface, and its stability and compactness increased with decreasing roughness. The corrosion mechanism changed from uniform corrosion to pitting corrosion.

Furthermore, other studies have reported that different preparation methods can also lead to differences in the coating microstructure, leading to homogeneous coatings with different corrosion resistances. Zhang et al. [16] reported that the porosity of HVOF-sprayed WC-24Cr₃C₂-6Ni coating was reduced and the interface was densified following laser treatment. Compared with the homogeneous coating without laser treatment, the local corrosion of the treated one was reduced. Laser heat treatment improved the pitting corrosion resistance, and the densified structure prevented the electrolyte from reaching the substrate. Gao et al. [17] compared the corrosion resistance of Al₂O₃ coatings prepared by laser cladding and plasma spraying. It was found that the laser-cladded coating had more stable α -Al₂O₃ phase and lower porosity, such that it was not easy for the electrolyte to penetrate into the deep part. As a result, the corrosion potential of the polarization curve of the laser-cladded Al₂O₃ coating was higher than that of the plasma-sprayed coating, and the corrosion current density was lower. Rolink et al. [18] reported that the corrosion resistance was significantly influenced by the surface morphology and the electrolyte penetration rate. In their study, the corrosion current density (icorr) of laser-cladded AlSi₂₀ coating was close to that of laser-remelted plasma-sprayed coating, while the corrosion potential (E_{corr}) of the former coating was significantly higher than the others.

In summary, different methods and forming processes can deeply affect the corrosion performance of coatings. Simultaneously, some articles have reported that the hard phase can show different morphologies and distributions even when the same commercial powder is used [19–22]. Consequently, different states of the hard phase can significantly influence the corrosion performance. Therefore, it is worth comparing the characteristics achieved when using different methods and investigating the corrosion performance resulting from different hard phase morphologies.

In this study, a novel method was used to prepare WC-reinforced coating. This method is called electric contact strengthening (ECS). The corrosion behavior and corrosion resistance of the WC-reinforced coatings prepared by ECS were investigated and compared with those of HVOF-sprayed coatings in 3.5% NaCl solution. In addition, the geometric influence of hard phase (WC) on corrosion behavior was evaluated. The same raw powders, including commercial WC-12Co powder and simple substance powder, were used to prepare the coatings in both of the methods. The surface morphology, cross-sectional microstructure and element distribution before and after corrosion were observed using field

emission scanning electron microscopy (FESEM) with an energy dispersive spectrometer (EDS). The phase composition was detected using X-ray diffraction (XRD). The corrosion resistance of these WC-reinforced coatings was tested using an electrochemical workstation. Based on the test for corrosion, the distinctions of corrosion behavior between these coatings were analyzed.

2. Materials and Methods

2.1. Sample Preparations

Commercial WC-12Co powder (5–35 µm, Sinopharm Chemical Reagent Co. Ltd., Shanghai, China) and simple substance WC, Co and Ni powders (300 mesh, Sinopharm Chemical Reagent Co. Ltd., Shanghai, China) were used to prepare different WC-reinforced coatings. These coatings were prepared using the ECS and HVOF methods with certain proportions; detailed information on the proportions and their labeled names is presented in Table 1. The substrate was made of medium carbon steel C45E4. The hard phase of the H1, H2, E1, E2 coatings was commercial WC-12Co powder, while the E3 coating was the simple substance WC powder. Commercial WC-12Co powder is a common powder with a porous and spherical geometry, formed by the agglomeration and sintering of Co and WC with small particle size, as shown in Figure 1. The commercial WC-12Co powder was used directly to prepare coatings H1 and E1. For coatings H2 and E2, commercial WC-12Co powder and Ni powder were ball milled together in alcohol for 1 h to obtain a mixed raw powder. The simple substance WC, Co and Ni powders were dry milled for 1 h as well.

Table 1. Preparation method and raw composition.

Labeled	Preparation Method Coating Composition (wt.%)			
H1	HVOF	100% (WC-12Co) (commercial powder)		
H2	HVOF	75% (WC-12Co) (commercial powder) + 25% Ni (milled powder)		
E1	ECS	100% (WC-12Co) (commercial powder)		
E2	ECS	75%(WC-12Co) (commercial powder) + 25% Ni (milled powder)		
E3	ECS	66% WC (milled powder) + 9% Co (milled powder) + 25% Ni (milled powder)		



Figure 1. Microstructure of commercial WC-12Co powder.

