Corrosion of Hot-dip Aluminized 9Cr-1Mo Steel in $N_2/0.1\%H_2S$ Gas

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Abstract: ASTM P91 steel (viz. 9Cr-1Mo steel) was hot-dip aluminized to form a $\sim 70\ \mu m$ thick coating, and was then corroded in $N_2/0.1\%H_2S$ gas for 50 h at 800 and 900 °C, to study the effect of aluminizing on the corrosion resistance of P91 steel in highly corrosive H_2S environments. Before corrosion, the aluminized coating consisted of an Al-rich topcoat, an $Al_{13}Fe_4$ upper layer, and an Al_5Fe_2 lower layer from the surface. During corrosion, Al, oxygen, and sulfur diffused inwardly, while substrate elements diffused outwardly. Impurity oxygen in the gas reacted preferentially with Al to form $\alpha-Al_2O_3$ at the surface, which increased the corrosion resistance significantly. Bare P91 steel corroded quickly, to form bi-layered, fragile, nonprotective FeS scales. The coating transformed into either a ($Al_{13}Fe_4$, Al_5Fe_2)-mixed layer, AlFe layer, $AlFe_3$ layer, and $\alpha-Fe(Al)$ layer when corroded at 800 °C/50 h, or into a $AlFe_3$ layer and $\alpha-Fe(Al)$ layer when corroded at 900 °C/50 h. Interdiffusion that occurred during corrosion increased the total coating thickness, and transformed (high Al)-Fe phases to (low Al)-Fe phases. The corrosion accompanied the formation of voids and microcracks in the coating.

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Keywords: aluminizing, P91 steel, H_2S gas corrosion, hot dipping

1. INTRODUCTION

Ferritic 9Cr-1Mo steels (ASTM P91) are used in heat exchangers, boilers, and pipes because of their high strength and thermal conductivity, low thermal expansion coefficients, and resistance to creep, thermal fatigue, and oxidation. They form outer Fe-rich oxides such as Fe_2O_3 and Fe_3O_4 , and inner Cr-rich oxides such as $FeCr_2O_4$ or $(Fe,Cr)_3O_4$ during high-temperature oxidation in air [1], steam [2], and ultra-supercritical power plant [3]. Although they form protective Cr-rich oxide scales during high-temperature oxidation [4], they become less protective in sulfur-containing environments because sulfides are generally highly nonstoichiometric and have lower melting points than their corresponding oxides [5,6]. Sulfur is usually present in fossil fuels, fluxes, or chemical feedstock. In reducing atmospheres, it exists in the gas stream as $H_2S(g)$, which limits operating temperature and

efficiency during processing in oil refineries, petrochemical units, coal gasification, natural gas reforming, and energy generation in thermal power plants [7-10]. H_2S dissociates into sulfur and hydrogen. Hydrogen atoms dissolve to cause embrittlement, ingress into the scale and metals interstitially, generate lattice point defects and hydrogen clusters, resulting in a significant increase in the corrosion of metals [11].

When corroded in H_2/H_2S gas at 575-650 °C, 9Cr-1Mo steel formed (FeS, $FeCr_2S_4$)-sulfide scales which tended to crack and spall during cooling [12]. In N_2/H_2S gas at 600-700 °C, it corroded fast and almost linearly, forming outer FeS scales and inner (FeS, $FeCr_2O_4$)-mixed scales [13]. Particularly, $Fe_{1-x}S$ grows fast due to its nonstoichiometry ($x=0-0.2$), and makes the scales highly fragile, non-adherent, and nonprotective [14].

This study aimed to investigate the microstructure and corrosion behavior of hot-dip aluminized 9Cr-1Mo steel in a highly corrosive H_2S environment. Aluminizing is widely used on metals in order to protect alloys from oxidation/corrosion [15]. Hot-dip aluminizing of steels leads to the formation of an Al-rich topcoat and the Al-Fe intermetallic

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layer consisting of Fe_2Al_5 and FeAl_3 [16]. In this study, the corrosion behavior of hot-dip aluminized 9Cr-1Mo steel in $\text{N}_2/\text{H}_2\text{S}$ gas was investigated at 800-900 °C, because the industrially important H_2S gas is particularly destructive at high temperatures. Nitrogen was employed as the balance gas in order to simulate coal gasification systems [8]. This study will be a useful contribution to the development of corrosion-resistant coatings for iron-base alloys.

