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Study on the influencing factors of interfacial chemical reactions and adhesion effects in different metal coating composition systems

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Abstract: This article studies the interfacial chemical reactions and adhesion properties of typical metal coating systems of zinc based, aluminum zinc based, and zinc aluminum magnesium during continuous hot plating and electroplating processes. Combining SEM, EDS, and XRD analyses, this study investigates the roles of aluminum, magnesium, and other elements in the formation and diffusion of interfacial reaction layers, and explores the effects of steel composition, surface oxidation state, and cooling rate on interfacial stability. The results indicate that magnesium element helps to improve the continuity of the interface coated layer, while aluminum content has a significant regulatory effect on the thickness and adhesion strength of the reaction layer, providing reference for the design of high-performance coatings.

1. Introduction

The interface bonding state between the coating and the steel substrate is the core factor determining the service life of the coating, and the chemical reactions, element diffusion behavior, and phase composition at the interface directly determine the stability of the interface structure and the adhesion effect of the coating. Existing research has mostly focused on optimizing the corrosion resistance of coatings[1], addressing the interfacial chemical reaction mechanisms of coatings with different composition systems, as well as the synergistic effects of alloy elements, substrate characteristics, and process parameters on adhesion performance. However, there is still a lack of systematic comparative analysis and quantitative data support.

Based on this, this article selects three types of coating systems, namely pure zinc based, Zn-55Al aluminum zinc based, and Zn-6Al-3Mg zinc aluminum magnesium, which are widely used in industrial applications, as research objects. The interface reaction characteristics under continuous hot plating and electroplating processes are compared and analyzed, and the role of aluminum and magnesium alloy elements in the formation of interface reaction layers and element diffusion is clarified. The influence of steel based composition, surface oxidation state, and cooling rate on interface stability and adhesion performance is quantitatively analyzed, in order to provide reference for the composition design and process optimization of high-performance alloy coatings.

2. Experimental Materials and Methods

2.1 Test Materials

The experiment selected Q235 low carbon steel commonly used in industry as the base material, with a sample size of 100mm × 50mm × 2mm. Its main chemical composition (mass fraction) is: C 0.12%0.20%, Mn 0.30%0.65%, Si ≤0.30%, S ≤0.045%, P ≤0.045%, The remaining amount is Fe.

Three types of coating systems were designed for the experiment, namely: pure zinc coating (Zn, purity ≥ 99.99%), aluminum zinc based coating (Zn-55Al, mass fraction, the same below), and zinc aluminum magnesium coating (Zn-6Al-3Mg). The metal raw materials used for the plating solution were all industrial grade high-purity ingots with a purity ≥ 99.9%.

2.2 Experimental Equipment and Process

The coating preparation adopts laboratory continuous hot plating simulation testing machine and DC electroplating equipment. Hot dip plating process parameters: The sample is first washed with alkali to remove oil → acid washed with hydrochloric acid to remove rust → treated with a plating aid (NH_4Cl-$ZnCl_2$ composite system) → dried, and then immersed in a plating solution at 460 ℃ and 520 ℃ for 310 seconds. After removal, the cooling rate is controlled by three methods: air cooling, air cooling, and water cooling, with a cooling rate range of 5 ℃/s50 ℃/s. Electroplating process parameters: Potassium chloride zinc plating system is used, current density of 13A/dm ^{2}; plating solution temperature of 25 ℃, plating time of 20 minutes, and coating thickness controlled between 8-10 μ m.

To investigate the effect of oxidation state on the surface of steel substrate, the acid washed samples were subjected to three treatments: fresh surface, slight oxidation at 25℃ room temperature for 24 hours, and severe oxidation at 100℃ oven for 2 hours. To investigate the influence of steel composition, three types of low carbon steel samples with carbon contents of 0.05%, 0.12%, and 0.20% were selected for parallel testing.