The H1 and H2 coatings were prepared using the HVOF method with the spray equipment (JP-5000, Praxair Tafa, Concord, NC, USA). The substrate was C45E4. The spraying parameters are shown in Table 2.

Table 2. Spraying parameters of HVOF.

Spraying Parameters	Value	Unit
O ₂ flow rate	53	m ³ /h
Kerosene flow rate	23	L/h
Powder feed rate	75	g/min
Carrier gas flow rate (N_2)	0.6	m ³ /h
Spraying distance	350	mm

The E1, E2 and E3 coatings were prepared using the ECS method with self-developed ECS equipment. The procedure for coating preparation is illustrated in Figure 2, and a schematic diagram of the equipment is shown in Figure 3. In this method, the raw powders were first evenly mixed with the polymer binder (mainly polyvinyl acetate) to obtain raw slurry. Then, the slurry was brushed onto the surface of the substrate (Figure 4a) and self-cured to form the pre-coating (Figure 4b). The thickness of the pre-coating was about $500-550 \mu m$. Then, during the ECS process, the heating–pressing action was carried out to form the WC-reinforced coating from the pre-coating. The outer rim of the electrode wheel was brought into contact with the pre-coating and rotated at matching linear speed. Simultaneously, the translation of the driven electrode wheel with external stress enabled the entire area of the pre-coating to be processed. The electrode wheel travelled translationally from one side to the other and then returned. This reciprocal travel was treated as one process period. Each pre-coating underwent one period (about 30 min) in order to form the WC-reinforced coating. The E1, E2 and E3 coatings were prepared using the same parameters. The parameters of the ECS process are shown in Table 3, and they are indicated near their corresponding components in Figure 3, as well.



Figure 2. Coating preparation procedure.



Figure 3. Schematic diagram of the ECS equipment.



Figure 4. Macroscopic appearance in the ECS process: (a) substrate; (b) pre-coating; (c) formed coating.

Table 3. Preparation parameters of ECS.

Preparation Parameters	Value	Unit
Voltage (U)	8	V
Current (I)	35	kA
Contact pressure (P_c)	0.05	MPa
Rotational speed (V_r)	0.5	rpm
Translational speed (V _t)	1	mm/min

2.2. Characterization Methods

The surface morphologies, cross-sectional microstructure and element distribution of the coatings were observed using field emission scanning electron microscopy (FESEM) (Quanta 250 FEG; FEI Ltd., Brno, Czech Republic) with an energy dispersive spectrometer (EDS) (AZtec X-Max 20, Oxford Instruments, Abingdon, UK). The testing voltage was 15 kV, and the tests were carried out under high vacuum. The porosity was calculated by the software ImagePro Plus based on the area proportion of pores within the whole coating cross-section. The surface roughness (Ra) of each coating was measured using a stylus profilometer (JB-4C, Shanghai Optical Instrument factory No.1, Shanghai, China). The phase compositions of the coatings were detected using X-ray diffraction (XRD) (D/Max-2550PC, Rigaku Co.Ltd., Akishima, Japan). The power of the XRD machine was 18 kW, the angle range was 25° – $90^{\circ}/2\theta$, and the angular step was $0.02^{\circ}/2\theta$, with 2 s per step. The compositions and chemical states of the corroded specimens were detected using XPS (Escalab 250Xi, Waltham, MA, USA) with the Al Ka standard as the exciting source and XRD (D/max-2550PC, Rigaku Co.Ltd., Akishima, Japan) with Cu K-alpha radiation ($\lambda = 0.154056$ nm) operated at 40 kV and 200 mA, ranging from 30° to 80° with a step size of 0.026°. The binding energy of the XPS target elements was measured at a pass

energy of 23.5 eV with a resolution of 0.1 eV, using the binding energy of standard carbon (C1s: 284.8 eV) as the reference.

