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# Effect of nickel pre-plating on high-temperature oxidation behavior of hot-dipped aluminide mild steel



Wei-Jen Cheng\*, Yi-Jhang Liao, Chaur-Jeng Wang

Department of Mechanical Engineering, National Taiwan University of Science and Technology, Taipei 10607, Taiwan, ROC

## ARTICLE DATA

### Article history:

Received 19 November 2012

Received in revised form

6 March 2013

Accepted 14 May 2013

### Keywords:

Hot-dip aluminizing

Mild steel

Nickel pre-plating

High-temperature oxidation

## ABSTRACT

An aluminide/nickel duplex coating was prepared on mild steel by nickel pre-plating before hot-dip aluminizing. The isothermal and cyclic oxidations of the hot-dipped aluminide mild steel were performed at 750 °C in a static atmosphere. The effect of nickel pre-plating on the high-temperature oxidation behavior of the aluminide mild steel was studied. The results from the aluminide steel after isothermal oxidation show that the phase transformation in the aluminide layer was dominated by Ni/Al interdiffusion, leading to the aluminide layer being composed of Ni–Al intermetallic phases. Meanwhile, the aluminide layer was void-free. The result from the aluminide steel after cyclic oxidation is similar to that after isothermal oxidation. The aluminide layer has excellent thermal shock resistance, which did not form any cracks during cyclic oxidation. The oxidation kinetic result from the aluminide steel, after high-temperature oxidation, is consistent with the microstructure and phase constitution of the aluminide layer. The low oxidation rate of the aluminide steel proves that nickel pre-plating before hot-dipping can improve the oxidation resistance of the hot-dipped aluminide steel, especially under condition of cyclic oxidation.

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## 1. Introduction

Alloys for high-temperature environments are required to have good mechanical properties and oxidation resistance at their operating temperatures. Stainless steels and superalloys are the commonly used high-temperature alloys [1]. However, the high cost of these alloys is always a consideration when contemplating using them for high-temperature applications. Carbon steels, on the other hand, have poor resistance to high-temperature oxidation. They are usually used in mild atmosphere environments as structural materials due to their low prices. Aluminizing is a surface treatment that can efficiently improve the high-temperature oxidation resistance of carbon steel by forming a protective aluminide layer over the surface of the steel [2]. Several aluminizing methods have been developed,

such as thermal spray [3], pack cementation [4] and hot-dipping [5,6]. Among all these techniques, low cost and high efficiency make hot-dip aluminizing widely adopted in industry as a conventional aluminizing method.

Hot-dipped aluminide steels have been comprehensively studied [7–9]. The aluminide layer formed on carbon steel after hot-dip aluminizing can be distinguished into an outer aluminum topcoat and an inner Fe–Al intermetallic layer. Also, the Fe–Al intermetallic layer is composed of mainly Fe<sub>2</sub>Al<sub>5</sub> phase and possesses a rough interface with the carbon steel substrate [5]. The reason that the Fe<sub>2</sub>Al<sub>5</sub> phase can become the major phase in the Fe–Al intermetallic layer is because of the vacancies on the c-axis of the orthorhombic crystal structure of Fe<sub>2</sub>Al<sub>5</sub>. These vacancies in Fe<sub>2</sub>Al<sub>5</sub> are seen as rapid migration tunnels, which help in accelerating the rate of Fe/Al interdiffusion in Fe<sub>2</sub>Al<sub>5</sub>. Once the Fe/Al interdiffusion in Fe<sub>2</sub>Al<sub>5</sub> is increased,

\* Corresponding author at: No. 43, Sec. 4, Keelung Rd., Taipei 10607, Taiwan, ROC. Tel.: +886 2 2733 3141x7309; fax: +886 2 2737 6460.  
E-mail address: [d9603505@mail.ntust.edu.tw](mailto:d9603505@mail.ntust.edu.tw) (W.-J. Cheng).

Fe<sub>2</sub>Al<sub>5</sub> phase will be able to grow faster than the rest of the Fe–Al intermetallic phases by fixing its c-axis along the Fe/Al interdiffusion direction [7,8]. However, the thick brittle Fe–Al intermetallic layer and the rough intermetallic layer/steel substrate interface caused by the fast growth of Fe<sub>2</sub>Al<sub>5</sub> are considered as harmful characteristics, which will degrade the mechanical properties and oxidation resistance ability of the hot-dipped aluminide steel [9]. The studies [10–12] concerning the oxidation behavior of hot-dipped aluminide steel have revealed that the formation of dense and continuous alumina scale on the aluminide layer during high-temperature oxidation is the reason that hot-dip aluminizing can dramatically improve the oxidation resistance of the bare steel. Because the growth of alumina scale is dominated by the interdiffusion between oxygen and aluminum, the oxidation behavior of the hot-dipped aluminide steel generally follows a parabolic law. However, the aluminum concentration in the aluminide layer will decrease due to the interdiffusion between the aluminide layer and the steel substrate when hot-dipped aluminide steel is used at high temperatures. Once the aluminum concentration in the aluminide layer is lowered to less than ~15 at.%, the aluminum cannot sustain the formation of a continuous alumina scale. Then iron oxide will start to form over the alumina scale which will result in the failure of the aluminide layer [13]. According to the studies of aluminide/nickel duplex coating on steel prepared by nickel pre-plating followed by pack cementation [14–16], the nickel layer between the aluminide layer and the steel substrate has shown an advantage in retarding the interdiffusion between aluminide layer and steel, which implies that the lifetime of the aluminide steel under high-temperature conditions would be extended. The mechanical properties of this aluminide steel might also be enhanced because the rough Fe–Al intermetallic layer/steel substrate interface is replaced by a flat Ni–Al intermetallic layer/nickel layer.

