

# Corrosion Resistance of Aluminized Diffusion coating for Fe-25Cr-20Ni steel

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**Abstract:** High-temperature corrosion resistance and characteristics of Fe-25Cr-20Ni steel was studied at 800 °C in air and (N<sub>2</sub>/H<sub>2</sub>O/H<sub>2</sub>S)-mixed gas condition up to 100 hours. The weight gain of each test coupon after the oxidation and (N<sub>2</sub>/H<sub>2</sub>O/H<sub>2</sub>S)-mixed gas corrosion test was measured using a micro-balance. The corroded test coupons were characterized by a SEM, an EDS, an XRD with Cu-K $\alpha$  radiation, and an EPMA. As a result of the experiments, aluminized diffusion coating was formed the FeAl<sub>2</sub> intermetallic phases. Thus aluminized diffusion coated Fe-25Cr-20Ni steel has reasonable corrosion resistance, because it can form a protective alumina scale, such as  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> layer.

**Key words:** Corrosion, aluminized diffusion coating, Fe-25Cr-20Ni steel, oxidation, sulfidation.

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## 1. Introduction

High-temperature corrosion resistance is very important in coal gasification plant facilities. These are essential for understanding of the conditions necessary for the development of dense corrosion layers with both mechanical properties and diffusion barrier effects. Coal gasification plant syngas consisted primarily of N<sub>2</sub>, H<sub>2</sub>, CO, H<sub>2</sub>S, and H<sub>2</sub>O vapors, etc. Among these, the most corrosive gas is H<sub>2</sub>S that dissociates into sulfur and hydrogen, which shorten the lifetime of the material and give a low strength and creep resistance. Therefore, there is a need for a corrosion resistance enhancement measure which can improve this [1]. The aluminizing is one of pack-cementation diffusion coating processes for Fe-base alloys. Coating are generally applied as a layer of a protective material onto a low cost material and elevated temperature applications. Pack-cementation can provide excellent elevated temperature performances through highly protective oxide layer formation. In addition to increase the metallic materials lifetime, fracture toughness and soft or ductile intermetallic phases on metallic materials [2]-[5]. A number of groups have reported the formation of FeAl, Fe<sub>2</sub>Al<sub>5</sub> and Fe<sub>3</sub>Al etc. on Fe-rich alloys, depending on the aluminized diffusion coating condition [6]-[9]. In this study, the aluminized diffusion coating was applied on Fe-Cr-Ni alloy substrate by pack-cementation, and its corrosion behavior was studied at 800 °C in (N<sub>2</sub>/H<sub>2</sub>O/H<sub>2</sub>S)-mixed gas. Aluminized diffusion coating was formed the FeAl<sub>2</sub> intermetallic phases. The FeAl<sub>2</sub> intermetallic phases have reasonable corrosion resistance, because it can form a protective alumina scale, such as  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> layer with and without some Fe<sub>2</sub>O<sub>3</sub>.

## 2. Experimental

Fe-25Cr-20Ni steel plate with a whole composition of Fe-25Cr-20Ni-0.85Si-0.08Mn in wt.% was cut to 2 × 10 × 15mm<sup>3</sup> sized rectangular coupons, ground on 1500 grit SiC papers, and ultrasonically cleaned with

acetone and alcohol before the start of the aluminized diffusion coating. Aluminizing was performed by heating the powder pack that consisted of 8% Al(source metal)+1%NaF(activator)+91%Al<sub>2</sub>O<sub>3</sub>(inert filler) at 1000 °C for 5h in an H<sub>2</sub> atmosphere. After pack-cementation, aluminized samples were separately charged into a quartz reaction tube, heated from room temperature to 800 °C in Ar within 20min, corroded at 800 °C for up to 100h in air and 1atm of (N<sub>2</sub>/3.1%H<sub>2</sub>O/2.42%H<sub>2</sub>S)-mixed gas, and furnace-cooled to room temperature. The N<sub>2</sub> and H<sub>2</sub>S gases were 99.999 and 99.5% pure, respectively. After corrosion, the weight gain of each sample was measured using a microbalance to evaluate the extent of corrosion. The analyzed composition and corrosion mechanism of the samples were characterized by a scanning electron microscopy (SEM), an energy dispersive spectrometry (EDX/EDS), an electron probe microscopic analysis (EPMA-EDS), a high power X-ray diffractometer (XRD) with Cu-K<sub>α</sub> radiation at 40 kV and 300 mA in  $\theta/2\theta$  configuration, and electron backscatter diffraction (EBSD) analysis.