2.3. Electrochemical Measurements

Before the electrochemical test, the samples were cut into little pieces with a test square 1 cm². The test coatings were all used without polishing so that the exposed surfaces could retain their original morphology.

The electrochemical tests were carried out in 3.5 wt.% NaCl solution at room temperature. Open circuit potential (OCP), polarization curves and electrochemical impedance spectroscopy (EIS) were tested using the electrochemical workstation CS310H (Wuhan Corrtest Instruments Corp., Ltd., Wuhan, China). These tests were performed according to the ASTM standard F2129. The standard three-electrode system consisted of a working electrode (coating samples), a counter electrode (platinum sheet), and a reference electrode (saturated calomel electrode, SCE). Each test sample was immersed in the solution for 4 h to obtain steady OCP values. Then the EIS test was performed at steady OCP. The sinusoidal signal was 5 mV rms and the frequency range was from 100 kHz to 1 mHz, with 10 points per frequency decade. The Bode and Nyquist plots were fitted by the software ZSimpWin. The potentiodynamic polarization curves were scanned from -1000 mV to +1000 mV with a scanning rate of 2 mV/s. Then, the corrosion potential (E_{corr}) and corrosion current density (i_{corr}) were calculated using general electrochemistry software. All the tests were repeated three times.

3. Results and Discussion

3.1. Coating Microstructure

3.1.1. Surface Morphology

Figure 5a–e show the surface morphologies of the H1, E1, H2, E2 and E3 coatings. The order in which the subsequent morphology figures are displayed follows this sequence. All of the SEM images were taken in secondary electron imaging (SE) mode, as were the images in the following text. The Ra values for coating surface roughness are listed in Table 4. The surface roughness (Ra) values of the ECS-prepared coatings were generally lower than those of the HVOF-sprayed coatings. The H1 and H2 coatings exhibited the common morphology of the HVOF-sprayed coatings. Both of them exhibited high surface roughness due to the impact and uneven stacking of the raw powders in flight. Cracks could be observed in the rough surface of the H1 coating because of the lack of bonding phase. However, the E1 and E2 coatings exhibited a coexistence of sintered bulk and loose sintered structures. On the surface of these two coatings, the original spherical geometry of the commercial WC-12Co raw powder could be observed. In particular, the E2 coating showed more sintered bulk structure than that of the E1 coating, owing to the higher proportion of bonding phase, meaning that enough bonding phase had clung to and crept to cover the spherical WC-12Co particles, resulting in a smoother coating surface being obtained. The surface morphology of the E3 coating was similar to that of the HVOFsprayed coatings. Because the E3 coating was formed by entire milled powders, and the deposition mode was in situ forming rather than impact stacking, its surface presented the lowest roughness and the highest density.

Table 4. Coating properties.

Labeled	Thickness (µm)	Surface Roughness (Ra/µm)	Porosity (%)
H1	415 (±6.5)	6.21 (±0.32)	9.24 (±0.16)
H2	409 (±4.9)	5.63 (±0.18)	7.87 (±0.27)
E1	412 (±7.9)	4.28 (±0.26)	3.69 (±0.22)
E2	413 (±3.8)	3.55 (±0.20)	3.35 (±0.21)
E3	410 (±4.2)	3.48 (±0.22)	2.97 (±0.18)



Figure 5. Surface morphologies: (a) H1; (b) E1; (c) H2; (d) E2; (e) E3.

3.1.2. Cross-Sectional Microstructure

Figure 6a–e shows the cross-sectional microstructure of the coatings; the thickness and porosity of each coating is also listed in Table 4. All of the coating thicknesses were controlled to be between 410 and 420 μ m. The porosities of the HVOF coatings were much higher than those of the ECS coatings. On the basis of the interface morphology of the H1 and H2 coatings, it was found that not only did plastic deformation occur at the substrate surface, but some hard phase was also embedded due to the high-kinetic-energy impact of

raw powders. Simultaneously, there were many pores at the interfacial area, which was consistent with the interface characteristics of the HVOF-sprayed coatings. Meanwhile, no pores were found at the interface of the ECS-prepared coatings. The interfaces of the E1–E3 coatings were smooth and well bonded. At the coating area, the brightly colored part represents the hard phase, and the dark-colored part represents the bonding phase. Owing to the lower content of bonding phase, the H1 and E1 coatings exhibited a greater number of pores than the other coatings. Compared with the Ni-added coatings (H2, E2 and E3), although the porosity of the E1 coating was lower than that of the H1 coating, the porosity values of these two were still high. During the ECS process, the continuous heating–pressing action could have promoted the healing of pores. Owing to the different forming modes of the two methods, the pores of the EVS-prepared coatings were mainly distributed in the interior area, while the pores of the ECS-prepared coatings were mainly distributed at the coating skin.