2. EXPERIMENT DETAILS

A P91 steel plate with a nominal composition of Fe-9Cr-1Mo-0.45Mn-0.4Si-0.2V-0.08Nb-0.1C (wt%) was ground to a 1000 grit SiC finish, immersed in a 10 vol% HCl solution to remove surface oxides, subjected to a liquid flux treatment with 20 vol% ($\text{KCl}+\text{AlF}_3$ in 4:1 weight ratio) solution, dried, and dipped in Al(l) for 5 min at 800 °C, on top of which a solid flux ($\text{KCl}+\text{NaCl}+\text{AlF}_3$ in 2:2:1 weight ratio) was spread to protect Al(l) from oxidation. After aluminizing, the cleaned samples were corroded at 800 and 900 °C for 50 h under 1 atm of flowing $\text{N}_2/0.1\%\text{H}_2\text{S}$ gas inside a quartz reaction tube furnace. The purity of the N_2 and H_2S gas was 99.999% and 99.95%, respectively. The samples were inspected using a field-emission scanning electron microscope (SEM) equipped with an energy dispersive spectrometer (EDS), a field-emission electron probe microanalyzer (EPMA), and a high-power X-ray diffractometer (XRD) with $\text{Cu-K}\alpha$ radiation at 40 kV and 150 mA. Microstructures were examined by etching the aluminized coating with Keller's reagent and the steel matrix with Villella's reagent, together with electron-back scattered diffraction (EBSD). Hereafter, the compositions are denoted in atomic percentages (at%).

3. RESULTS AND DISCUSSION

Figure 1 shows the EPMA/XRD/EBSD results of aluminized 9Cr-1Mo steel. The coating consisted of an ~20 μm thick topcoat and ~50 μm thick intermetallic layer, which were formed by interdiffusion between the Al(l) and substrate [16] (Fig. 1(a)). XRD patterns obtained before and after the successive grinding are shown in Figs. 1(b-d). The topcoat primarily consisted of Al with a small amount of $\text{Al}_{13}\text{Fe}_4$ (Fig. 1(b)). When the topcoat was partially ground

off, $\text{Al}_{13}\text{Fe}_4$, Al_5Fe_2 and Al peaks came out, indicating the presence of $\text{Al}_{13}\text{Fe}_4$ and Al_5Fe_2 below the Al-rich topcoat (Fig. 1(c)). Further surface grinding revealed Al_5Fe_2 only (Fig. 1(d)). The Powder Diffraction File (PDF) numbers of phases in the Al-Fe binary phase diagram are as follows. Al (85-1327), $\text{Al}_{13}\text{Fe}_4$ (29-0042), Al_5Fe_2 (47-1435), AlFe (33-0020), AlFe_3 (50-0955), and $\alpha\text{-Fe}$ (06-0696).

According to the concentration gradients of Al and Fe created during hot dipping, an Al-rich topcoat containing a small amount of $\text{Al}_{13}\text{Fe}_4$ and Al_5Fe_2 precipitates, an upper $\text{Al}_{13}\text{Fe}_4$ layer containing a small amount of Al_5Fe_2 precipitates, and a lower Al_5Fe_2 layer were formed on the surface (Fig. 1(e)). This phase distribution is also recognizable in the Al map shown in Fig. 1(f). The Al_5Fe_2 layer was thick and somewhat columnar, because orthorhombic Al_5Fe_2 has 30% vacancies along the c-axis, through which the interdiffusion of Al and Fe occurred fast [17,18] (Fig. 1(e)). Dissolution of the alloying elements such as Cr, Mo, and Mn in the Al_5Fe_2 layer flattened the coating/substrate interface and narrowed the Al_5Fe_2 layer (Figs. 1(a, f)). It is noted that, in the case of low-alloyed carbon steels, the coating/substrate interface exhibited a finger- or tongue-like morphology that was oriented along the c-axis of Al_5Fe_2 layer, and the Al_5Fe_2 layer was typically thick [19-21].