### 3. Experimental Results

Fig. 1 shows SEM/EDS/XRD results of the aluminized Fe-25Cr-20Ni steel. Aluminized diffusion coating formed almost 220  $\mu\text{m}$  thick coated layer. The characteristic of aluminized diffusion coating layer was seen the three different particle layer (Fig. 1a). The 1<sup>st</sup> outer layer was the 60  $\mu\text{m}$ -thick aluminized coating. The middle layer 2<sup>nd</sup> was the 110~120  $\mu\text{m}$ -thick diffusion zone where about 60 %Al was dissolved. The inner layer 3<sup>rd</sup> was 45  $\mu\text{m}$ -thick. The composition of the topcoat was 27Fe-11Cr-10Ni-47Al-5O in at.%. Figure 1(b) shows XRD patterns of aluminized Fe-25Cr-20Ni steel before corrosion. In the case of aluminized Fe-25Cr-20Ni steel, the surface consisted of FeAl, FeAl<sub>2</sub> and  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>. The FeAl<sub>2</sub> coating layer has been consist of high aluminum content, and this intermetallic compounds cause beneficial to the metallurgical bonding of the surface alloying layer with substrate materials [6], [7]. Figure 1(c) shows the EDS concentration spot analysis (at.%). Spots 1-11 corresponded to the top, diffusion coating layer, and the matrix, respectively. The Al-O compositional ratios (at. %) at spots 1-7 were suggesting that mainly  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>, FeAl<sub>2</sub>, FeAl were formed in the alloy layer between the top and the substrate. EDS analysis indicated that very small amount of the aluminum was detected below spot 8-10.

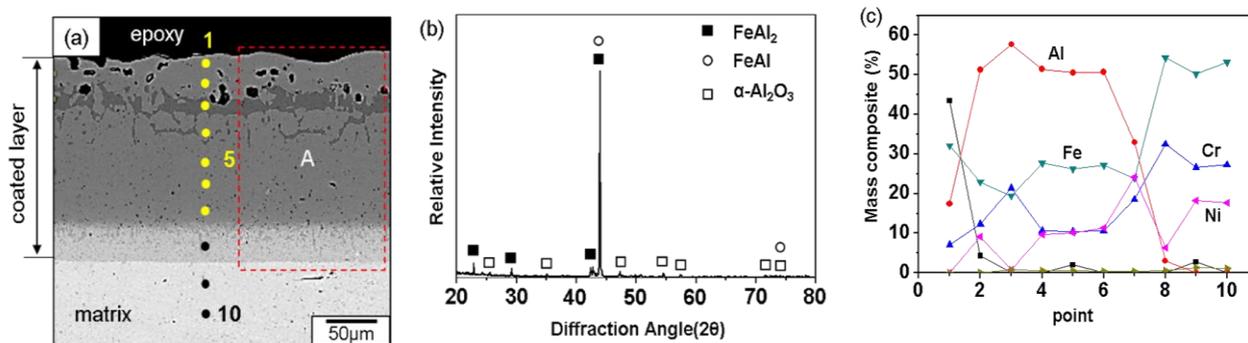


Fig. 1. Aluminized Fe-25Cr-20Ni steel. (a) cross-sectional SEM back-scattered electron image, (b) XRD pattern after aluminizing, (c) concentration profiles of Fe, Cr, Al, and Ni from the coating surface.