2.3 Characterization and Performance Testing

The microstructure of the coating cross-section interface was observed using a Zeiss Sigma 300 scanning electron microscope (SEM), and the interface element line scanning and surface distribution analysis were performed using an X-Max 50 energy dispersive spectrometer (EDS); Use Brooke D8 Advance X-ray diffractometer (XRD) for interface phase analysis, with a testing range of 2 θ=10 °~90 ° and a scanning rate of 5 °/min.

The adhesion performance test of the coating shall be conducted in accordance with GB/T 9286-1998 "Cross cut Test for Coatings and Varnishes", and the adhesion effect shall be evaluated in combination with grades 0-5. The WDW-100 universal testing machine was used for tensile shear testing to test the interfacial adhesion strength between the coating and the steel substrate. The overlap area of the sample was 20mm × 20mm, and the tensile rate was 2mm/min. A 180 ° bending test was adopted to evaluate the anti-cracking and anti-peeling performance of the coating. The bending mandrel diameter was set to twice the sample thickness, and coating peeling at the bending position was observed. Three parallel samples were prepared for each test, and the average value of the results was adopted.

3. Results and Analysis

3.1 Interface chemical reaction characteristics of different coating systems

The interface reaction between the coating and the steel substrate is the process of mutual diffusion between metal elements in the plating solution and Fe elements in the steel substrate at high temperatures, forming an intermetallic compound layer. The type, thickness, and morphology of the interface reaction products directly determine the interface bonding state. Figure 1 shows the SEM morphology of the cross-sectional interface of three typical coating systems in the hot-dip state. Table 1 presents the statistical results of the phase composition, thickness, and adhesion strength of the interface reaction layer for different coating systems.

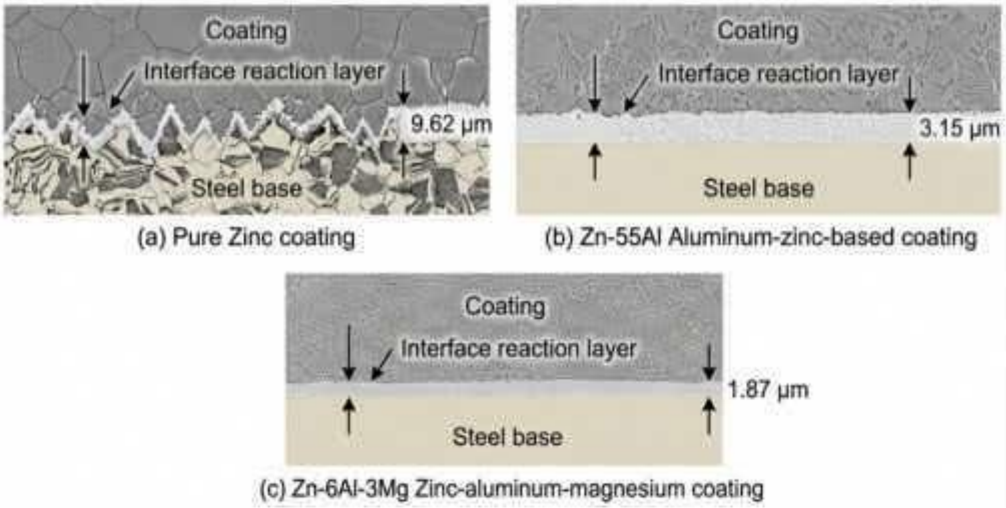

Figure 1: SEM morphology of interfaces of different coating systems (a) pure zinc coating; (b) Zn-55Al aluminum zinc based coating; (c) Zn-6Al-3Mg Zinc Aluminum Magnesium Coating

Table 1: Interface reaction characteristics and adhesion properties of different coating systems

Coating system	Main phase composition of the interface	Average thickness of interface reaction layer/μ m	Interface adhesion strength/MPa	Grid test level	180 °bending test results
Pure zinc coating	Γ-Fe3Zn10, δ-FeZn10, ζ-FeZn13	9.62	24.8	2 level	Localized cracking and detachment of the coating
Zn-55Al coating	Fe2Al5, FeAl3	3.15	32.6	1 level	No cracking or detachment
Zn-6Al-3Mg coating	Fe2Al5, MgZn2, Fe-Al-Zn	1.87	41.2	0 level	No cracking or detachment

