Surface & Coatings Technology 240 (2014) 269–274

Contents lists available at ScienceDirect

Surface & Coatings Technology

journal homepage: www.elsevier.com/locate/surfcoat

Effect of strip entry temperature on the formation of interfacial layer during hot-dip galvanizing of press-hardened steel

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ARTICLE INFO

Article history:
Received 14 July 2013
Accepted in revised form 17 December 2013
Available online 29 December 2013

Keywords:
Hot-dip galvanizing
Interface layer
Hot stamping
Press-hardened steel

ABSTRACT

Strip entry temperature is one of the important factors that affect alloying reactions at the steel/coating interface. This study focuses on the influence of strip entry temperature on phase formation at the press-hardened steel (PH) substrate/coating interface in zinc baths with 0.12 wt.% and 0.2 wt.% Al content respectively. The microstructure of the interfacial layer was characterized by Scanning Electron Microscope (SEM) and Glow Discharge Optical Emission Spectrometer (GDOES). It was determined that the microstructure components of steel/coating interface are significantly influenced by the strip entry temperature within 440–480 °C. In the zinc bath with 0.12 wt.% Al, the interfacial layer consists of mainly ζ phase ($FeZn_{13}$), and the size of the ζ phase increases with the strip temperature. However, the amount of ζ phases and the Al content of the interfacial layer decreases with the strip temperature. Meanwhile, in the zinc bath with 0.2 wt.% Al, a continuous inhibition layer Fe_2Al_5 was formed on the steel surface. The Al enrichment at the interfacial layer increased with increasing strip temperature, accompanied with which is the coarser inhibition layer morphology.

1. Introduction

To meet modern fuel consumption saving and passengers' safety guarantee, the automotive industry is increasing the use of press-hardened steel [1]. Press-hardened steel has a good formability and lower springback during hot stamping process. However, the steel surface is easy to be oxidized and decarburized during heating up and hot-stamped parts have a poor corrosion resistance. So, different types of coatings have been developed to overcome these problems, such as Al–Si coating, zinc based coating and Zn–Ni coating [1–4]. To meet the demand of the galvanic edge protection, zinc based coatings for hot stamping parts were developed, the coating still provides an adequate cathode corrosion protection after austenitization, and the zinc based coatings also have the advantage of high productivity and low cost [5].

During hot dip galvanizing, interface reactions take place, and $Fe_{2-}Al_5Zn_x$, $FeAl_3$, ζ or δ phases form on the strip surface [6]. The morphology of this interface layer depends on the chemical composition of steel substrate, strip entry temperature, bath temperature, immersion time and Al content in zinc bath [7–11]. Many experimental and theoretical studies on this interfacial phase transformation have been made in the last decades [12,13]. However, there is very few work that dealt with the effect of strip temperature on interface layer formation [8,14], and there is no report about the hot-dip galvanization of press-hardened steel.

The press hardened $22MnB_5$ is a boron-added C–Mn steel. During the in-line intercritical annealing prior to hot-dip galvanizing, alloy element Mn tends to segregate at the steel surface and form oxides which cannot be reduced by classic industrial annealing atmospheres [15]. Some investigations claimed that MnO formed on the steel surfaces can be reduced by dissolved or effective Al in the Zn bath during galvanizing [16,17]. Furthermore, the consumption of Al will cause the unbalance of Fe–Al phase layer formation [15]. So far, it is still not clear how the strip entry temperature exactly affects interfacial reactions on the surface of high strength steel during hot-dipping galvanizing.

In order to control the microstructure of galvanized coatings and the consumption of Al during galvanizing, it is of critical importance to understand the phase transformation behaviors that occurred at the liquid zinc/steel substrate interface. The present study aims to investigate the effect of strip entry temperature on the formation of interfacial layer during hot-dip galvanizing process. Alloying element segregation and the occurrence of selective oxidation at the steel surface prior to galvanizing have been analyzed, and the chemistry and morphology of interfacial layer have been characterized and interpreted in relation to the

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http://dx.doi.org/10.1016/j.surfcoat.2013.12.040

Table 1
Composition of 22MnB5 steels (wt.%).

C	Mn	Si	Al	P	S	N	Ti	B	Cr
0.2207	1.202	0.279	0.0401	0.0139	0.0048	0.0027	0.0349	0.0028	0.197

Table 2
Process parameters in hot-dip galvanizing simulation experiments.

Strip entry temperature (°C)	480	460	440
Dissolved Al in bath (wt.%)	0.12	0.20	

different strip temperatures under the 0.12 wt.% and 0.2 wt.% bath Al content.