Aluminized 9Cr-1Mo steel was corroded at 800 °C for 50 h, and XRD'ed while successively grinding the coating surface. In Fig. 2(a), the $\text{Al}_{13}\text{Fe}_4$ and Al_5Fe_2 peaks are strong, while the $\alpha\text{-Al}_2\text{O}_3$ peaks are weak. This implies that the Al-rich topcoat transformed to $\text{Al}_{13}\text{Fe}_4$ and Al_5Fe_2 through the interdiffusion of Al and Fe, and was also partially oxidized to $\alpha\text{-Al}_2\text{O}_3$ at the surface. Moving deeper in the coating, the concentration of Al decreased, while that of Fe increased. Hence, strong Al_5Fe_2 and weak AlFe peaks came out after the first grinding (Fig. 2(b)). Only AlFe peaks came out after the second grinding (Fig. 2(c)). Strong AlFe_3 peaks came out after the third grinding in addition to the $\alpha\text{-Fe}$ substrate peak (Fig. 2(d)). In brief, the XRD results indicated the formation of ($\alpha\text{-Al}_2\text{O}_3$ surface scale)/($\text{Al}_{13}\text{Fe}_4$, Al_5Fe_2)-mixed layer)/(AlFe layer)/(AlFe_3 layer) on the $\alpha\text{-Fe}$ substrate. This is explained further in Fig. 3.

The compositions of spots 1, 2, and 3, denoted in Fig. 3(a), were 35.5Al-62.1O-0.3Fe-2Si-0.1S, 31.1Al-65.5O-1.7Fe-0.1Cr-0.4Mn-1.1Si-0.1S, and 39.7Al-58.6O-1.1Fe-0.1Cr-0.4Si-

Fig. 1. Hot-dip aluminized 9Cr-1Mo steel. (a) cross-sectional secondary electron (SE) image acquired from EPMA, (b) XRD pattern of the topcoat, (c) XRD pattern taken after first grinding, (d) XRD pattern taken after second grinding, (e) EBSD phase map, (f) EPMA maps of (a).

0.1S (at%), respectively, according to FESEM-EDS analysis (Fig. 3(b)). This implies that spots 1 and 2-3 correspond to the detached and adherent $\alpha\text{-Al}_2\text{O}_3$ scale, respectively. A small amount of substrate elements that diffused outwardly during heating presumably dissolved in the $\alpha\text{-Al}_2\text{O}_3$ scale. The standard Gibbs free energy changes and equilibrium partial pressures for sulfidation/oxidation reaction of Al at 800 °C are as follows [22].

$$= -1337.1 \text{ (kJ)}, P_{\text{O}_2(\text{eq})} = 4 \times 10^{-44} \text{ (atm)}$$

Since Al_2O_3 is much more stable than Al_2S_3 , the aluminum at spots 1-3 preferentially reacted with impurity oxygen in $\text{N}_2/0.1\%\text{H}_2\text{S}$ gas, resulting in the formation of protective $\alpha\text{-Al}_2\text{O}_3$ in preference to the less protective Al_2S_3 .

The alumina at spot 1 inevitably spalled for the following reasons. Firstly, growth stress developed due to the dissolution of foreign elements and the phase transformation of Al to $\alpha\text{-Al}_2\text{O}_3$. Particularly, the sulfur incorporation deteriorated the scale adherence [23]. Secondly, thermal stress was generated due to the mismatch in thermal expansion coefficients between spot 1 and spot 2, whose

Fig. 2. XRD patterns of hot-dip aluminized 9Cr-1Mo steel after corrosion at 800 °C for 50 h in N₂/0.1% H₂S gas. (a) before grinding, and after (b) first, (c) second, and (d) third grinding.

compositions were different. Thirdly, hydrogen released from H₂S gas might have dissolved in α -Al₂O₃ and deteriorated the adherence of α -Al₂O₃ [11]. Spots 4-5 belonged to the (Al₁₃Fe₄, Al₅Fe₂)-mixed layer (Figs. 2(a-b) and 3(a-b)). Spots 5-8 belonged to the AlFe layer (Figs. 2(c) and 3(a-b)). The AlFe layer consisted of coarse, columnar grains that aligned along the interdiffusion direction of the Al and substrate elements (Figs. 2(b-c) and 3(a-b)). AlFe can dissolve (23.3-55)% Al, according to the Al-Fe phase diagram. Spots 8-10, 10-12, and 12-13 belonged to the AlFe₃ layer, the Al-dissolved α -Fe layer, and the α -Fe substrate, respectively (Figs. 2(d) and 3(a-b)).

