



Characterization of ferritic ductile iron subjected to successive aluminizing and austempering

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ABSTRACT

In this study, GJS-500-14 solution-strengthened ferritic ductile iron was subjected to successive hot-dip aluminizing (HDA) and austempering. The combination of these processes resulted in the formation of a thin Al_2O_3 layer on the iron surface, an intermetallic layer beneath the surface, and an ausferritic microstructure in the interior. Scanning electron microscopy-energy dispersive X-ray spectrometry and X-ray diffraction analyses allowed the qualitative identification of the intermetallics formed due to the aluminizing and their transformation after austempering. Corrosion properties of the final product were also evaluated by potentiodynamic polarization and salt spray corrosion tests, and the surfaces and subsurfaces of the coating layer were examined. The results revealed that this combined process improved the corrosion resistance of ductile iron and enhanced its mechanical properties.

1. Introduction

Solution-strengthened ferritic ductile iron (SSF) is an engineering material that has attracted considerable attention recently due to its low production cost, high ductility, and good castability and machinability compared to conventional ferritic-pearlitic grades [1–3]. In this iron, the ferritic matrix is solution-strengthened by silicon contents ranging from 3 to 4.3 wt% instead of pearlite. This provides a higher yield strength and higher ductility with the same tensile strength [4]. Silicon is a graphitizer, and it promotes the formation of a fully ferritic microstructure in ductile iron. To extend the demand and service life of ductile iron components, austempering heat treatment is applied to conventional ductile iron to convert the initial as-cast microstructure into a unique combination of acicular ferrite (α) and retained austenite (γ). Austempered ductile iron (ADI) is gaining popularity because of its improved mechanical and tribological properties and unique microstructure [5,6]. Despite the good mechanical and tribological properties of ADI, its poor corrosion resistance strictly restricts its use in humid and marine environments [7,8].

The corrosion properties of ADI can be improved by surface modification methods such as physical vapor deposition [9] and electroless nickel plating [10]. On the other hand, hot-dip aluminizing (HDA) is a promising alternative to these methods as it requires short time and can be applied to a metallic substrate without the need for sophisticated equipment.

HDA is generally performed by dipping the substrates into molten Al

[11] and Al–Si alloys [12], and results in the formation of various Fe–Al intermetallics along the thickness of the coating. Immersing the substrate into pure Al produces Fe_2Al_5 close to the substrate and $FeAl_3$ close to the surface. Also, a high-temperature diffusion annealing is generally applied following HDA. This annealing allows further interdiffusion between the layers of the coatings, thus facilitating the variation in the type and order of the layers.

Previous research has mostly focused on the characterization and transformation kinetics of intermetallics formed during aluminizing and subsequent annealing. Their effects on the oxidation and corrosion resistance of the substrate have also been studied [13–15]. In addition, some studies have explored the additional benefits of aluminate layers to extend aluminizing into various industrial areas. For example, Jiang et al. [16] used aluminizing in combination with galvanizing to modify steel in the fabrication of aluminum/iron bimetallic composites and obtained a more uniform and compact layer at the aluminum-iron interface compared to that obtained without galvanizing and aluminizing. Jamnapara et al. [17] performed plasma and thermal tempering of P91 steel aluminized in Al–7Si at 720 °C. Plasma tempering produced a stable $\alpha-Al_2O_3$ layer on the steel surface, while thermal tempering resulted in the formation of a $\theta-Al_2O_3$ layer. They stated that the $\alpha-Al_2O_3$ layer on the aluminized P91 steel is a promising candidate to protect the surface from attack by flowing Pb–17Li in the thermal blanket module of fusion reactors.

HDA can be directly applied to ductile iron with initial as-cast microstructures [18,19]. However, when HDA is applied to an

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austempered sample, the ausferritic microstructure, which is produced by austenitizing at 840–975 °C followed by austempering at 250–400 °C, may deteriorate. As a result, the good mechanical properties of ADI are lost. Therefore, we propose that austempering should be performed after HDA to avoid any loss in the mechanical properties. Therefore, this sequence was followed in the present study to improve the corrosion resistance of ADI by maintaining its ausferritic microstructure. Another major finding was that this process protected the iron surface from decarburization and scaling without the need for a protective atmosphere during austenitizing.

2. Experimental procedure

2.1. Sample preparation

Disk-shaped SSF GJS-500-14 samples with a diameter of 15 mm and thickness of 5 mm were cut from cast round cylindrical bars. The chemical composition of the GJS-500-14 ductile iron samples was as follows (in wt%): 3.3 C, 3.7 Si, 0.2 Mn, 0.029 P, 0.007 S, 0.05 Mg, and balance Fe. Prior to HDA, the samples were ground using #800 grit SiC abrasive papers, ultrasonically washed with acetone, and completely dried in air at room temperature, and then treated with a phosphoric acid solution to destroy the oxide layer on the surface.

2.2. Hot-dip aluminizing

Similar to the method presented in Ref. [20], aluminum (99.9 wt%) was melted in a graphite crucible placed in a resistance furnace, and the aluminizing was performed at 750 °C for 5 min to form a thick aluminate coating on the substrate. The temperature was monitored using a Ni–NiCr thermocouple that was placed directly in the molten aluminum to ensure that the desired temperatures were maintained. The aluminum bath was covered with a flux material to prevent the oxidation of the surface before and after the hot-dipping process. The samples fixed by a stainless steel wire were immersed into the molten aluminum and then pulled out from the molten bath. They were finally air cooled to room temperature, and no shedding of the coating layer was observed after cooling.

