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Low-Temperature Slurry Aluminizing: Investigating the Influence of Aluminizing Time on the Corrosion Performance of Aluminide Coating on 304 SS

Ambali Ibrahim Owolabi^{a,b}, Anasyida Abu Seman^{a*}, and Tuti Katrina Abdullah^a

^a*School of Materials and Mineral Resources Engineering, Universiti Sains Malaysia, 14300 Nibong Tebal, Pulau Pinang, Malaysia*

^b*Department of Materials & Metallurgical Engineering, University of Ilorin, Ilorin, Nigeria*

Abstract

The surface of austenitic stainless steel (304 SS) was modified with aluminium and alumina powders using a slurry aluminizing route to enhance its lifespan at high temperatures. The substrate was subjected to low heat treatment temperature (680°C) at various aluminizing times (4, 6, 8, and 10 hours). The corrosion resistance of the aluminide coating was evaluated by exposing them to a mixture of molten solar salt containing 60 wt.% NaNO₃ and 40 wt.% KNO₃ at 600°C for 100 hours. The coatings were characterized using FESEM, EDX, and XRD. Coating thickness, hardness, multi-layered phases, and corrosion products were determined before and after corrosion. The results indicated that a dense and continuous inner layer made up of FeAl-based intermetallic improved the corrosion resistance of 304SS. Coating thickness increased with increasing aluminizing time, with a maximum thickness of 75.12 µm observed for samples with 10 hours of aluminizing. The highest coating hardness of 1060 HV was observed on Fe₂Al₃ of aluminide layer heat treated at 10 hours. The corrosion product found on the aluminide layer was NaAlO₂ and the sample heat treated for 6 hours exhibited the lowest corrosion rate of 0.21 mm/year.

Keywords: *Slurry aluminizing; corrosion rate; FeAl-based intermetallic; molten nitrate salt; high-temperature application*

1. Introduction

Austenitic stainless steels are preferred for high temperature applications such as exhaust engines and power generation due to their high chromium and nickel content, availability, low cost, and similar properties to other high-grade steels that provide high oxidation and corrosion resistance in harsh environments[1]. Austenitic stainless steel is also selected as the storage media for storing molten solar salt in concentrated solar power plant. The molten solar salt is composed of 40%KNO₃ and 60% NaNO₃ which deliver thermal energy to heat exchangers for power generation [2]. Zarie *et al*[3] reported that the high-temperature oxidation of stainless steel is due to the ability of these materials to form a dense oxygen diffusion barrier, in the form of a Cr₂O₃ layer on their surface. However, the degradation of this oxide layer during exposure to high temperatures can lead to spallation and breakdown. The insufficient protective properties of oxide films result in poor resistance to oxidation, particularly in molten solar salt. To improve the corrosion resistance of stainless steel and optimize its lifespan for high-temperature applications, there is need for surface modification with aluminium which produces more stable oxides than chromium. Aluminide coating is an alternative method of producing aluminium-containing layers on the surface of substrates, improving the oxidation resistance by generating a fine and dense Al₂O₃ scale on the surface of the steel for high temperature application. Aluminide coating can be produced through the following techniques such as pack cementation, hot dip aluminizing (HDA), chemical vapor deposition (CVD), physical vapor deposition (PVD), and slurry aluminizing. Slurry route aluminizing has gained popularity and commercial viability among all other techniques due to its simplicity, flexibility, low cost, reparability, non-hazardous and a preferred choice for mass production of coating large components[4–6]. However, the relationship between different aluminizing time for the formation of intermetallic compounds and the corrosion performance of aluminide coatings on austenitic stainless steel remains intricate and requires thorough investigation. This study focuses on slurry aluminide coatings on 304SS exposed to the molten nitrate corrosion (60% NaNO₃ and 40%KNO₃) at 600°C for 100 h.

2. Methodology

Stainless steel (304SS) with dimensions of 10 mm x 10 mm x 2 mm was used as the substrate. The substrate was ground with 240 SiC grit size under the running tap water. The samples were ultrasonically cleaned in an acetone solution and dipped in a prepared solution of NaOH and HCl to remove any oxides formed on the substrates. The slurry was prepared by a percentage mixing ratio of 57% of solvent binder system (distilled H₂O and PVA 14000) and