From Figure 1 and Table 1, it can be seen that the interface reaction layer of the pure zinc coating exhibits a distinct serrated morphology, which is caused by the non-uniform growth of Fe Zn intermetallic compounds during the interdiffusion process of Fe and Zn elements. The XRD test results (Figure 2) show that the interface region of the pure zinc coating is mainly composed of three Fe Zn intermetallic compounds: Gamma Fe3Zn10, δ - FeZn10, and Zeta - FeZn13. Among them, the δ phase is the main constituent phase, accounting for more than 70%. This type of Fe Zn phase exhibits significant brittleness. When the thickness of the interface reaction layer exceeds 8 μ m, the coating is prone to cracking along the brittle phase during the stress process, leading to coating detachment. This is also the core reason for the low adhesion strength of pure zinc coatings.

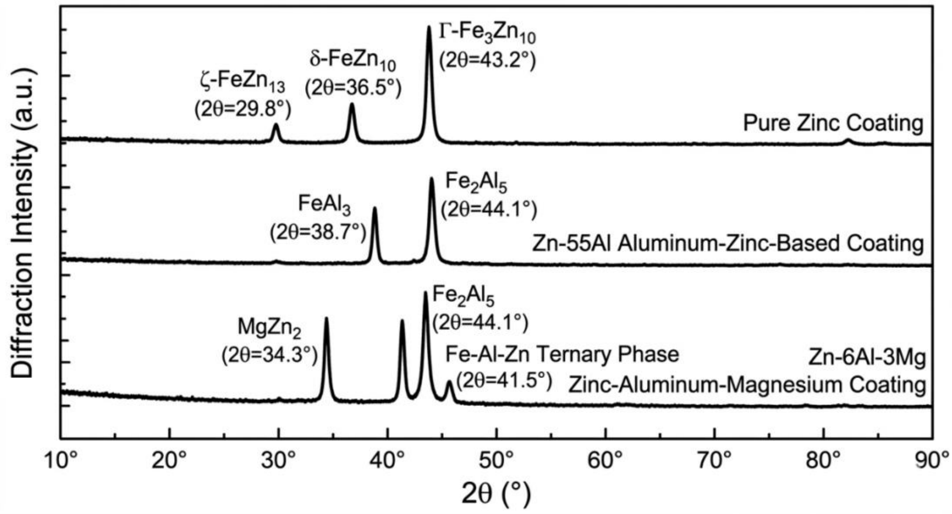

Figure 2: XRD diffraction patterns of interface regions in different coating systems

The interface reaction layer of Zn-55Al aluminum zinc based coating presents a flat and dense morphology without obvious serrated structure. The average thickness of the interface reaction layer is only 3.15 μ m, which is much lower than that of pure zinc coating. The EDS line scan results showed a significant enrichment peak of Al element at the interface, and the interdiffusion between Fe element and Zn element was significantly inhibited. The XRD results indicate that the main phases at the interface are Fe2Al5 and FeAl3 intermetallic compounds, and no obvious Fe Zn brittle phase was detected. This is because the affinity between Al element and Fe element is much higher than that of Zn element. During the hot-dip coating process, Al element will preferentially diffuse to the surface of the steel substrate and react with Fe to form a dense Fe2Al5 barrier layer, effectively hindering the interdiffusion of Fe and Zn elements, suppressing the formation of thick and brittle Fe Zn phases, thereby significantly reducing the brittleness of the interface reaction layer and improving the adhesion strength of the coating.