Figure 6. Cross-sectional microstructure: (a) H1; (b) E1; (c) H2; (d) E2; (e) E3.

When the bonding phase proportion was high enough, the internal porosity of the coatings was greatly reduced. In addition, the internal structure of the E2 coating was denser than that of the H2 coating. Compared with the H1 coating, the uneven distribution of hard phase and bonding phase was obvious in the H2 coating. Most of the agglomerated WC-Co spherical particles were not able to retain their original spherical shape. These particles experienced significant fracture deformation during flight impact and finally exhibited an irregular shape [23]. The distribution of hard phase and bonding phase in the E2 coating was relatively uniform, and the porosity was lower. In particular, compared with E1 coating, it could be clearly seen that the spherical geometry of WC-12Co had been retained through the ECS process. Because the E3 coatings were formed using ball-milled powder, the microstructure of the E3 coating was the most compact. However, due to the agglomeration of hard phase, a large size of WC was produced through the forming process.

3.1.3. XRD Results

Figure 7 shows the XRD spectrum of the formed coatings before the corrosion test. The composition phases of the H1 and E1 coatings were WC and Co, while those of the H2, E2 and E3 coatings were WC, Co and Ni, due to the addition of bonding phase Ni. Because of the short thermal reaction of raw materials in the HVOF process, it is difficult to find the solid solution phase Co_3W_3C . However, when using the ECS method, the coatings underwent a longer heating–pressing action during the forming process. Therefore, it was possible to observe Co_3W_3C in the E1, E2 and E3 coatings, corresponding to peaks at about 40° and 42.5° , respectively. The reaction of WC dissolution in Co is represented by the following equation:

$$WC \diamond Co \bullet Ni \lor Co_3W_3C$$

$$-E3$$

$$-E2$$

$$-H2$$

$$-H2$$

$$-H1$$

$$-H1$$

$$-H1$$

$$-H1$$

 $2\theta/^{\circ}$

$$3Co + 3WC + O_2 \rightarrow Co_3W_3C + 2CO \tag{1}$$

Figure 7. XRD before corrosion.

3.2. Corrosion Performance

3.2.1. Open Circuit Potential Test

Figure 8 shows the open circuit potential (OCP) curve of each coating in 3.5% NaCl solution for 4 h. The stable E_{ocp} values are listed in Table 5. The OCP curves of all coatings reached a stable state at about 4000 s, meaning that the corrosion state on these sample surfaces gradually stabilized. The steady state value was recorded as the OCP value (E_{ocp}) of the coating. It can be seen that the E_{ocp} values of the WC-reinforced coatings (H2, E2 and E3) with Ni addition were generally higher than those for coatings without Ni addition. Additionally, the results indicate that the E_{ocp} values of the ECS-prepared coatings were all higher than those of the HVOF-sprayed coatings. Zhang et al. [24] proved

that a higher E_{ocp} was negatively correlated with corrosion tendency. Therefore, it is evident that the corrosion resistance of the ECS-prepared coatings was higher than that of HVOF-sprayed coatings.



Figure 8. OCP curves.

Table 5. Electrochemical results of OCP and potentiodynamic polarization.

