Changing Regularity of Structure and Phase of HDA-steel under Diffusion Treatment

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Abstract

In this paper, the changing regularity of structure and phase of hot dip aluminizing (HDA) steel after diffusion treatment at different temperature and time was studied by means of SEM and XRD. The results showed that, after diffusion treatment at 550°C~650°C,the surface layer of the HDA coating , which is the main source to apply aluminum to the HDA coating, gradually becomes thinner, even disappears. With diffusion temperature increasing between 550°C and 650°C and time prolonging, the transition layer in HDA coating gradually becomes thicker. As a result of diffusion treatment at 700°C~900°C, the aluminum in the HDA coating quickly evaporated due to without protecting the surface of HDA-steel with protective materials. This gives rise to the appearing of internal oxidation in the transition layer. With diffusion temperature increasing between 700°C and 900°C, diffraction intension of FeAl phase in the transition layer in XRD spectrums becomes stronger, but Fe_2Al_5 phase still exists in the transition layer. After diffusion treatment at 900°C, the thickness of FeAl phase becomes from 25 μm to 50 μm . FeAl phase existing in the transition layer strikingly improves oxidation resistance of HDA coating on steel at high temperature in air.

Keywords: hot-dip aluminum, dip diffusion aluminum, surface coating, transitional layer

1. Introduction

Recent years, hot dip aluminized steel (HDA-steel) was applied widely in such fields as petrochemical and chemical industry, building engineering, electric power communication, transportation and oceanographic engineering. Hot dip aluminizing process is a technology to form a kind of surface composite coating on steel materials. The coating consists of aluminum surface layer and Fe-Al intermetallic compound transition layer[1, 2, 3]. The aluminum surface layer enhanced hot dip aluminizing steel's corrosion resistance because it is apt to form stable compact Al_2O_3 film .And the transition layer offered hot dip aluminum coating oxidation resistance at high temperature.

In recent studies on HDA, more attentions were paid on technology and anti-corrosion performance but less on oxidation resistance at high-temperature[4, 5]. It was generally accepted that HDA provided steel with both corrosion and heat resistance, and can be directly applied under corrosive and high temperature surroundings .In practice, as long as hot dip aluminum coating is continuous and compact, it can directly been applied to environment of corrosion and high temperature, but the oxidation resistance of the coating is different from corrosion resistance .Below 450°C, even if heated over a long time, the surface appearance colour of HAD-steel coating did not changed .But at high temperature and in temperature going up processes, the oxidation resistance performance not only depends on the Al surface layer ,but primarily also depends on the Fe-Al intermetallic compound in transition layer .But the microstructure evolution of hot-dip aluminum coating was ignored[6, 7].So it is important to analyses the changing regularity of microstructure of HDA-steel under diffusion treatment at different temperature and time respectively.

In the present paper, the changing regularity of structure and phase of hot dip aluminizing (HDA) steel under diffusion treatment at different temperature and time was studied by means of SEM and XRD ,and the oxidation resistance of hot dip aluminum steel was tested with high temperature oxidation test.

2. Experimental

The specimen were Q235 steel plates (10 mm × 10 mm × 1.5 mm) and cylinders (Φ10 mm × 15 mm) respectively. The HDA procedure is as follows:

Degreasing → Rinse → Pickling → Rinse → Activating → Rinse → Fluxing → Drying → Hot Dip Aluminizing. HDA condition is at 720°C for 1min and 6 minutes respectively. The surface coating is 70μm thick . Diffusion treatment conditions are at 550°C, 650°C, 750°C, 850°C and 950°C for 30 or 60 min.

High-temperature oxidation tests were conducted in a self-made oxidation oven. Firstly, HDA-Q235 steel specimens were put into silica crucible that had been baked to constant weight. Then, the silica crucible was heated to 800°C. Finally, the specimens in silica crucible were oxidized in air at 800°C for 300 hour. XRD analysis was carried out on Japanese D/Max-rB XRD instrument. Graphite monochromatic wave filter and Cu target were employed at 40 KW and 120 mA, and morphology of HDA-coating observation was carried out on KYKY-2800B SEM .

3. Results and Discussion

3.1 Changing Regularity of Microstructure of HDA-Coating Treated at 550°C~650°C

The results of SEM observation on the section morphology after diffusion treatment at 550°C for 30 min showed that surface layer became thinner (shown in Fig.1), and the thickness of transition layer did not change. At 600°C, prolonging diffusion treatment time to 60 min, surface layer almost disappeared (shown in Fig 2), transition layer became thicker, many tiny cracks and small amount of corrosion spots appeared. This is because surface layer is very thin and disappeared quickly after diffusion treatment. The results of SEM observation showed that, for 720°C, 6 min hot dip aluminizing specimen, after diffusion treatment at 600°C for 60 min, the thickness of surface layer is also 15~20 μm, and transition layer gradually became thicker .After diffusion treatment at 650°C for 60 min, for the specimen formed at 720°C 6 min, the surface layer of HAD-coating nearly disappeared.