Such can also be envisaged in the Al map shown in Fig. 3(c). The detached scale at spot 1 consisted of fine, round α -Al₂O₃ grains, reflecting its slow growth rate (Fig. 3(d)). It cracked easily (Fig. 3(e)). All the substrate elements diffused outwardly, while Al, sulfur, and oxygen diffused inwardly, following the concentration gradients (Figs. 3(b-c)). This broadened and transformed the original coating. The Kirkendall effect that was produced by the unequal mass

flow, as well as the phase transformation of low density, high Al-containing phases to high density, low Al-containing phases, facilitated the formation of voids at spots 3-10 (Fig. 3(a)). The densities of Al₁₃Fe₄, Al₅Fe₂, AlFe, and AlFe₃ were 3.849, 3.96, 5.67, and 6.57 g·cm⁻³, respectively. The phase transformation also produced volume shrinkage, developing microcracks to relieve the stress. Cracks were observed between spots 3 and 5, as shown in Fig. 3(a).

The generation of voids that can act as stress concentration sites, the mismatch in thermal expansion coefficients among the transformed Al-Fe phases, and the dissolution of sulfur and possibly hydrogen also facilitated the microcracking in the coating. The detachment of the α -Al₂O₃ surface layer, and the development of easy-diffusion paths such as voids and microcracks, resulted in the development of internal oxides in the coating, as depicted in the oxygen map shown in Fig. 3(c). Like oxygen, sulfur also diffused inwardly through voids, grain boundaries, and microcracks, but to a lesser amount because of its very low solubility in most oxides and metals [24]. This is recognizable from the sulfur map shown in Fig. 3(c).

Figure 4 shows an unetched image of the corroded sample. The etched image was shown in Fig. 3(a). In Fig. 4, a scratch was engraved in the surface prior to corrosion in order to locate the original sample surface. No significant outward diffusion of substrate elements was found to have occurred, although the outward diffusion was confirmed from the formation of internal voids as shown in Fig. 3(a). The voids appeared smaller down in the coating. Cracks propagated across the (Al₁₃Fe₄, Al₅Fe₂)-mixed layer and AlFe layer (Fig. 4). Among Al-Fe intermetallics, Al₁₃Fe₄ and Al₅Fe₂ are brittle, AlFe is ductile, and Al₃Fe is the most ductile [25]. The α -Al₂O₃ surface scale was partially detached, below which the adherent α -Al₂O₃ layer existed (Fig. 4).

In order to evaluate the effect of aluminizing on corrosion resistance, bare 9Cr-1Mo steel was corroded under the corrosion condition given in Figs. 3-4. Prior to corrosion, inert Pd powder (< 1 μ m Φ) was sprayed onto the sample surface for the marker test. The outer and inner scale was about 75-200 and 50 μ m thick, respectively, between which agglomerated Pd powder existed (Fig. 5(a)).

Hence, it can be seen that the outer scale grew by the outward diffusion of substrate elements, while the inner scale

Fig. 3. Hot-dip aluminized 9Cr-1Mo steel after corrosion at 800 °C for 50 h in $N_2/0.1\%H_2S$ gas. (a) cross-sectional SE image acquired from EPMA (etched), (b) EDS concentration profiles along spots 1-13 shown in (a), (c) EPMA maps of (a), (d) SEM top view, (e) enlarged cross-sectional SEM image of the $\alpha-Al_2O_3$ scale.

grew by the inward diffusion of oxygen and sulfur (Fig. 5(b)). The XRD analysis indicated that both the outer and inner scales primarily consisted of FeS (Figs. 5(c-d)). FeS, whose defect structure is $Fe_{1-x}S$ ($x=0.0.2$), grows quickly by the outward diffusion of Fe^{2+} ions [14]. The poor corrosion

resistance of the bare 9Cr-1Mo steel was attributed to the formation of nonprotective FeS according to the reaction; $Fe+H_2S \rightarrow FeS+H_2$. Hydrogen evolution, excessive thickening of the FeS scale, and mismatch in thermal expansion coefficients between the outer and inner scale made the outer

Fig. 4. SEM cross-sectional image of hot-dip aluminized 9Cr-1Mo steel after corrosion at 800 °C for 50 h in $N_2/0.1\%H_2S$ gas (unetched).

scale nonadherent.