The interface reaction layer of Zn-6Al-3Mg zinc aluminum magnesium coating is the most uniform and dense, with an average thickness of only 1.87 μ m, and there are no defects such as voids or cracks at the interface junction. The XRD and EDS analysis results showed that in addition to Fe2Al5 phase, MgZn2 intermetallic compound and Fe Al Zn ternary phase were also generated at the interface, and Mg element exhibited uniform distribution characteristics at the interface. The addition of Mg element further refines the interface grains, transforming the interface reaction layer from columnar crystals to equiaxed crystal structure, effectively reducing the internal stress at the interface. At the same time, Mg element can fill the micro defects at the interface, improve the density of the interface, and thus achieve an adhesion strength of 41.2 MPa for the coating. The scratch test reaches level 0, and there is no cracking or detachment after 180 °bending, indicating the optimal adhesion performance.

Comparing the interface characteristics of continuous hot plating and electroplating processes, it is found that there is no obvious intermetallic compound reaction layer at the interface of electroplated pure zinc coating. The coating and steel substrate are mainly mechanically and physically bonded, with an interface adhesion strength of only 18.3 MPa, far lower than that of hot-dip pure zinc coating. Although there is no high-temperature reaction layer at the interface of the electroplated Zn Al Mg alloy coating, the metallurgical bonding tendency between the coating and the steel substrate can be improved through the co deposition of alloy elements, and the adhesion strength can reach 27.5 MPa, which is still lower than that of the hot-dip alloy coating. This indicates that the interface metallurgical reaction layer formed during the hot plating process is the core structure that ensures high adhesion performance of the coating.

3.2 The regulatory effect of alloying elements on interfacial reactions and adhesion properties

3.2.1 Effects of Aluminum Element

To clarify the regulation law of aluminum element on interface reaction, five sets of experiments were set with fixed Mg content of 0 in the plating solution and Al content of 0%, 5%, 15%, 35%, and 55%, respectively. The thickness of the interface reaction layer and the adhesion strength of the coating were tested under different Al contents. The results are shown in Figure 3.

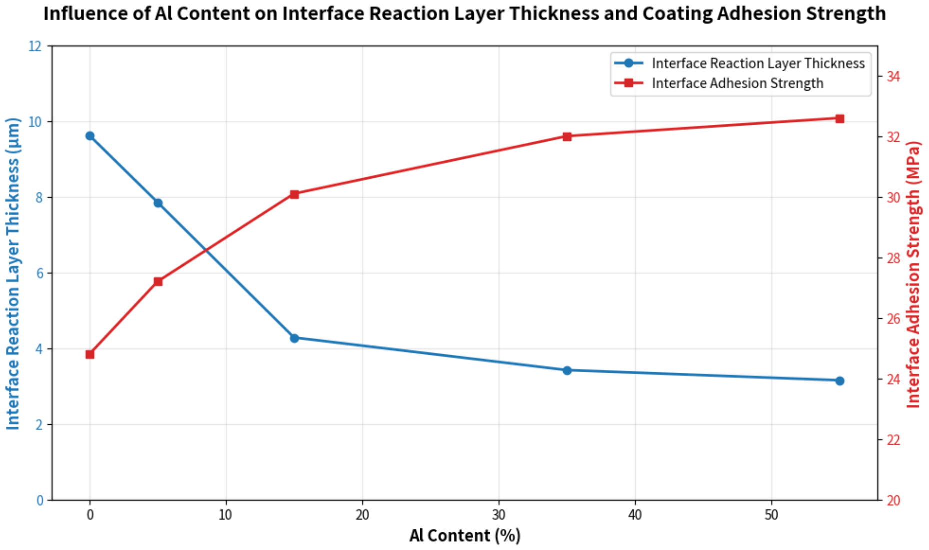

Figure 3: The Effect of Al Content on the Thickness of Interface Reaction Layer and Coating Adhesion Strength

From Figure 3, it can be seen that with the increase of Al content in the plating solution, the thickness of the interface reaction layer shows a continuous decreasing trend, while the adhesion strength of the coating shows a rapid increase followed by a stable trend. When the Al content is 0, the thickness of the interface reaction layer is 9.62 μ m, and the adhesion strength is only 24.8 MPa; When the Al content is increased to 15%, the thickness of the interface reaction layer decreases to 4.28 μ m, and the adhesion strength increases to 30.1 MPa; When the Al content reaches 55%, the thickness of the interface reaction layer decreases to 3.15 μ m, and the adhesion strength reaches 32.6MPa. Continuing to increase the Al content does not significantly improve the adhesion strength.