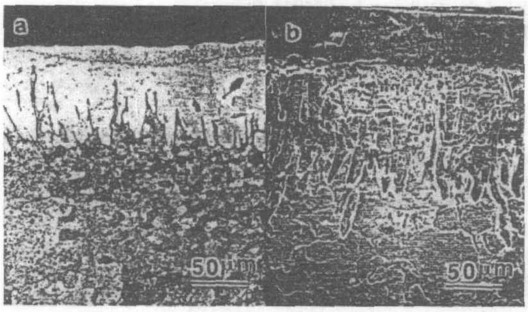

Fig. 1 SEM morphology of HDA coating diffused at 550°C for 30 min HDA process: (a) 720°C 1 min; (b) 720°C 6 min

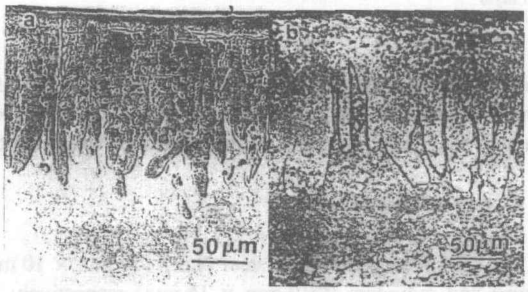

Fig. 2 SEM morphology of HDA coating diffused at 600°C for 60 min HDA process: (a) 720°C 1 min; (b) 720°C 6 min

It is generally believed[8] that Al and Fe atom can diffuse into each other when temperature exceeding 480°C. Experimental results showed that the thicken rate of transitional layer is higher than the oxidation rate of the surface layer under diffusion treatment at 600°C. But, if the surface layer of HDA-coating is so thin that it could not become to continuous Al_2O_3 film, oxygen atom in air will come from the surface layer into the transition layer and react with Fe and Al there. This will decline the oxidation resistance of HDA-coating. So, in order to improve the oxidation resistance of HAD-steel, the thickness of surface layer must exceed a certain degree of thickness.

3.2 XRD Analysis of Surface Layer of HDA-coating Treated at High Temperature

Fig. 3. shows the XRD pattern of HDA-coating on steel after diffusion treatment at 550~650°C for different temperature. It can be seen that the diffraction peak of Al (200) face of HDA-coating is obvious (shown in Fig.3a), this result shows that Al and Fe_2Al_5 are main component on the surface and transition layer respectively. With diffusion treatment temperature raising and time prolonging, the diffraction peak strength of Al (002) face weaken (shown in Fig. 3b) and the diffraction peak of Al (111) face almost disappears. This result shows that, with temperature raising and time prolonging, Al content in surface layer decreased, and Fe_2Al_5 phase content increased. Raising diffusion temperature further and prolonging time to 60 min (shown in Fig. 3c), there was little pure Al in the surface layer and $FeAl_3$ phase disappeared. But Fe_2Al_5 phase diffraction peak still appeared in the XRD pattern because Fe_2Al_5 phase is the main component in the hot dip aluminum coating .As the thickness of surface layer reduces after diffusion treatment and Al is out of supply, diffraction peaks of FeAl (200) phase appear in XRD pattern. This result shows that FeAl phase content gradually increased after diffusion treatment .

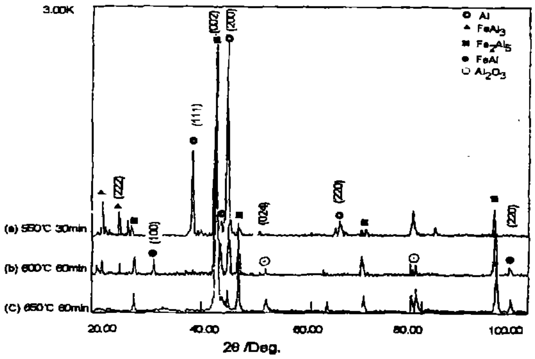

Fig. 3 XRD analysis of HDA coating diffused at different times from 550°C to 650°C

3.3 Changing Regularity of Microstructure of HDA-coating Treated at High Temperature

Fig. 4 shows the SEM photograph of HDA-coating diffused at 700°C for 30 min. From the morphology of view ,the surface layer of the coating nearly disappeared and the transition layer gradually become thicker. With the thickness of transition layer increasing, needle-like phases appears in the front of serrated-shape structure (shown in Fig. 3 A region). Spectroscopy analysis show that the phase is Fe_3Al which Al content is 71.4at %.

