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The effects of zinc bath temperature on the coating growth behavior of reactive steel

*Jianhua Wang, Hao Tu, Bicao Peng, Xinming Wang, Fucheng Yin, Xuping Su**

School of Mechanical Engineering, Xiangtan University, Xiangtan, 411105, China
Key Laboratory of Materials Design and Preparation Technology of Hunan Province, Xiangtan University, Xiangtan, 411105, China

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ABSTRACT

The purpose of this work is to identify the influence of zinc bath temperature on the morphology and the thickness of reactive steel (Fe–0.1 wt.%Si alloy) coatings. The Fe–0.1 wt.% Si samples were galvanized for 3 min at temperatures in the range of 450–530 °C in steps of 10 °C. The coatings were characterized by using scanning electron microscopy/energy dispersive X-rays analysis. It was found that the coating thickness reaches the maximum at 470 °C and the minimum at 500 °C, respectively. When the reactive steel is galvanized at temperatures in the range of 450–490 °C, the coatings have a loose ζ layer on the top of a compact δ layer. With the increase of the galvanizing temperature, the ζ layer becomes looser. When the temperature is at 500 °C, the ζ phase disappears. With the increase of temperature, the coatings change to be a diffuse-Δ layer (δ+ liquid zinc).

1. Introduction

According to the Fe–Zn phase diagram, when iron is immersed in molten zinc at the typical galvanizing temperature (450–490 °C), the following layers should form: zinc saturated α-iron, Γ phase layer, Γ_1 phase layer, δ phase layer, ζ phase layer and η phase layer [1]. Galvanizing temperature is the key parameter in hot-dip galvanizing. Iron loss experiment was always used to study the effect of galvanizing temperature on the kinetics of the reaction between liquid zinc and pure iron [2], but it is probably not accurate because during the stripping process some unalloyed iron is removed by acid [3]. Verma [4] compared the coatings formed in the temperature range of 520–550 °C with that formed at 450 °C. It was found that the coating thickness reaches the maximum at 530 °C when low carbon steel (0.042 wt.%C, 0.2 wt.%Mn) was galvanized in zinc bath.

Horstmann [5] pointed out that the compositions in the steel have considerable effects on hot-dip galvanizing, especially the element silicon. Silicon is frequently introduced into steels either as a deoxidant in continuous casting in the steel making practice or as a strengthening alloying element [6]. However, in general galvanizing, the presence of silicon at certain levels (near 0.1 wt.% and above 0.3 wt.%) in steels, known as reactive steels, gives rise to dull grey coatings of excessive thickness, which results in the deterioration of the coating quality [7,8]. The low insolubility of silicon in the ζ phase leads to its segregation to phase boundaries and the formation of liquid zinc pockets between the ζ crystallites. This results in the formation of an incoherent and loose ζ layer in the coating [9]. The liquid zinc directly attacks the substrate or δ layer which leads to an excessive iron loss and the formation of ζ crystallites in the liquid [10]. Foct et al. [11] proposed that a thin layer of liquid

* *Corresponding authors.* Tel.: +86 0732 8292213; fax: +86 0732 8292210.
E-mail addresses: xuping@xtu.edu.cn (X. Su), super_wang111@hotmail.com (J. Wang).

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zinc saturated with silicon on the steel surface lead to the loose and overly-thick coating during galvanization. So it is significant to study systematically the coating growth behavior of reactive steel at different temperatures. The main objective of this work is to explore the effect of galvanized temperature on the coating microstructure and growth laws of the reactive steel (Fe–0.1 wt.%Si).

2. Experimental

The reactive steel (Fe–0.1 wt.%Si) was used in the experiment. The composition of the alloy is listed in Table 1. The Fe–0.1 wt.%Si alloy was prepared by smelting the mixture of commercial pure iron and silicon powders. The purity of the silicon powders is 99.99 wt.%. The total weight of the sample for each melting was 100 g. The samples were melted under argon atmosphere in a non-consumable tungsten electrode smelting furnace and then annealed at 800 °C for 15 days. After that the samples were cut into specimens of 12 mm × 12 mm × 2 mm. The specimens were degreased for 15 min in an alkaline cleaner, followed by pickling in 100–200 g/l HCl for 5 min. Next the sheets were dipped in a flux bath consisting of 500 g/l of 25/75 wt.% ZnCl2/NH4Cl salt solution which was kept at 80 °C. The fluxed sheets were dried for 30 min at 70 °C in a drying box. Then the sheets were galvanized in the entire temperature range of 450–530 °C in steps of 10 °C for 3 min. The zinc bath consists of pure zinc (99.5 wt.%Zn) containing almost no Al, Pb and Fe. Three specimens were galvanized at each temperature in order to obtain more accurate and stable experimental results. All hot-dip galvanized specimens were cross-sectioned, embedded and polished. A nital solution (nitric acid: 4 wt.%) was used to reveal the microstructures of the specimens. A JSM-6360 scanning electron microscope equipped with an OXFORD INCA energy dispersive X-ray spectroscope was utilized to study the morphology and chemical compositions of various phases in the coatings.