43% of additives powders (aluminium and alumina powder). The prepared slurry was sprayed evenly on the substrates with the aid of Sagola spray gun at an interval of 30 mins for each coating on all the samples and dried at room temperature. The coating procedure was controlled to achieve a mass/area of 15 -30 mg. The samples were allowed to dry for 1 hour before subjected to heat treatment in a carbolite tube furnace in an argon environment at 680°C for 4, 6, 8 and 10 hours under the control heating rate at 10°C/min. After the aluminizing treatments the weight of sample was measured. Then, the corrosion test was conducted according to ISO/NP 17245- 2015 standard protocol (corrosion of metals and alloys test method for high-temperature corrosion testing) by completely immersed in alumina crucible containing mixture of molten salt (60 wt.% NaNO₃ +40wt.% KNO₃) in the furnace at 600°C for 100 hours. Corrosion samples were chemical cleaned in 10% HNO₃ in distilled water (ASTM designation G1), dried and weighed after corrosion test. The mass loss of the samples was measured between the weight of the aluminide samples before and after corrosion and the corrosion rate was calculated using a gravimetric method in equation 1 (ASTM GI 8.1). Aluminide coating samples were prepared for metallurgical examination by grinding and polishing to a mirror like. The samples were characterized for morphology, coating thickness and chemical compositions using field emission scanning electron microscope FESEM (ZEISS SUPRA 35VP) equipped with energy-dispersive X-ray (EDX). Aluminide layer phases were examined via X-ray diffraction (XRD) at diffraction angle (2θ) range was 20°–90°. The hardness of the intermetallic compounds was determined by a Vickers microhardness test using a load of 25 g and dwell time of 15 s.

$$\text{Corrosion rate: } (K \times W)/(A \times T \times D)$$

Equation (1)

K= constant = 8.74 x 10⁴ mm/year for stainless steel

W= mass in gram

A= Area in mm

T= Time of exposure (100 h)

D= Density in g/mm² (7.94 g/mm³) for stainless steel

3. Results and discussion

3.1 SEM Morphology and EDX Analysis of Aluminide Coating

Fig. 1 shows the SEM cross-sectional image of the aluminized samples for all the time. The image revealed the two layer structures for all the heat treatment time namely: i) dark outer layer with rough structure known as diffusion zone and ii) fine and continuous inner layer also known as interdiffusion zone. Coating layers formed was due to the infiltration of active Al atoms from the coating diffusing toward the substrates during the aluminizing. Table 1 shows the EDX analysis of chemical compositions at four (4) different locations on the intermetallic layers to quantify the percentage ration of Fe and Al. It was found that point 3 and 4 consist of Al-rich intermetallic compound with major Fe₂Al₅ phases, that first to form because of the smallest free energy as reported by Dong et al. [7,8] and minor FeAl₃ on the outer layer while point 2 consists of Fe-rich intermetallic compound close to the substrate known as FeAl. In addition to the major elements, there are other trace elements like Ni, Cr and O also detected having no significant effect on the aluminide coating layers. Fig. 2 shows the XRD analysis of the aluminide coating which detected the following phases: Fe₂Al₅ (ICDD PDF number: 00-001-1228), FeAl₃ (ICDD PDF number: 00-069-0296) and FeAl (ICDD PDF number: 04-013-9779) to support the EDX result. Fe₃Al could not be found by the XRD and EDX result, this is contrast to Sun *et al.* [8] where all phases were detected at a longer aluminizing time. The Fe₃Al is the transition between the FeAl layer and the substrate. XRD also detected the alumina phase (ICDD PDF number: 04-005-4500). The Al₂O₃ scale as a result of the oxidation reaction of aluminum in the aluminizing process, providing oxidation resistance to the aluminide coating [9,10]. In Fig. 3, the surface structure of the modified aluminide coating on 304SS demonstrates random distribution of Al₂O₃. Addition of alumina reduced the activities of aluminium and slow down the diffusion of Al to form intermetallic compounds during aluminizing process. No cracks were found on the aluminized coating. This may be due to small mismatch of thermal expansion coefficients between the coating and the substrate which make the intermetallic phases more compatible, dense and continuous.

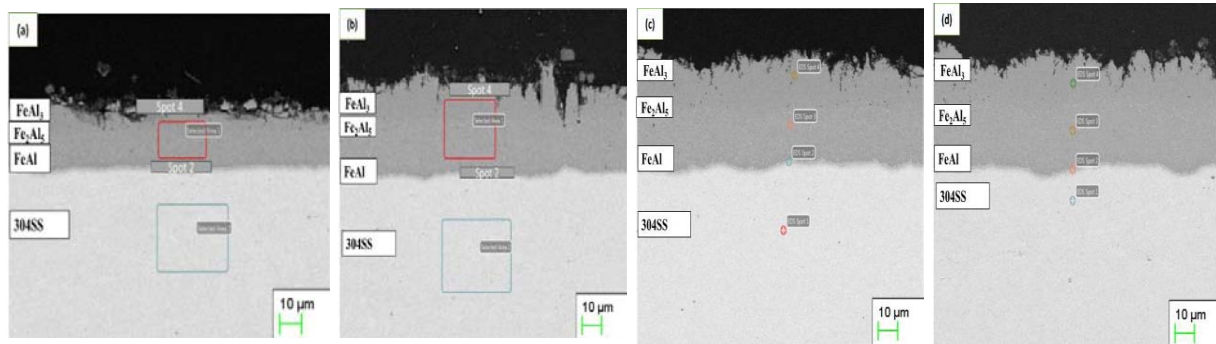


Fig. 1. SEM micrograph of cross section of sample heat treated at 680°C for (a) 4 h, (b) 6 h, (c) 8 h and (d) 10 h