The core mechanism of this change pattern is that when the Al content is low, a continuous and complete Fe2Al5 barrier layer cannot be formed on the surface of the steel substrate, and Fe and Zn elements can still undergo significant interdiffusion, generating brittle Fe Zn phases. When the Al content reaches 15% or more, a continuous Fe2Al5 barrier layer can be formed, completely suppressing the formation of Fe Zn brittle phase. The thickness of the interface reaction layer is significantly reduced, the brittleness is significantly reduced, and the adhesion strength is greatly improved. When the Al content exceeds 55%, there is no significant improvement in the density of the barrier layer, and the thickness change of the interface reaction layer tends to be gentle, so there is no significant change in the adhesion strength. At the same time, excessive Al content can lead to an increase in the content of hard and brittle phases in the coating, which can actually reduce the formability of the coating. Therefore, in industrial applications, the Al content of aluminum zinc based coatings is mostly controlled at around 55%[2].

3.2.2 Effects of Magnesium Element

The Al content in the fixed plating solution was 6%, and five sets of experiments were conducted with Mg contents of 0%, 1%, 3%, 5%, and 7% to test the continuity of the interface coated layer, interface defect rate, and coating adhesion performance under different Mg contents. The results are shown in Table 2.

Table 2 Test results of coating interface performance under different Mg contents

Mg content/%	Continuity of interface coated layer/%	Interface defect rate/%	Interface adhesion strength/MPa	Interface adhesion strength retention rate/% after 720 hours of neutral salt spray
0	62.3	8.75	29.7	58.2
1	78.5	4.21	35.3	71.5
3	94.7	0.86	41.2	89.6
5	92.1	1.23	38.5	85.3
7	86.4	2.57	34.1	78.7

From Table 2 and Figure 4, it can be seen that with the increase of Mg content, the continuity of the interface coated layer and the adhesion strength of the coating show a trend of first increasing and then decreasing, reaching the optimal value at Mg content of 3%. When the Mg content is 0, the continuity of the interface coated layer is only 62.3%[3-4], and the interface defect rate is as high as 8.75%. After 720 hours of neutral salt spray test, the interface adhesion strength retention rate is only 58.2%. When the Mg content is increased to 3%, the continuity of the interface coated layer reaches 94.7%, the interface defect rate drops to 0.86%, and the adhesion strength retention rate after salt spray test reaches 89.6%. The long-term service stability is significantly improved.

The mechanism of action of magnesium element is mainly reflected in three aspects: firstly, Mg element can form MgZn2 intermetallic compounds with Zn element at the interface, refine the interface grains, reduce the internal stress at the interface, and reduce the occurrence of micro cracks and defects such as voids; Secondly, the electrode potential of Mg element is lower than that of Zn and Al, and it can preferentially undergo anodic dissolution in corrosive environments, forming a dense magnesium containing coated film at the interface, effectively blocking the penetration of corrosive media into the interface and avoiding corrosion delamination at the interface; Thirdly, Mg element can suppress the abnormal growth of Fe2Al5 phase at the interface, keeping the interface reaction layer uniform and thin, further reducing interface brittleness. When the Mg content exceeds 5%, brittle intermetallic compound slag phase of Mg Zn is easily generated in the plating solution, leading to an increase in internal defects of the coating and a decrease in adhesion performance. Therefore, the Mg content of zinc aluminum magnesium coatings is mostly controlled between 1% and 3%.

3.3 Influence of Matrix and Process Parameters on Interface Adhesion Effect

3.3.1 Influence of Steel Foundation Composition

Three types of low-carbon steel with carbon contents of 0.05%, 0.12%, and 0.20% were selected to prepare pure zinc coatings. The thickness and adhesion strength of the interface reaction layer were tested, and the results are shown in Table 3.

