



# Formation and phase transformation of aluminide coating prepared by low-temperature aluminizing process



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## ABSTRACT

A low-temperature aluminizing process involving a surface slurry pre-coating and powder embedded aluminizing process, with  $\text{AlCl}_3$  as the chosen activator, was developed to avoid changes in the mechanical properties of the substrate at high temperatures. An electrolytic polishing process using an acidic electroplating solution was further adopted to improve the average surface roughness of the aluminide coating and the smoothing mechanism was explained. Heat treatment promotes the diffusion of iron into the coating and aluminum into the substrate, the phase structure of the coating changed simultaneously. After a 30 h heat treatment at 700 °C, the brittle phases of  $\text{Fe}_2\text{Al}_5$  and  $\text{FeAl}_3$  disappeared and the aluminide coating completely transformed into the  $\text{Fe}_3\text{Al}$  and  $\text{FeAl}$  phases no cracks were observed inside the coating. Through the above processes, a dense and ductile aluminide coating was obtained with a thickness of about 60  $\mu\text{m}$ .

## 1. Introduction

Aluminide coatings are considered to be ideal hydrogen barrier materials because of a high penetration reduction factor, low thermal mismatch, excellent metallurgical bonding, and self-healing ability [1–3]. Commonly used aluminide layer preparation methods are physical vapor deposition (PVD), chemical vapor deposition, metal organic chemical vapor deposition, hot-dipping aluminizing (HDA), electrochemical deposition (ECD), powder embedded aluminizing (PEA), plasma spraying (PS), etc. [4]. PVD is not suitable for the hydrogen barrier container with its complex shape; the coating is not uniform and easy to peel off. The coating prepared by HDA is prone to voids because of the existence of the Kirkendall effect, resulting in poor coating quality [5]. ECD is suitable for complex geometric shaped components; the coating performance is stable, however, only after high temperature heat treatment for a certain amount of time, will an iron-aluminum intermetallic layer be obtained [6,7]. Compared with other methods, PEA is a good choice because of its simple equipment, high platability and suitability for complex components.

Much research has been performed on the powder embedded aluminizing technique. Wu et al. [8] adopted traditional solid powder embedded aluminizing. The mixture was incubated at 900 °C for 2 h, followed by heat treatment at 1050 °C for 5 h. The surface of the coating was inhomogeneous with high porosity. Yang et al. [9] achieved high quality aluminized coatings by a pack cementation vapor

process at 770 °C for 12 h. The iron-aluminum layer was dense, homogeneous and had a thickness of about 20  $\mu\text{m}$  with good surface roughness. Considering that a high embedded aluminizing temperature has a potential impact on the mechanical properties of the substrate, low-temperature aluminizing has become a good choice [10–15]. Bates et al. [12] substituted pure Al powder with binary Cr-Al masteralloys to reduce the activity of Al in the embedded aluminizing process and avoid the formation of the  $\text{Fe}_2\text{Al}_5$  phase. An  $\text{FeAl}$  layer, with a thickness of 12  $\mu\text{m}$ , was obtained at 700 °C for 12 h. Guo et al. [14] combined a surface mechanical attrition treatment with low-temperature duplex aluminizing process to obtain an aluminide surface layer on ferritic-martensitic steel P92.

In the aluminizing process, the aluminized coating usually contains  $\text{Fe}_3\text{Al}$ ,  $\text{FeAl}$ ,  $\text{Fe}_2\text{Al}_5$ ,  $\text{FeAl}_3$  and other intermetallic compounds, however, these compounds do not necessarily appear at the same time. Iron-aluminum intermetallic compounds exhibit different mechanical properties owing to their different stoichiometry. In general, aluminum-rich intermetallic compounds near the outer layer, such as  $\text{Fe}_2\text{Al}_5$  and  $\text{FeAl}_3$ , exhibit low toughness, and are not the phases required for this layer. However, the  $\text{Fe}_3\text{Al}$  and  $\text{FeAl}$  phases near the inner layer of the substrate exhibit excellent properties. The formation of different types of  $\text{Fe}_x\text{Al}_y$  is affected by the composition of the aluminizing agent, aluminizing temperature and time [16]. However, most studies have focused on the effect of heat treatment on the change in phase structure in the aluminized layer [17–21,22,23]. S. Kobayashi et al. [21] studied the