In our previous study [17] focusing on the microstructure and phase constitution of aluminide mild steel fabricated by nickel pre-plating followed by hot-dip aluminizing, it was observed that the coating possesses an aluminide/nickel duplex microstructure. The aluminide layer formed above the nickel layer is composed of an outer aluminum topcoat and an inner Ni–Al intermetallic layer. The Ni–Al intermetallic layer has a thickness of 5 μm and a flat interface with the nickel layer. In comparison to the hot-dipped aluminide mild steel without nickel pre-plating, it can be predicted that the hot-dipped aluminide steel with aluminide/nickel duplex microstructure might have the potential to better resist high-temperature oxidation. Thus, high-temperature oxidation of the hot-dipped aluminide mild steel with aluminide/nickel duplex microstructure was performed in this study in order to investigate the effect of the nickel pre-plating on the oxidation behavior of the hot-dipped aluminide steel.

## 2. Experimental

### 2.1. Nickel Plating of Mild Steel

A commercial AISI 1005 steel was used as the substrate material in this study. The nominal chemical composition of the adopted steel is Fe–0.05C–0.24Mn–0.01P–0.01S (wt.%). Specimens were

cut to the dimensions of 20 mm × 10 mm × 2 mm and had a hole drilled, 1 mm in diameter, for hanging by stainless wire. Specimens were then ground sequentially with #100 to #800 SiC papers. Before the nickel plating, specimens were cleaned ultrasonically in alkaline, acid and acetone solutions. Nickel plating of mild steel was performed by electrodeposition. Details of the nickel plating process have been described in our previous study [17]. The thickness of the nickel layer on specimens was controlled to 30 ± 3 μm.

### 2.2. Hot-dip Aluminizing

Specimens pre-plated with nickel were aluminized by hot-dipping (hereafter termed Ni-HDA). Mild steel specimens without nickel pre-plating were also aluminized for comparison (hereafter termed HDA). Details of the hot-dip aluminizing of specimens have been described in our previous study [18]. Specimens for hot-dipping were immersed in a molten pure aluminum bath at 700 °C for 15 s. The weight gains of mild steel specimens, with and without nickel pre-plating, after hot-dipping are 3.68 ± 0.53 and 8.81 ± 0.72 mg/cm<sup>2</sup>, respectively. This shows that the aluminum content in HDA is ~2.5 times the aluminum content in Ni-HDA.

### 2.3. High-temperature Oxidation

Isothermal and cyclic oxidations were both performed to study the oxidation behavior of the hot-dipped aluminide specimens. Isothermal oxidation was conducted at 750 °C in a static air for 24–600 h. After isothermal oxidation, specimens were cooled down to room temperature in air. The weight changes of specimens after isothermal oxidation were measured by electronic balance (0.1 mg accuracy), and the value of weight change per unit area was averaged from eight specimens under each experimental condition. Cyclic oxidation was conducted at 750 °C in a static air for 20–720 cycles. Each cycle consisted of 20 min at 750 °C and 60 min at room temperature, which was long enough to cool the specimens below 30 °C. The weight changes of specimens after cyclic oxidation were also measured by electronic balance (0.1 mg accuracy).

### 2.4. Analysis Method

Specimens after isothermal and cyclic oxidations were prepared for cross-sectional metallographic examination using standard grinding and polishing. Cross-sectional specimens were observed using field emission gun-scanning electron microscopy (FEG-SEM) in backscattered electron (BSE) mode. This revealed the atomic number contrast images and distinguished phases by their chemical composition differences. The chemical composition analysis of the phases observed from SEM/BSE micrographs was performed by energy dispersive spectroscopy (EDS) used in tandem with the FEG-SEM. Specimens after oxidation were also observed from top-view using FEG-SEM in secondary electron (SE) mode to reveal the surface morphologies of the aluminide coatings. The crystallographic structures of the phases presented in the aluminide coatings were analyzed by electron backscatter diffraction (EBSD) and X-ray diffraction (XRD) using monochromatic Cu-Kα radiation at 40 kV and 100 mA. The crystallographic structures of the surfaces of the

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