2.3. Austempering

The aluminized samples were heated to an austenitizing temperature of 975 °C at a heating rate of < 12 °C/s and held at this temperature for 2 h. The austenitized samples were rapidly transferred to a salt bath containing Petrofer® AS135 commercial annealing salt and austempered at 270 °C, 330 °C, and 390 °C for 1 h. In the last step of the heat treatment, the samples were removed from the bath and cooled to room temperature in air. The temperature was measured and controlled by a Ni–NiCr thermocouple during the austenitizing and austempering steps. The samples were represented as ADI-270, ADI-330, and ADI-390 depending on their austempering temperatures.

2.4. Structural characterization

Qualitative phase analysis of the coatings was performed by X-ray diffraction (XRD, GBC MMA-027) using Cu-K α ($\lambda = 0.154$ nm) radiation. The accelerating voltage and applied current were 35 kV and 28.5 mA, respectively. The samples were scanned over a 2θ range of 20°–90° in steps of 0.02° at a scanning speed of 1° min⁻¹. The obtained XRD patterns were then analyzed by software (X'pert HighScore Plus) to identify the peak positions. A scanning electron microscopy (SEM) system (JEOL JSM 6335 F FEG) equipped with an energy dispersive spectrometry (EDS) system (JEOL NeoScope JCM-6000) was used to examine the surface morphology of the coatings and elemental distribution of the phases. The cross-sectional morphology of the coatings and the microstructures of the samples were examined by field-

emission SEM (FE-SEM, Zeiss Ultra Plus) after etching with 4 vol% nitric solution. The elemental analysis of the coatings was performed using an energy dispersive X-ray spectroscopy (EDX) system (Bruker XFlash 5010) integrated into the SEM system. Quantitative phase analysis of the cross-section of the samples was conducted using XRD with Cu-K α radiation over a 2θ range of 60°–105° in steps of 0.05° and at a scanning speed of 1° min⁻¹ to estimate the volume fraction of ferrite (X_{α}) and austenite (X_{γ}) utilizing the integrated intensities of the (200) and (220) planes of body-centered cubic (bcc) ferrite and the (220) and (311) planes of face-centered cubic (fcc) austenite according to the direct comparison method [21].

2.5. Hardness and tensile tests

Hardness measurements were performed at the cross sections of the coatings and the substrates by a Zwick universal hardness tester (Roell ZHU 2.5) with a diamond Vickers indenter. During indentation, the maximum test load was 100 g with a dwell time of 20 s. At least 5 measurements were taken for each sample, and the results were averaged.

Duplicate tensile tests of the samples were performed with a gage length of 25 mm using a universal servo-hydraulic test machine (Instron 8801) according to the ASTM E8/E8M standard, and the average results were reported. All tests were conducted at a cross-head speed of 2 mm min⁻¹ at room temperature.

2.6. Corrosion tests

Anodic potentiodynamic polarization corrosion tests were performed in a 3.5 wt% NaCl solution to simulate an aggressive chloride-containing aqueous environment. The samples were placed in a potentiostat/galvanostat apparatus (EGMA 273), and a standard saturated calomel electrode (SCE) was used as the reference electrode and platinum was used as the counter or auxiliary electrode. The contact area in all cases was 1 cm², and the temperature was maintained at 25 °C during the corrosion tests. The electrode potential was raised from -1.2 V to 0.4 V at a scanning rate of 1 mV/s. All polarization curves were obtained after 20 min of free immersion of the samples to ensure a steady open-circuit potential. The corrosion parameters were evaluated by Tafel extrapolation of the polarization curve.

Salt spray corrosion tests (SSCTs) were performed in a chamber containing a 5 wt% NaCl solution for 48 h in accordance with the ISO 9227 standard. The pH was maintained between 6.5 and 7.2, and the temperature was maintained at 35 ± 2 °C during the corrosion tests. After the SSCTs, the surface morphology of the samples was examined by SEM (Hitachi TM-1000). Following the SSCTs, the corrosion products were completely removed by chemical cleaning according to the ISO 8407 standard. The weight loss due to corrosion was measured using an analytical balance with a precision of 0.1 mg. Cross-sectional SEM images of the samples were also obtained after the chemical cleaning.

3. Results and discussion

3.1. Formation of intermetallic layers between solid iron and molten aluminum

Fig. 1 shows (a) the surface XRD patterns and (b) cross-sectional SEM micrographs of the hot-dip aluminized sample before austempering. From the XRD analysis, it was found that the coating was composed of Al (JCPDS No. 085-1327), FeAl₃ (JCPDS No. 050-0797), and Fe₂Al₅ (JCPDS No. 029-0043) phases. These phases were also identified by EDX analysis shown in the inset of Fig. 1(b). The phases could be seen in the cross-sectional SEM images, with a top layer of Al (with FeAl₃), middle layer of FeAl₃, and inner layer of Fe₂Al₅. The tongue-like morphology of Fe₂Al₅ was due to its preferential growth on

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