The outer FeS scale was coarse and fragile yielding inter- and trans-granular cracking, while the inner FeS scale was fine and adherent (Fig. 5(e)). Small amounts of Cr, Mo, Mn, Si, and V diffused outwardly to dissolve in the outer FeS grains (Fig. 5(b)). The consumption of sulfur in the outer FeS scale decreased the sulfur potential and thereby increased the

oxygen potential in the inner scale (Fig. 5(b)). However, the amount of oxygen in $N_2/0.1\%H_2S$ gas was at the impurity level, so the inner scale primarily consisted of FeS (Fig. 5(d)). In contrast, aluminized 9Cr-1Mo steel displayed good corrosion resistance by forming highly protective, stable $\alpha-Al_2O_3$ through the reaction of Al with impurity oxygen in $N_2/0.1\%H_2S$ gas, as outlined in Figs. 2-4.

Figure 6 shows XRD/SEM/EDS/EPMA results for the 9Cr-1Mo steel after corrosion at 900 °C for 50 h. Figure 6(a) indicates the formation of $\alpha-Al_2O_3$, Fe_2O_3 , and $FeAl_2O_4$ around the $AlFe_3$ coating. This suggests that aluminum in the original Al-rich topcoat oxidized to $\alpha-Al_2O_3$. At the same time, iron that diffused outward across the original Al-rich topcoat according to the concentration gradient oxidized to Fe_2O_3 , and some of Fe_2O_3 was further transformed to the more stable $FeAl_2O_4$ spinel. When the surface was ground off, $\alpha-Fe$ newly came out besides $\alpha-Al_2O_3$, Fe_2O_3 , and $AlFe_3$ (Fig. 6(b)). This indicates the presence of $\alpha-Fe$ underneath the $AlFe_3$ coating containing some oxides. The above oxides and coating phases are illustrated in Fig. 6(c).

Spot 1 in Fig. 6(c) indicates $\sim 5 \mu m$ -thick, nonadherent $\alpha-$

Fig. 5. Bare 9Cr-1Mo steel after corrosion at 800 °C for 50 h in $N_2/0.1\%H_2S$ gas. (a) cross-sectional SE image acquired from EPMA, (b) EPMA line profiles along A-B denoted in (a), (c) XRD pattern (outer scale), (d) XRD pattern (inner scale), (e) SEM top view.

Fig. 6. Hot-dip aluminized 9Cr-1Mo steel after corrosion at 900 °C for 50 h in $\text{N}_2/0.1\%\text{H}_2\text{S}$ gas. (a) XRD pattern before grinding, (b) XRD pattern after grinding, (c) SEM cross-sectional SE image (etched), (d) EDS concentration profiles along spots 1-13 shown in (c), (e) EPMA maps of (c), (f) SEM top view of the detached scale.

Al_2O_3 scale with a composition of 32.5Al-66.6O-0.1S-0.1Cr-0.2Mo-0.5Si (%), according to the EDS spot analysis (Fig. 6(d)). The dissolution of foreign elements, including sulfur, favored spallation of the $\alpha\text{-Al}_2\text{O}_3$ surface scale. Spot 2 was located at the (adherent scale)/(AlFe_3 coating) interface. The adherent scale was $\sim 10\ \mu\text{m}$ -thick, and mostly consisted of $\alpha\text{-Al}_2\text{O}_3$, Fe_2O_3 and FeAl_2O_4 tended to exist underneath the adherent or nonadherent $\alpha\text{-Al}_2\text{O}_3$ -rich surface scale, as can

be noted from the O-Al-Fe maps shown in Fig. 6(e).