Table 3: Effect of Carbon Content in Steel Base on the Interface Properties of Pure Zinc Coating

Carbon content of steel base/%	Interface reaction layer thickness/μ m	Interface adhesion strength/MPa	Proportion of interface Fe Zn brittle phase/%
0.05	6.34	29.5	58.7
0.12	9.62	24.8	72.4
0.20	13.57	19.2	85.1

From Table 3, it can be seen that as the carbon content of the steel base increases, the thickness of the interface reaction layer of the pure zinc coating significantly increases, the proportion of Fe Zn brittle phase continues to increase, and the interface adhesion strength decreases significantly. This is because the C element in the steel matrix significantly promotes the interdiffusion of Fe and Zn elements, accelerates the growth of Fe Zn intermetallic compounds, increases the thickness and brittleness of the interface reaction layer, and is prone to intergranular cracking during the stress process, resulting in a decrease in the adhesion performance of the coating. For alloy coatings, due to the presence of Fe2Al5 barrier layer, the influence of steel based carbon content on interfacial reaction is significantly reduced. When the carbon content is increased from 0.05% to 0.20%, the thickness change of the interfacial reaction layer of Zn-55Al coating does not exceed 0.5 μ m, and the change rate of adhesion strength is less than 8%.

3.3.2 Influence of surface oxidation state on steel substrate

Three treatments were applied to the steel substrate, including fresh surface treatment, slight oxidation, and severe oxidation, to prepare Zn-6Al-3Mg coating. The interfacial adhesion performance was tested, and the results are shown in Table 4.

Table 4: The Influence of Oxidation State on the Interface Properties of Coating on Steel Base Surface

Surface oxidation state	Void rate at interface junction/%	Interface adhesion strength/MPa	Grid test level
Fresh surface	0.32	41.2	Level 0
slight oxidation	3.75	32.6	Level 2
Severe oxidation	11.84	20.5	Level 4

From Table 4, it can be seen that the higher the degree of oxidation on the steel substrate surface, the higher the porosity at the interface junction, and the lower the adhesion strength of the coating. This is because the oxide scale on the surface of the steel substrate (mainly FeO, Fe2O3) hinders the contact and interdiffusion of elements such as Al and Zn with Fe, making it impossible to form a continuous and dense interface metallurgical reaction layer, resulting in a large number of voids and unbound areas at the interface, and a significant decrease in the bonding force between the coating and the steel substrate[5]. Therefore, in the process of coating preparation, it is necessary to ensure the acid pickling and rust removal effect on the steel substrate surface to avoid residual oxide layer, which is a prerequisite for ensuring the adhesion performance of the coating.

3.3.3 Effects of Cooling Rate

Three cooling methods were set up: air cooling (5 ℃/s), air cooling (20 ℃/s), and water cooling (50 ℃/s) to prepare Zn-6Al-3Mg coating. The thickness of the interface reaction layer and the adhesion strength were tested, and the results are shown in Figure 4.