	Eocp	Forwa	ard Scan	Reverse Scan		
Sample	(V_{SCE}, V)	E _{corr} (V _{SCE} , V)	i _{corr} (μA·cm ^{−2})	E _{corr} (V _{SCE} , V)	i _{corr} (µA·cm ^{−2})	
H1	-0.639	-0.805	$3.145 imes 10^{-5}$	-0.534	$3.25 imes 10^{-4}$	
E1	-0.561	-0.734	$2.836 imes10^{-5}$	-0.466	$1.01 imes 10^{-4}$	
H2	-0.458	-0.591	$1.731 imes 10^{-6}$	-0.358	$6.28 imes10^{-5}$	
E2	-0.321	-0.485	$1.804 imes 10^{-6}$	-0.292	$8.26 imes 10^{-5}$	
E3	-0.406	-0.560	$4.523 imes 10^{-6}$	-0.387	$2.39 imes 10^{-4}$	

3.2.2. Potentiodynamic Polarization Test

Figure 9 shows the potentiodynamic polarization measurements of each coating. The polarization curves of all coatings show typical anodic behavior, meaning that the corrosion current density (i_{corr}) increased exponentially with the corrosion potential (E_{corr}), before finally tending towards being stable. The stable values are also listed in Table 5. Compared with the H1 coating, the passivation behavior of the E1 coating was obviously stronger. Although the distinctions of the anodic passivation of Ni-added coatings (H2, E2 and E3) were not so remarkable, it can still be observed that the E2 coating expressed the strongest passivation tendency. With respect to the two homogeneous couples (H1 and E1, H2 and E2), the i_{corr} values were too close to be distinguished. In general, higher E_{ocp} and E_{corr} indicate better chemical stability and lower corrosion tendency. Meanwhile, a lower i_{corr} indicates a lower corrosion rate. Taking into account experimental error, it can be concluded that the corrosion rates of each couple were almost the same. Therefore, compared with the HVOF-sprayed coatings, the ECS-prepared homogeneous coatings expressed higher E_{ocp} and E_{corr} values, meaning that the ECS-prepared coatings had a lower corrosion tendency. In the reverse scan curve, all of the coatings showed a negative

hysteresis loop at first, followed by positive hysteresis. The areas of the negative hysteresis loops belonging to the E1 and E2 coatings were larger than those of the H1 and H2 coatings, indicating more serious pitting corrosion [25,26]. This is associated with the spherical geometry of WC and corresponds to the coating surface morphology after corrosion, as shown in Figure 10. In addition, the E_{corr} values of the E1 and E2 coatings were generally higher than those of the H1 and H2 coatings. Meanwhile, the i_{corr} values of H1 and H2 coatings were higher than (or close to) those of the E1 and E2 coatings. Therefore, it can be concluded that the degree of pitting corrosion of E1 and E2 was higher than that of H1 and H2, but the uniform corrosion degree was still lower.



Figure 9. Potentiodynamic polarization curves.

3.2.3. Morphology after Corrosion

Figure 10 shows the surface morphology and element distribution of each coating after the potentiodynamic polarization test. The mass fraction and atomic fraction of the elements are shown in Table 6. All of the coatings exhibited loose and porous microstructures with lots of corrosion pits. Compared with the HVOF-sprayed coatings (H1 and H2), the ECS-prepared coatings (E1 and E2) retained the original spherical geometry of their hard phase. Due to the addition of Ni, the E2 coating exhibited a less hollow spherical structure than the E1 coating. This phenomenon was the same for the H2 coating vs. H1. The surface morphology of the E3 coating was similar to that of the H1 and H2 coatings, but it attained more of a block structure, and the WC skeleton was less exposed.

It has been reported that in 3.5% NaCl solution, the E_{corr} of pure WC is about +0.20 V, that of pure Co is about -0.31 V [27], and that of pure Ni is about -0.20 V [28]. Therefore, in this study, the noble level was WC > Ni > Co. It was also for this reason that Ni addition was able to enhance corrosion resistance. It has been confirmed that the dominant corrosion mechanism of the WC-reinforced coating was micro-galvanic corrosion in 3.5% NaCl solution, where WC acts as the cathode and the adhesive phase acts as the anode dissolution [29]. In the H1 and E1 coatings, corrosion preferentially took place via Co oxidation, leaving the skeleton of WC particles [30]. In the other coatings in this study, Ni also participated in the anodic reaction, with a lower dissolution priority than Co. In the micro-galvanic corrosion of the WC-reinforced coating, the main reactions of bonding phase at anode were as follows:

$$\mathrm{Co} \to \mathrm{Co}^{2+} + 2e^{-} \tag{2}$$

$$\mathrm{Co}^{2+} + 2\mathrm{H}_2\mathrm{O} \to \mathrm{Co}(\mathrm{OH})_2 + 2\mathrm{H}^+ \tag{3}$$

and

$$\mathrm{Ni} \to \mathrm{Ni}^{2+} + 2e^{-} \tag{4}$$

$$Ni^{2+} + 2H_2O \rightarrow Ni(OH)_2 + 2H^+$$
 (5)

where the hydroxide (compound form $M_x(OH)_y$) was the corrosion product, containing either or both $Co(OH)_2$ and/or $Ni(OH)_2$. Souza et al. [31] reported that WC also becomes the corrosion anode when the bonding phase is insufficient, consequently producing WO_3 as the corrosion product. The reaction was as follows:

$$WC + 5H_2O \rightarrow WO_3 + CO_2 + 10H^+ + 10e^-$$
 (6)



Figure 10. Cont.



Figure 10. Morphologies after corrosion and element distribution: (a) H1; (b) E1; (c) H2; (d) E2; (e) E3.

Sample	(Atom%/Mass%)	Detected Elements						
		W	С	0	Со	Ni	Fe	Cr
111	at.%	18.72	45.61	23.63	6.69	-	2.76	2.59
HI	wt.%	68.14	10.85	7.49	7.81	-	3.05	2.67
T 1	at.%	16.33	36.91	34.55	7.7	-	2.36	2.15
EI	wt.%	63.93	9.44	11.77	9.66	-	2.81	2.38
110	at.%	11.24	38.22	33.93	3.02	6.07	3.82	3.7
H2	wt.%	51.55	11.45	13.55	4.44	8.89	5.32	4.80
EO	at.%	10.88	33.02	40.82	2.84	6.2	3.19	3.05
EZ	wt.%	53.65	9.38	15.93	4.08	8.75	4.34	3.87
E3	at.%	11.5	36.07	37.21	2.81	6.05	3.25	3.11
	wt.%	52.76	10.81	14.86	4.13	8.86	4.53	4.04

Table 6. Element contents from EDS detection.

In other words, the corrosion loss of the supporting bonding metal promotes the oxidation and removal of WC phase. Therefore, it can be inferred that E1 and E2 coatings, with more WC retention, should lead to increased WO₃. According to Table 6, the O contents at the E2 and E3 coating skin were significantly higher than those of the H1 and H2 coatings. Because the enrichment area of the O element coincides with that of the W element, it can be concluded that the presence of the O element partially reflects the amount of WO₃ present. Therefore, the more the WC skeleton (spherical geometry) is exposed, the greater the amount of WO₃ that should be produced.

3.2.4. XRD and XPS Results after Corrosion

The phase composition after the potentiodynamic polarization test is shown in Figure 11. In addition to the original phase of the coatings, two remaining corrosion products were detected, which were the solid solution phase oxides $CoWO_4$ (or $NiWO_4$) and hydrated tungsten oxide $WO_3 \cdot H_2O$. These solid solution phase oxides were more obvious in the ECS coatings (E1, E2 and E3) than in the HVOF coatings. Furthermore, this phase, in the E1 and E2 coatings, showed exclusive peaks at about 30.7° . These corresponded to the significant solid solution phase in ECS coatings before the potentiodynamic polarization test. The $WO_3 \cdot H_2O$ peaks of E1 and E2 coatings were more remarkable than those of the other coatings at about 23.8° and 54.2° . According to the corroded morphology in Figure 10b,d, this could prove that the remaining porous spherical WC skeleton resulting in the generation of more WO_3 .



Figure 11. XRD results after corrosion.

The above inference that the spherical WC skeletons were heavily oxidized is also supported by the XPS results of the corroded coating surface, as shown in Figure 12. In the W 4f spectra, the peaks at 35.2 eV and 37.4 eV indicate that the presence of the oxide film WO₃ or WO₃·xH₂O. In the corrosion products of the H1, H2 and E3 coatings, WC was also detected at 34.1 eV and 31.5 eV [32]. Meanwhile, the WO₃ contents of E1 and E2 coatings were much higher than those of other coatings. In the Ni 2p and co 2p spectrums, the detection results corresponded to single or double binder phase corrosion products Ni(OH)₂ and Co(OH)₂.