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phase transition of an aluminide coating on low carbon steels at different heat treatment temperatures and durations. The results showed that the  $\text{Fe}_4\text{Al}_{13}$  and  $\text{Fe}_2\text{Al}_5$  phases changed to FeAl and  $\text{Fe}_3\text{Al}$  phases with an increase in time. Mild steel was coated by hot-dipping in a molten bath containing Al-10wt%Si by Wei-Jen Cheng et al. [22]. They determined that the aluminide coating consisted of an external Al-Si eutectic top layer and an internal Fe-Al-Si and Fe-Al intermetallic layer. The  $\tau_5\text{-Al}_7\text{Fe}_2\text{Si}$  and  $\tau_6\text{-Al}_4\text{FeSi}$  phases began to form at 750 °C as the heat treatment duration increased. After 60 min, the FeAl phase began to precipitate. Finally, the aluminide coating completely transformed to the FeAl<sub>2</sub> and FeAl phases. Windmann et al. [23] prepared an aluminum-based coating on 22MnB5 steel using the hot-dipping process. They studied the influence of austenitizing temperature and dwell time on the phase transition. The results showed that higher temperatures and longer dwell time supported the formation of a ductile FeAl phase.

The average surface roughness of coating has great influence on its quality. Purushothaman et al. [24] studied the effect of different mechanical surface modifying processes on the surface roughness and quality of the coating by hot-dipping aluminization. The coating with substrates modified by surface milling and shot blasting was smoother, uniform, and had small porosity. Zhan et al. [25] studied the change of surface roughness during the preparation of coating by a pack cementation aluminizing process. The average surface roughness of the substrate was 25 nm, then increased to 200 nm after aluminization and finally in the range of 199–257 nm after oxidation.

In the traditional powder embedded aluminizing process,  $\text{NH}_4\text{Cl}$  is chosen as the activator [26,27] although  $\text{AlCl}_3$  produced in the reaction is favorable for the production of activated aluminum according to the studies [28,29]. In this work, a new combined process including surface slurry pre-coating at room temperature [30–33] and low-temperature powder embedded aluminizing is proposed. The slurry pre-coating process is intended to increase the concentration of aluminum atoms on the substrate surface, which contributes to reducing the temperature required for aluminizing. The aluminizing temperature in this process was 680 °C, much lower than the traditional powder embedded aluminizing temperature which is in the range of 900–1150 °C [8,34], but similar to the HDA temperature which is generally 700–750 °C [5,22,35–37]. This process innovatively combines dipping aluminizing at room temperature with powder embedded aluminizing, which not only circumvents the voids that are easily produced in hot-dipping aluminizing, but also reduces the temperature of embedded aluminizing. An iron-aluminum alloy layer with a certain thickness was obtained on the 316L substrate using this process. Electrolytic polishing and heat treatment were further used to obtain the aluminide coating with the desired surface roughness and phase structure.

## 2. Materials and methods

### 2.1. Surface slurry pre-coating

The substrate in our current experiments was selected to be 316L stainless steel, which was cut to disk of  $\phi 25 \times 0.8$  mm, ground to 800# sandpaper, ultrasonically cleaned in the acetone and dried rapidly. We dissolved 2.7 g binder polyvinyl butyral (AR) in 55 g solvent ethanol (AR) (1: 20, mass ratio), in which 10 g metal powder mixture was added to prepare pre-coating slurry. The metal powder mixture was composed of 86 wt% Al(99%, < 220 mesh), 5 wt%  $\text{Ce}_2\text{O}_3$  (99.99%, < 220 mesh) and 9 wt% Cr(99%, 200–220 mesh). In the mixture, Al powder provided aluminum source;  $\text{Ce}_2\text{O}_3$  acted as infiltration-aids; a small amount of Cr was added in order to reduce the aluminum activity in the slurry. The substrate was dipped into the slurry, then pulled out at a speed of 140 mm/min and dried in the air. The above dipping and drying process was repeated three times. Finally, the sample was placed in a dry oven at 100 °C for 1 h.