Spots 2-5 indicate a $\sim 40\ \mu\text{m}$ -thick AlFe_3 layer containing some oxides and voids (Fig. 6(c)). Their average composition was 26.8Al-64.4Fe-2.6O-0.2S-5.2Cr-0.2Mo-0.4Mn-0.1Si-0.1V (%), implying the incorporation of oxygen, sulfur, and alloying elements in coarse AlFe_3 grains (Figs. 6(c-e)). Spots 5-12 indicate a $\sim 110\ \mu\text{m}$ -thick Fe(Al) layer. Their average composition was 76Al-11.2Al-8.4Cr-0.5Mo-0.4Mn-0.7Si-

0.2V-2.50-0.1S, indicating that coarse, columnar α -Fe grains were dissolved with a large amount of Al plus a small amount of alloying elements, oxygen, and sulfur (Figs. 6(c-e)). The oxygen map shown in Fig. 6(e) indicates scattered internal oxides at spots 2-5. Dark dots at spots 5-12 were voids that formed primarily due to the Kirkendall effect (Fig. 6(c)). The sulfur map shown in Fig. 6(e) indicates the presence of sulfur sporadically distributed around the surface.

Oxygen and sulfur can ingress rather easily through Kirkendall voids, and imperfect oxide scale. However, the sulfur concentration was less than 1% at spots 1-13, because the solubility of sulfur in most oxides and metals was very low [24] (Fig. 6(d)). The detached scale at spot 1 primarily consisted of fine, round α - Al_2O_3 grains, reflecting the slow growth rate of alumina (Fig. 6(f)). This is also displayed in Fig. 3(d). Despite the detachment and imperfection of the α - Al_2O_3 -rich scale, the aluminized coating effectively resisted the harsh H_2S -gas corrosion.

Results obtained from Figs. 2-4 and 6 can be summarized as follows. Corrosion at 800 °C for 50 h produced an α - Al_2O_3 layer with a small amount of Fe_2O_3 , an $(\text{Al}_{13}\text{Fe}_4, \text{Al}_5\text{Fe}_2)$ -mixed layer, an AlFe layer consisting of columnar grains aligned along the interdiffusion direction, and an α -Fe(Al) layer. Corrosion at 900 °C for 50 h produced the α - Al_2O_3 and AlFe_3 layers with some Fe_2O_3 and FeAl_2O_4 , and the α -Fe(Al) layer consisting of fully grown columnar grains. This transformation of the Al-Fe phases in the coating at 800-900 °C originated from the interdiffusion of Al and Fe in the Al/Fe diffusion couple. For example, the interdiffusion coefficient of Fe and Al in the iron aluminide was determined to be 5.93×10^{-16} and $2.92 \times 10^{-14} \text{ m}^2/\text{s}$ at 550 and 640 °C, respectively [26]. Likewise, increasing the corrosion temperature from 800 to 900 °C transformed (high Al)-Fe phases to (low Al)-Fe phases, and drastically broadened the coating, as schematically illustrated in Fig. 7.

4. CONCLUSIONS

ASTM P91 steel was subjected to a hot-dip aluminized coating, which consisted of an Al-rich topcoat with a small amount of $\text{Al}_{13}\text{Fe}_4$ and Al_5Fe_2 precipitates, an $\text{Al}_{13}\text{Fe}_4$ layer having a small amount of Al_5Fe_2 precipitates, and an Al_5Fe_2 layer with dissolved substrate elements. Aluminizing

Fig. 7. Schematic diagram showing the phase transformation and thickness variation of hot-dip aluminized coating on 9Cr-1Mo steel. (a) before corrosion, and after corrosion for 50 h at (b) 800 °C, and (c) 900 °C.

effectively improved corrosion resistance by forming Al_2O_3 -rich surface scales through the preferential reaction of Al with impurity oxygen in corrosive $\text{N}_2/0.1\%\text{H}_2\text{S}$ gas. When a bare sample was exposed, a nonprotective, thick, bilayered FeS scale formed. In the aluminized samples, substrate elements diffused outwardly, while Al, sulfur, and oxygen diffused inwardly, which broadened and transformed the Al-Fe phases during corrosion. This was accompanied by the formation of internal oxides, voids, and microcracks in the coating.

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