3.2.5. EIS Results

The EIS curves in 3.5% NaCl solution were measured after all coatings had been stabilized in the OCP test for 4 h. The measured Nyquist plot and the corresponding Bode plot are shown in Figure 13a–d. The dots represent the actual data and the line represent fitting data, as in the following figures. In the Nyquist plots, the curve radii of the ECS-prepared coatings were all larger than those of HVOF-sprayed coatings, as shown in Figure 13a,c.

During the corrosion process of the WC-reinforced coating in 3.5% NaCl solution, the hard phase acted as the cathode, and the bonding phase acted as the anode in galvanic corrosion. The chloride ions preferentially corrode the bonding phase. Therefore, the hard phase remained in the form of a WC skeleton, before gradually peeling off from the coating surface [33]. Consequently, the corrosion products freely diffused, and the newly exposed bonding phase corroded continuously. Many works have reported that HVOF-sprayed coating exhibits linear diffusion dominated by a semi-infinite plane in the neutral medium.

Therefore, the equivalent circuit of WC-reinforced coating prepared by HVOF usually exhibits a semi-infinite diffusion plane model (R(Q(R(QR)))). In this study, for the coatings that retained the original spherical geometry of the hard phase, a more suitable fitting equivalent circuit was achieved by adding Warburg impedance. Warburg impedance was used to represent the diffusion control mechanism in the system, so the equivalent circuit model (R(Q(R(Q(RW)))) was applied for E1 and E2 coatings, as shown in Figure 13e. Table 7 shows the detailed EIS fitting parameters. In the circuit, R_s represents the resistance of the corrosion solution; R_{ct} and R_f represent the charge transfer resistance and passive film resistance, respectively; Q_{dl} and Q_f (CPE) represent the double-layer capacitor and passive film capacitor, respectively.

Song et al. [34] analyzed the influence of microstructure differences on the corrosion behavior of homogeneous coatings with respect to the particle shape and size distribution of the coating body. Compared with the typical semi-infinite plane diffusion, the corroding plane consisted of intercalated spherical particles possessing a different local interface impedance. These two planes corresponded to bounded diffusion. In addition, their theoretical calculations indicated that the impedance modulus was higher at low frequencies, and the phase angle was higher at intermediate frequencies. This was verified in the test results for H1 and E1 coatings, as shown in Figure 13a,b. The impedance modulus at low frequency and the phase angle at intermediate frequency for the H1 coating were lower than those for the E1 coating. Meanwhile, in Figure 13c,d, although the E2 coating showed the highest modulus at low frequency, the distinctions were not so obvious in this comparison group (H2, E2 and E3). In this group, the phase angles were obviously deviated to the low-frequency region, and the E2 coating had the largest deviation. This provides evidence that the E2 coating exhibited the best corrosion resistance performance in 3.5% NaCl solution.

According to the surface morphology after potentiodynamic polarization test, the ECSprepared WC-reinforced coatings retained their original geometry of WC-12Co spheres, leaving a large number of spherical WC-based skeletons. The equivalent circuit shows that the E1 and E2 coatings should exhibit bounded diffusion in the corrosion process. In the WC-reinforced coating system, the corrosion process preferentially started from the interface between the bonding phase and the hard phase. The corrosion dissolution of the bonding phase and the rapid diffusion of the corrosion products were the direct causes of the exposure of the WC skeleton and the subsequent WC detachment. It has been reported that the presence of adhesive and protective corrosion products can lead to lower corrosion rates, as well as differences in the measured pitting amount [35,36]. In the work of Hong et al. [37], the attachment of microorganisms affected the corrosion rate of the WC-10Co-4Cr coating. Due to the attachment of microorganisms, the dissolution of corrosion products and the rate of ion exchange in the solution were changed. Therefore, the (R(Q(R(Q(RW))))) model was applied in consideration of these factors, with Warburg impedance representing the change in diffusion control in the system.



Figure 12. XPS result after corrosion.