Weight gain of aluminized diffusion coating on Fe-2Cr-1Mo, Fe-9Cr-1Mo, Fe-25Cr-20Ni steels that measured after corrosion at 800 °C for 100h in air were appeared in Fig. 2. The increased weight of aluminized diffusion coating on Fe-2Cr-1Mo, Fe-9Cr-1Mo, Fe-25Cr-20Ni steels was parabolically owing to the formation of protective  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> scale. On the other hand when Fe-9Cr-1Mo steel corroded at 600-700 °C during cyclic oxidation in 10%H<sub>2</sub>O+90%Ar atmosphere, they corroded almost linearly, because it forms thick Fe<sub>2</sub>O<sub>3</sub>, (Fe,Cr)<sub>3</sub>O<sub>4</sub> scale [10]. Oxygen in air fast increases oxidation rate of Fe-9Cr-1Mo steel, but aluminized diffusion coating steels formed FeAl<sub>2</sub> phase at the matrix, which was increase the corrosion resistance.

Fig. 3 shows the FE-SEM/EDS/XRD results of the aluminized Fe-25Cr-20Ni steel after corrosion at 800 °C for 50 h in (N<sub>2</sub>/3.1%H<sub>2</sub>O/2.42%H<sub>2</sub>S)-mixed gas. After corrosion cross-section image was similar to those observed after aluminized diffusion coating. The surface was covered with thin  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> scale (Fig. 3b). Corrosion resistance is enhanced by aluminized diffusion coating, so the scale primarily consisted of FeAl<sub>2</sub>, FeAl as the major matrix peak and  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> as the minor one, implying that corrosion was governed by not sulfidation but oxidation (Fig. 3c). The  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> scale interrupted internal diffusion of O and S as a protective layer formed on the outside. Therefore, despite the high temperature, the external diffusion of Fe and Cr did not occur, so that (Fe, Cr) -O and S were not formed (Fig. 3d).

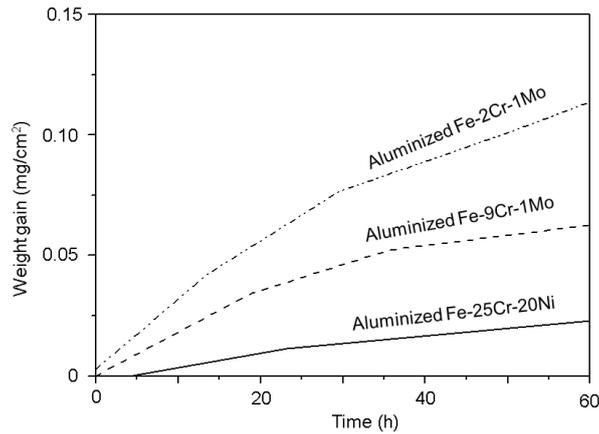


Fig. 2. Weight gain versus oxidation time curve of aluminized diffusion coating on Fe-2Cr-1Mo, Fe-9Cr-1Mo, Fe-25Cr-20Ni steels at 800 °C for 100h in air.

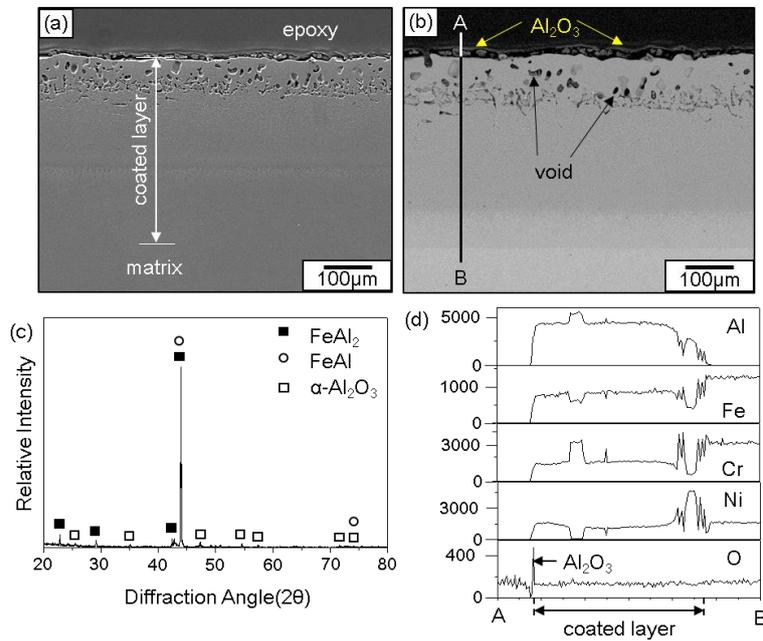


Fig. 3. Aluminized Fe-25Cr-20Ni steel after corrosion at 800 °C for 50 h in N<sub>2</sub>/H<sub>2</sub>O/H<sub>2</sub>S-mixed gas. (a) cross-sectional SEM secondary electron image, (b) SEM back-scattered electron image, (c) XRD pattern after aluminizing, (d) EDS line profiles of Al, Fe, Cr, Ni, and O.