Table1. EDX data analysis on cross section of 304SS after aluminizing at 680 °C for 6 h

Spot/Area	Al	Fe	O	Cr	Ni	Phases
1	1.21	69.49	1.74	20.42	7.15	substrate
2	37.54	40.99	1.3	13.04	7.12	FeAl
3	63.03	25.34	1.19	8.76	1.68	Fe ₂ Al ₅
4	69.97	22.47	0.17	4.38	3.01	FeAl ₃

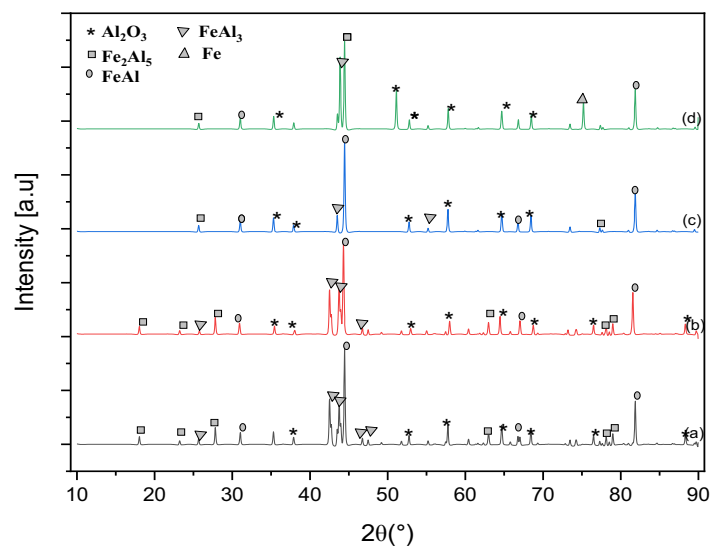


Fig. 2. XRD pattern of aluminized 304SS heat treated at 680°C for (a) 4 h, (b) 6 h, (c) 8h and (d) 10 h

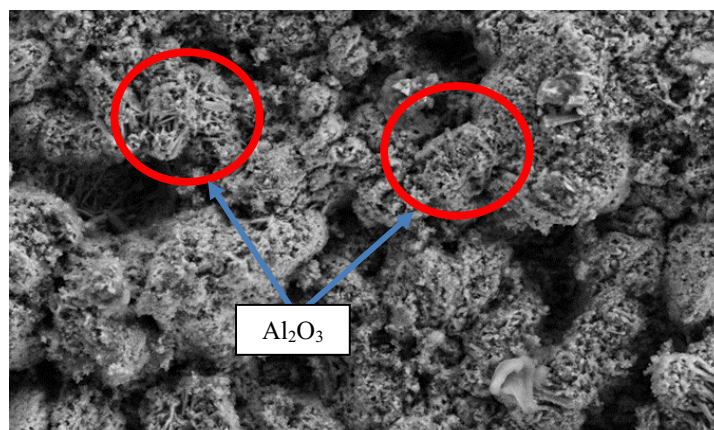


Fig. 3. Surface morphology of aluminide coating heat treated for 680°C at 6h

3.2 Coating thickness

The variation in the coating thickness of the aluminide layers was observed for aluminizing process at 680°C for different aluminizing times. Coating thickness on aluminized steel was measured and recorded using Image J software at five different locations. Fig. 4 shows the trend of coating thickness of the aluminide coating for all the heat treatment time. Sample heat treated at 4 h has the least coating thickness (36.21 μm) while the highest thickness of 75.12 μm was recorded for sample heat treated at 10 hours. Increase in the coating thickness is attributed to the increase in aluminizing time. The increase in coating thickness with aluminizing time also reported by other studies [11–13].

3.3 Hardness

The overall average ranges of the hardness of the aluminide coating layers were assessed with microhardness test for 4, 6, 8 and 10 h aluminizing time. Fig. 5 shows the Vickers microhardness profiles of the aluminide coating modified with alumina. The transition zone between the topcoat and coating was found to be FeAl_3 with hardness value of 800-880 HV, and within the range of the reported value of 850 HV [14]. Underneath zone was Fe_2Al_5 with an average value of 940-1060 HV. The hardness value dropped toward the substrate and hardness of the FeAl was 420-630 HV. The hardness value is high at the Al-rich intermetallic zone due the higher Al concentration while decrease in the Fe-rich intermetallic zone as a result of high Fe concentration close to the substrate. The substrate has the hardness value of 202-221 HV.