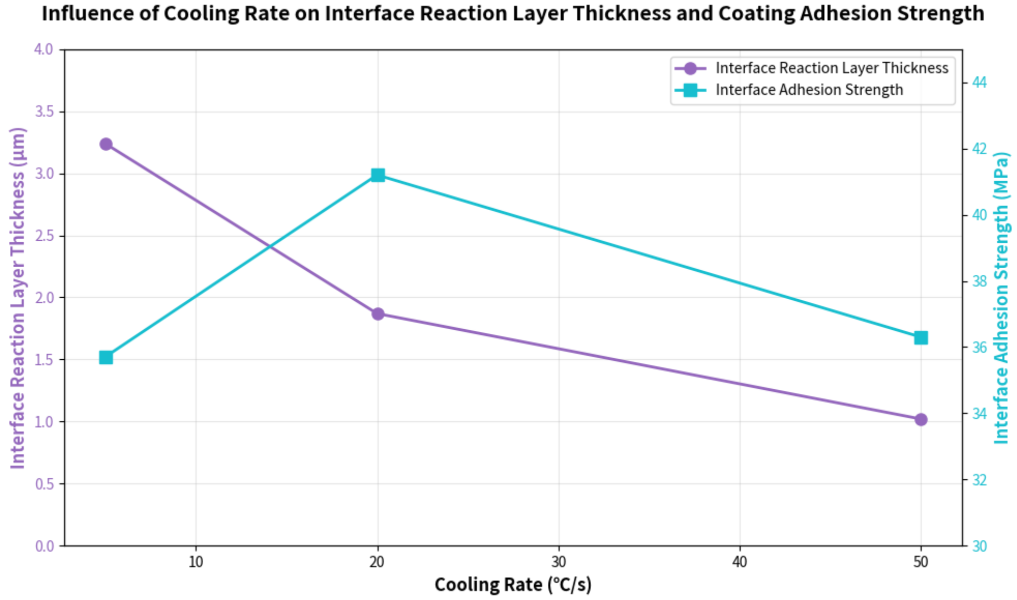

Figure 4: Effect of Cooling Rate on Interface Reaction Layer Thickness and Coating Adhesion Strength

From Figure 4, it can be seen that with the increase of cooling rate, the thickness of the interface reaction layer continues to decrease, while the adhesion strength of the coating shows a trend of first increasing and then decreasing, reaching the optimal value when air-cooled at 20 ℃/s. When the cooling rate is 5 ℃/s, the sample stays in the high-temperature zone for a long time, the interfacial elements diffuse sufficiently, the reaction layer thickness reaches 3.24 μ m, the proportion of interface brittle phases increases, and the adhesion strength is only 35.7 MPa. When the cooling rate is increased to 20 ℃/s, the thickness of the interface reaction layer decreases to 1.87 μ m, and there are no brittle phases with abnormal growth. The internal stress at the interface is relatively small, and the adhesion strength reaches 41.2 MPa. When the cooling rate is increased to 50 ℃/s, the thickness of the interface reaction layer further decreases to 1.02 μ m. However, due to the rapid cooling rate, the difference in thermal expansion coefficient between the coating and the steel substrate leads to a sharp increase in internal stress at the interface, which is prone to micro cracks and a decrease in adhesion strength to 36.3MPa. This indicates that the cooling rate needs to be controlled within a reasonable range, to avoid thickening of the reaction layer due to prolonged high-temperature residence time, and to prevent excessive internal stress caused by rapid cooling, thereby ensuring the adhesion performance of the coating.

4. Conclusion

This article takes three typical metal coating systems, namely pure zinc based, aluminum zinc based, and zinc aluminum magnesium, as the research objects, and systematically explores the interface chemical reaction characteristics and the influence mechanism of alloy elements, substrate, and process parameters on the adhesion performance of coatings. Research has found that there are significant differences in the interface reaction products and structures among the three types of coating systems. Among them, pure zinc coatings form thick and brittle Fe Zn intermetallic compound layers at the interface, with the worst adhesion performance. The interface of aluminum zinc based coating forms a dense Fe_2Al_5 barrier layer, which significantly improves the adhesion performance. The interface of zinc aluminum magnesium coating is the most uniform and dense, exhibiting the best adhesion performance. In terms of the influence of alloying elements, Al can effectively regulate the thickness and brittleness of the interface reaction layer by forming a Fe_2Al_5 barrier layer, which is the key to improving the adhesion strength. The best effect is achieved when its content is around 55%. Mg significantly enhances the continuity and density of the interface coated layer, which is beneficial for the long-term service stability of the coating. The optimal content is about 3%. The substrate conditions and process parameters also have a significant impact on the adhesion performance. An increase in carbon content in the steel substrate will accelerate the growth of Fe Zn brittle phases and weaken the adhesion performance. The presence of surface oxide layer will hinder interfacial metallurgical reactions and reduce bonding strength. A moderate cooling rate (about 20 ℃/s) can achieve a balance between thinning the interface reaction layer and controlling internal stress, thereby obtaining the optimal coating adhesion performance.

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