3. Results and Discussion

The reactive steel (Fe–0.1 wt.%Si) sheets were galvanized in the entire temperature range of 450–530 °C in steps of 10 °C with immersion time of 3 min, then cross-sectioned and studied by using SEM/EDS. The depth of the coating (only include ζ and δ phase layer) of each specimen was measured ten times at different positions. Then thirty measured values of three specimens at each galvanizing temperature were averaged as the coating thickness. The measured results of the coating thickness are shown in Fig. 1.

The coating thickness varies significantly in the temperature range of 450–530 °C as shown in the figure. The coating thickness reaches a maximum at 470 °C. Below 470 °C, the coating

Table 1 – Composition of Fe–0.1 wt.%Si alloy.

Element	C	S	Mn	Si	P	Cr	Mo	Fe
Fe–Si alloy	0.0073	0.0077	0.127	0.102	0.0143	0.0262-	0.003-	Bal.

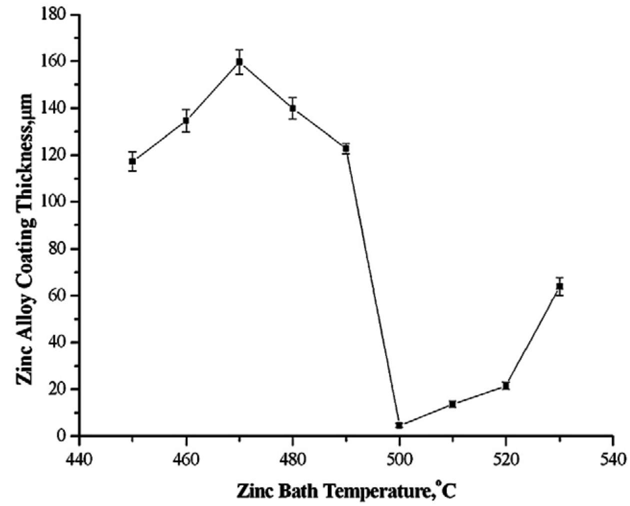

Fig. 1 – Coating thickness as a function of temperature.

thickness of the galvanized reactive steel increases with the increase of temperature. However, when the temperature exceeds 470 °C, the coating thickness decreases noticeably with the increase of temperature. The coating thickness of Fe–0.1 wt.%Si alloy reaches a minimum at 500 °C, then increases again with the increase of temperature in the range of 510–530 °C.

According to the characters of the morphology and the thickness of the coatings in the range of 450–530 °C, the entire galvanizing temperature ranges for Fe–0.1 wt.%Si alloy can be divided into five partial ranges: (1)450–460 °C; (2)470 °C; (3)480–490 °C; (4)500 °C and (5)510–530 °C.

When iron is immersed in molten zinc at the typical galvanizing temperatures (450 °C–490 °C), according to the Fe–Zn phase diagram, Horstmann [12] proposed that the following layers should form: zinc saturated α-iron, gamma (Γ) phase layer, gamma1 (Γ1) phase layer, delta (δ) phase layer, zeta (ζ) phase layer and an eta (η) phase layer. However, the sequential nucleation of the Fe–Zn phase occurs at the interface beginning with (1) the zeta (ζ) phase layer, followed by (2) the delta (δ) phase layer, and after some incubation time, (3) the gamma (Γ) phase layer.

The effect of temperature on the kinetics of the reaction between liquid zinc and pure iron, based on iron loss experiments [2], shows that up to 495 °C the total layer kinetics for long immersion times is parabolic(lower parabolic region), and it is again parabolic above 520 °C(upper parabolic). Linear attack (linear region) occurs between these two regions, but short time experiments up to 30 min also show parabolic attack in this region.

The representative SEM micrographs of the coatings of the galvanized Fe–0.1 wt.%Si alloy are shown in Fig. 2. Table 2 averages the results of the EDS analysis of these phases corresponding to Fig. 2(a)–(e), respectively. Comparing the analysis results with the composition variation range of Fe–Zn phases listed in Table 3 [13], it was found that the coatings illustrated in Fig. 2(a)–(c) consist of primarily zeta (ζ), delta (δ) and eta(η) phase. The coating illustrated in Fig. 2(d) is comprised of only the delta (δ) and eta(η) phases. The coating in Fig. 2(e) exists in the form of a diffuse-Δ layer (δ+ liquid zinc) and eta(η) phase, which is comprised of primarily delta (δ)