2. Experimental procedure

The cold-rolled 22MnB5 steel sheets having a thickness of 1.5 mm were employed for the current work, its chemical composition is listed in Table 1. The steel sheets were cut to sheets with dimension of 220 × 120 mm. Prior to heat treatment the steel sheets were degreased in a solution of 2 pct degreasing agent (SD-205) heated to 60 °C, rinsed with water and dried with warm air. The whole experiments were carried out in a hot-dip galvanizing simulator (IWATANI SURTEC HDPS-EUA IV). A K-type thermocouple was welded onto the surface of the steel sample to control the sample's temperature during heating up and cooling down. The samples were heated in infrared furnace and cooling was performed using two parallel gas platens. The samples were heated to 800 °C at a rate of 10 K s^{-1}, soaked for 60 s, followed by cooling down to various strip entry temperatures and a second soaking for 10 s before entering into the zinc bath. According to classic industrial practice, all samples were annealed in an atmosphere of 95% N_2 + 5% H_2 at a dew point of −30 °C.

In line with typical industrial galvanizing process, the bath composition was selected as iron saturated, with 0.12 and 0.20 wt.% dissolved Al respectively. The bath temperature was 460 °C and dipping time was 3 s, which equals the typical bath temperature and dipping time of a strip entry section during continuous hot-dip galvanizing. Three strip entry temperatures were chosen to examine the effect of this parameter on the formation of interface layer. The experimental parameters are summarized in Table 2.

To study the characteristics of the interfacial layer, the top pure Zn layer had been dissolved in 10 vol.% HCl with adding 5.0 wt.% sodium thiosulfate about 1 min. SEM/EDS (EVO-MA25) was employed to characterize the oxidized steel surface and the interfacial layer. A GDOES 750A was used to analyze the composition profile of the oxidized steel surface and interfacial layer.

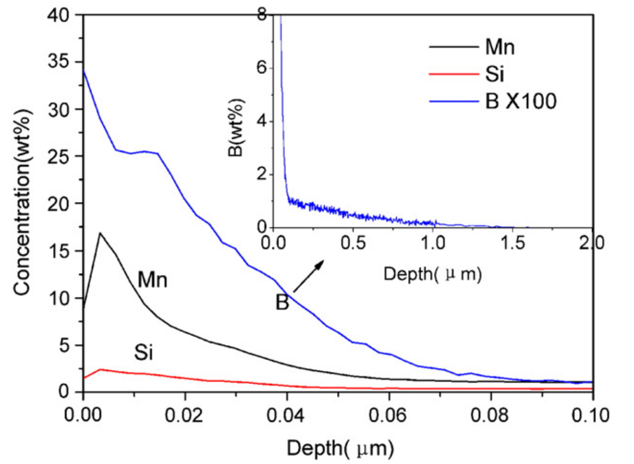


Fig. 2. GDOES concentration profiles of Mn and Si enrichment before dipping, for bright annealing at 800 °C/60 s in N_2-5%H_2 and DP −30 °C.

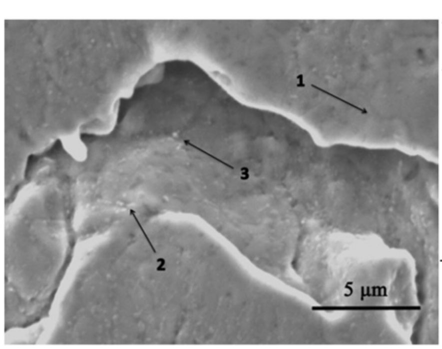
3. Results

3.1. Characterization of oxidized steel surface structure

The type and contents of oxides on steel surface after annealing were found to have a strong effect on interface layer [15]. To better understand the influence of entry-temperature on the interface layer at different bath Al level, it is necessary to examine the initial surface morphology of the annealed PH steel prior to galvanizing. As shown in Fig. 1, there are many cracks on the steel surface, which might be generated during the rolling process. It is found that only a few insular oxide particles are on the steel surface. EDS analysis indicated that these particles are rich in Mn, Si and O (Fig. 1), possibly being Mn–Si complex oxides [18]. This Mn–Si oxides were found to have a strong effect on interfacial reaction between the steel surface and the Al dissolved in the zinc bath [15,16]. GDOES analysis in Fig. 2 shows that Mn, Si and B segregated to the external steel surface during annealing. By measuring the depth with the Mn and Si concentration reaching to a half of their maximum value [16], the Mn–Si complex oxide thickness is assessed to be about 20 nm. In addition, it was observed that the depth of B enrichment at the surface was significantly greater than Mn, Si and is indicative of high-diffusivity in steel substrate.

3.2. Microstructure of interfacial layer

Fig. 3 shows the microstructure of the interfacial layer after galvanizing in 0.12 wt.% Al baths at different entry temperatures. Columnar Fe–Zn intermetallics are found on three samples, and this Fe–Zn intermetallics is identified as ζ-phase by EDS analysis



Element	Spec1	Spect2	Spect3
O	-	6.19	3.74
Si	-	1.63	1.59
Mn	-	5.05	7.31
Fe	100.00	87.13	87.36

Fig. 1. SEM image of $22MnB_5$ steel surface after annealing at 800 °C/60 s in N_2-5%H_2 and DP −30 °C. with EDS point analysis.