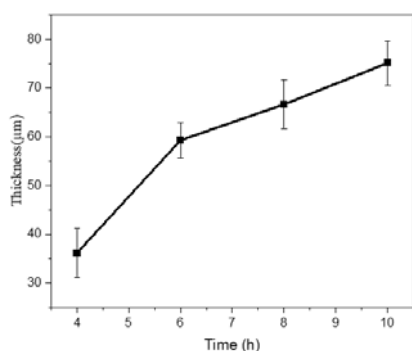


Fig. 4. Aluminide coating thickness

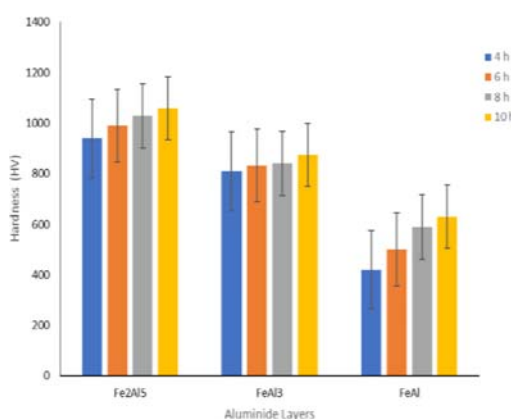


Fig. 5. Hardness value of the aluminide coating

3.4 Corrosion Mechanism of Aluminide Coating in Molten Nitrate Salt

The equation 5 shows the reaction that occurred during the corrosion process in which NaAlO_2 (corrosion product) and it remains on top of the intermetallic phase (Fe_2Al_5). Fe_2Al_5 formed during the aluminizing process served as a protective layer hindered the aggressive reaction of molten salt with substrates. Corrosion rate was calculated using equation 1 with sample heat treated for 6 h having the least weight loss. Fig. 6 shows the corrosion rate of the aluminide coating layer heat treated at different time in a molten nitrate. All the samples showed good corrosion resistance in molten nitrates. But the lowest corrosion rate of 0.21 mm/year was recorded for the sample heat-treated at 6 h while the highest corrosion rate of 3.012 mm/year was recorded at 10 h. The lower the corrosion rate, the better the corrosion resistance of the aluminide coating sample in a molten nitrate salt. Al coated exhibited a higher protection against the molten nitrates due to the formation of intermetallic layer formed on the surface of substrate during the aluminizing process[15–17]. Corrosion mechanisms of aluminized steel in contact with molten nitrates salt at high temperature occurred via the following reactions:

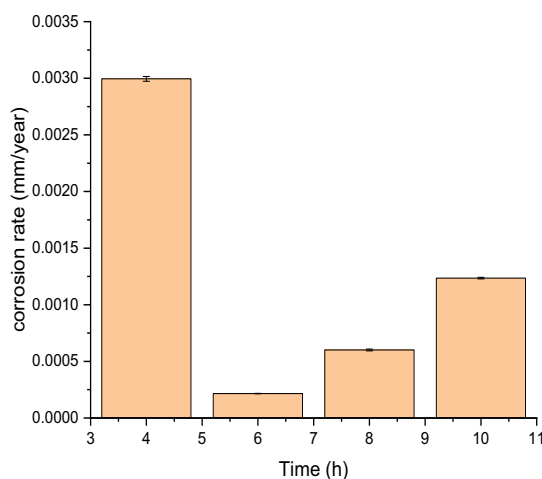
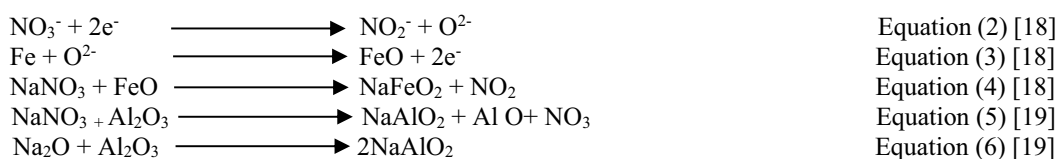


Fig. 6. Corrosion rate of aluminide coating on 304SS heat treated at 680°C for 4, 6, 8 and 10 h after corrosion

4.0 Conclusions

Aluminide coating formed a dense and continuous layer on the 304 SS at all the aluminizing time and increased with aluminizing time. The outer layer consist of (Fe_2Al_5 and FeAl_3 phase) and an inner layer consist of FeAl phase. The highest hardness was attained at Fe_2Al_5 phase. All the samples produced showed better corrosion resistance with the Al coated sample heat treated at 6 hours exhibited the lowest corrosion rate of 0.21 mm/yr.

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