### 2.2. Low-temperature aluminizing

The samples were aluminized by the powder embedded aluminizing technique after surface slurry pre-coating. The composition of the powder mixture was 30% Al, 65%  $\text{Al}_2\text{O}_3$  and 5%  $\text{AlCl}_3$ , in which the aluminum powder was reduced to about 35  $\mu\text{m}$  after ball milling. The powder was packed in 316L stainless steel cans; the samples were placed in parallel into the powder mixture and separated from each other by 10 mm. Then the cans was filled, pressed, and sealed. Finally, the cans was kept at 680 °C for 2 h and cooled in the furnace. The aluminized sample was ultrasonically cleaned for 1 min to remove the surface residue and then dried.

### 2.3. Electrolytic polishing

The thickness and the average surface roughness of the aluminide coating were about 50  $\mu\text{m}$  and 4  $\mu\text{m}$  respectively. Electrolytic polishing was further used to improve the surface roughness of the aluminide coating. The electrolyte consisted of 35%  $\text{H}_3\text{PO}_4$  (volume fraction), 35%  $\text{H}_2\text{SO}_4$  and 30% deionized water. Electrolytic polishing was conducted at 20  $\text{A}\cdot\text{dm}^{-2}$ , 10 V, and 70 °C for 40 s. The samples were washed with water and alcohol immediately after electrolytic polishing to remove the residual polishing products and then dried.

### 2.4. Heat treatment

The heat treatment temperature was 700 °C in case of changes in substrate performance at high temperatures. The samples after electrolytic polishing maintained at 700 °C for 30 min, 1 h, 4 h, 8 h, 15 h and 30 h respectively, and finally cooled to room temperature in the air.

### 2.5. Characterization

The surface and cross-sectional morphology of the aluminide coating were observed by scanning electron microscopy (SEM, ZEISS EVO 18). The chemical composition at the cross section of the aluminide coating was analyzed by energy dispersive spectroscopy (EDS). The phase compositions of the aluminide coating were verified by X-ray diffraction (XRD, RIGAKU D/MAX-RB) operated at 40 kV and 150 mA. The change in surface roughness of the aluminide coating was studied by a 3D Measuring Laser Microscope (Olympus). The following mentioned values for EDS results in each layer were average values for three spots, and the standard deviation was provided. The surface roughness was also obtained as average values, three parallel samples were provided under each experimental parameter.

## 3. Results and discussion

### 3.1. Characterization of aluminide coating

SEM micrographs of the surface and cross section of the aluminide coating after aluminizing are shown in Fig. 1. Fig. 1 (a) shows the surface morphology, the substrate is covered by fine and uniform grains, while there are some pits on the surface. The appearance of these pits is due to the technique of powder embedded aluminizing. Formation process of the aluminized layer is shown in Fig. 2. Active aluminum atoms first adsorb to the surface of the substrate and dissolve into the steel to form a solid solution. When the aluminum atoms increase to a certain concentration, the iron-aluminum alloy layer begins to form. Aluminum and iron atoms diffuse each other and the aluminized layer gradually thickens. Since the concentration of active aluminum atoms on the surface of the substrate could not be guaranteed to be the same, the degree of diffusion of aluminum atoms into the steel is also different. With the increase in time, some pits form on the surface of the coating. The cross section morphology is shown in Fig. 1 (b). The thickness of the aluminide coating is about 50  $\mu\text{m}$ . Compared with

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