Fig. 4 SEM morphology of HDA coating diffused at 700°C for 30 min

Fig. 5 (a, b, c)showed the SEM photograph of HDA-coating after diffused at 850°C for 30 min, 60 min and at 900°C for 30 min respectively. It can be seen that, with the diffusion treatment time prolonging, the serrated-shape structure in transition layer becomes from sharp shape to rounder and even shape. $FeAl_3$ phase still exists in shape of needle-like, discontinuous strip and which is parallel with the sharp of serrated-shape structure, and grey-white region appears between needle-like strip and the sharp of serrated-shape structure in transition layer.

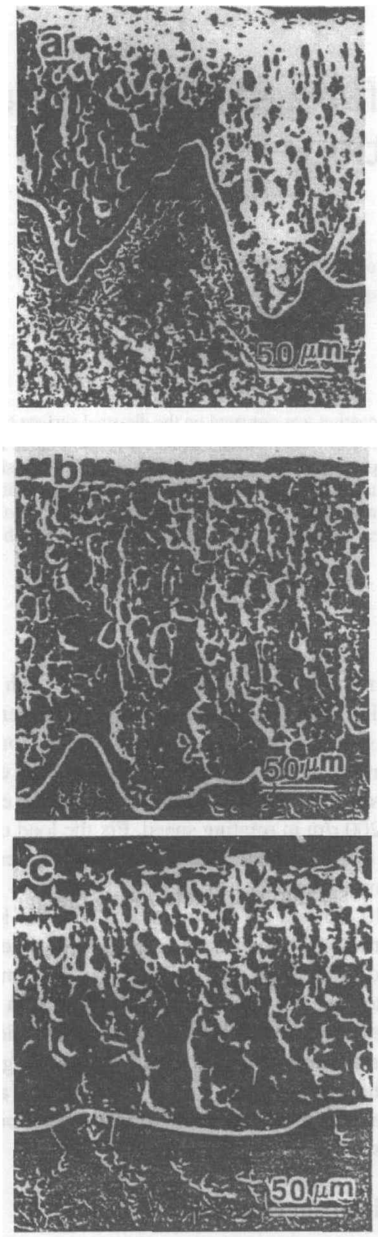

Fig. 5 SEM morphology of HDA coating diffused at 850°C~900°C for 30 min a. 850°C 30 min; b. 850°C 60 min; c. 900°C 30 min

Spectroscopy analysis showed that it was FeAl phase whose Al content is 38.72at%. It can be seen, after diffusion treatment at 850°C, FeAl phase could become independent phase layer and its thickness inscreased with diffusion treatment time prolonging. This phenomenon accords with FeAl equilibrium phase diagram. This is because that surface layer disappeared and Al atom was out of supply after diffusion treatment, and then the Fe_2Al_5 phase could not grow up quickly. The results also show that Fe_2Al_5 phase gradually transform into FeAl phase. Furthermore raising diffusion treatment temperature to 900°C, the serrated-shape structure disappears, and then, the thickness of FeAl phase increased to 50 μm. At the same time, the needle-like Fe_3Al phase becomes loose and exists in single discontinuous shape in transition layer substrate. SEM observation results show that, with diffusion temperature raising and time prolonging, the Al atom in transition layer will diffuse toward inner layer and turn into FeAl phase.

3.4 Oxidation Resistance of HDA-coating after Diffusion Treatment

Oxidation tests at 800°C in air showed that weight gained differed obviously under different diffusion treatment conditions, but all kinetic curves accorded with parabola growth rule. FeAl phase existing in the transition layer strikingly improves the oxidation resistance of HDA coating on steel at high temperature in air.

To sum up, it can be recognized that it was internal metallic compound in transition layer that acted as high temperature oxidation resistance. The oxidation resistance changed along with continuity, density, phase composition and crystal structure. So, to improve oxidation resistance of HDA coatings, suitable phase composition must be achieved through optimizing diffusion process.

4. Conclusion

(1) After diffusion treatment at 550°C~650°C, the surface layer of HDA coating gradually becomes thinner, even disappears, the transition layer of HDA coating gradually becomes thicker.

(2) After diffusion treatment at 700°C~900°C, in XRD spectrum, diffraction peaks for FeAl phase in the transition layer become stronger, but Fe_2Al_5 phase still exists in the transition layer.

(3) After diffusion treatment at 900°C, the thickness of FeAl phase layer increased from 25 μm to 50 μm. FeAl phase existing in the transition layer strikingly improves the oxidation resistance of HDA coating on steel at high temperature.

Acknowledgements

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