Figure 13. EIS results: (a) Nyquist plots of H1 and E1; (b) Bode plots of H1 and E1; (c) Nyquist plots of H2, E2 and E3; (d) Bode plots of H2, E2 and E3; (e) fitting models of equivalent circuit.

Table 7. Equivalent circuit parameter values.

		Q_{f}			Q	dl		
Sample	Rs (Ω cm ²)	Y ₀ (µF/cm ²)	п	$R_{\rm f} (\Omega \ {\rm cm}^2)$	Υ ₀ (μF/cm ²)	n	R_{ct} (Ω cm ²)	Z_w
H1	5.792	0.0009349	0.8692	4.585	0.001107	0.844	1082	
	(0.84%)	(1.82%)	(0.64%)	(4.95%)	(2.07%)	(8.03%)	(2.44%)	-
E1	5.757	0.0007054	0.8747	3.697	0.0009053	0.8484	1349	0.157
	(0.72%)	(1.06%)	(1.58%)	(8.81%)	(9.94%)	(9.91%)	(2.21%)	(7.59%)
H2	10.72	0.001678	0.657	32.53	0.001773	0.7426	2182	
	(0.90%)	(2.82%)	(5.46%)	(3.08%)	(2.75%)	(6.18%)	(5.34%)	-
E2	11.63	0.002229	0.7411	7.209	0.003117	0.7355	4169	3.079
	(0.49%)	(7.13%)	(1.17%)	(5.69%)	(5.17%)	(6.93%)	(7.93%)	(3.60%)
E3	11.19	0.001277	0.6736	10.24	0.00292	0.7168	3049	
	(0.97%)	(5.59%)	(9.58%)	(2.81%)	(2.49%)	(4.53%)	(6.51%)	-

The primary corrosion process for the WC coating is erosion of bonding phases, the formation of corrosion products, and the brittle detachment of hard phases [38]. It has been reported that the corrosion of thermal-sprayed WC-reinforced coatings corresponds to the semi-infinite diffusion model [39]. After the bonding phase around WC skeleton is completely corroded, these WC detach from the coating skin, as shown in Figure 14a. Meanwhile, in this study, the dissolution of the corrosion products and the ion concentration re-equilibrium were affected by the narrowing of the diffusion channels in the E1 and E2 coatings [37]. Du et al. [40] reported that when the hard phase maintains is multilayer structure and has difficulty detaching during the corrosion process, the concentration of the corrosion solution decreases gradually along the coating thickness direction. In addition, Warburg impedance was included in the equivalent circuit model to match this phenomenon. Additionally, it was demonstrated that the corrosion performance also depends on the distribution and diffusion of passive products [40,41]. The product diffusion rate is related to the geometric configuration of the residual WC skeletons, either the porous narrow channels inside the spherical WC skeleton itself, or those between multiple WC spheres. As shown in Figure 14b, the original spherical geometry of commercial WC-12Co powder was retained by the ECS process. During coating formation, once the bonding between the hard phases and the co-intercalation of spherical skeletons had taken place, the WC skeletons did not easily collapse and detach, even though the surrounding bonding phase was corroded and dissolved.



Figure 14. Diffusion model: (a) semi-infinite diffusion; (b) bounded diffusion.

4. Conclusions

Several kinds of WC-reinforced coatings were successfully prepared using the ECS method, and their corrosion resistance in 3.5% NaCl solution was compared with that of HVOF-sprayed homogeneous coatings. The following conclusions were obtained:

- (1) Coatings prepared using the ECS method were able to achieve lower porosity and higher density than the HVOF-sprayed homogeneous coatings. In addition, the pores of E1-E3 coatings were concentrated at the coating skin.
- (2) The ECS-prepared coatings using commercial WC-12Co powder was able to retain the original geometry of this spherical powder. WC and Co have better degree of solid solution, showing an obvious Co₃W₃C phase.
- (3) The ECS-prepared coatings had better corrosion resistance than the HVOF-sprayed homogeneous coatings in 3.5% NaCl solution.
- (4) The spherical WC-12Co powder in the ECS-prepared coatings retained its original geometry, which made it difficult for the dimensional WC skeleton to detach during the corrosion process. In addition, this led to a bounded diffusion model.

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