Fig. 4 shows the FE-SEM/EDS results of the aluminized Fe-25Cr-20Ni steel after corrosion after corrosion at 800 °C for 100 h. High components of Al and O appeared in the topcoat layer and the amount of Al gradually decreased as it entered the coating layer, and the amount of Fe, Cr, and Ni increased with the base material.

The thickness of the coating layer decreased to 160~180  $\mu\text{m}$  in Fig. 4(a), owing to the inner diffusion between the aluminized coating layer and the matrix. As the corrosion proceeded, Al in the coating continuously consumed to form  $\alpha\text{-Al}_2\text{O}_3$  at the outer scale. In particular, due to  $\text{FeAl}_2$  an excellent corrosion resistance phase, only a thin  $\alpha\text{-Al}_2\text{O}_3$  corrosion product was formed after corrosion at 800 °C for 100 hours (Fig. 4b).

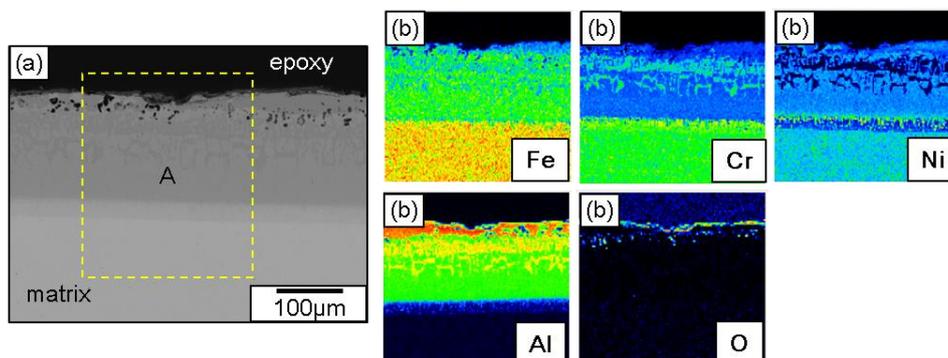


Fig. 4. Aluminized Fe-25Cr-20Ni steel after corrosion at 800 °C for 100 h in  $\text{N}_2/\text{H}_2\text{O}/\text{H}_2\text{S}$ -mixed gas. (a) cross-sectional SEM back-scattered electron image, (b) EDS maps of Al, Fe, Cr, Ni, and O.

#### 4. Conclusion

The aluminized diffusion coating on Fe-25Cr-20Ni steel was made by pack-cementation, and its corrosion behavior was studied by SEM/EDS, XRD, EPMA, and EBSD. When corroded at 800 °C for up to 100 h in air and ( $\text{N}_2/\text{H}_2\text{O}/\text{H}_2\text{S}$ )-mixed gas. The aluminized Fe-25Cr-20Ni steel corroded very slowly, forming scales that consisted primarily of  $\alpha\text{-Al}_2\text{O}_3$ ,  $\text{FeAl}_2\text{O}_4$  and FeO. Aluminized diffusion coating beneficially improved the corrosion resistance and effective surface protection of Fe-25Cr-20Ni steel by forming the  $\alpha\text{-Al}_2\text{O}_3$  scale on the surface.

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**Min Jung Kim** wrote and edited the paper, and contributed in all activities. Dong Bok Lee also substantial contributed to the analysis of the results. Min Jung Kim graduated from Sungkyunkwan University in Korea. Her major is advanced materials science & engineering and study field is metal corrosion mechanism. She published many papers on high-temperature corrosion of Fe-base alloy and coated sample. She is a visiting professor in School of Advanced Materials Science & Engineering, Sungkyunkwan University, Suwon, Korea. She is very enthusiastic to study eco-friendly power generation technology materials and her commitment and passion extends far beyond academic pursuit to solving real environmental issue.