

# Corrosion Behavior of Fe-Al-Cr Alloys in Sulfur- and Oxygen-Rich Environments in the Presence of Pyrite

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## ABSTRACT

Iron-aluminum-based alloys recently have been considered as weld overlay coatings for corrosion protection. These alloys previously have been shown to possess excellent oxidation and sulfidation resistance when exposed to gaseous environments. Although gaseous corrosion contributes greatly to the total amount of corrosion that can occur during exposure, it has been reported that a solid, reactive slag can accumulate on the boiler tube surface as well. Slag can consist of gaseous corrosion products, unburnt coal particles, and ash from burnt coal. Therefore, to better represent the corrosive conditions in the coal-burning furnace, several Fe-Al-Cr-based alloys were exposed to two reactive gases at 500°C while in contact with reactive pyrite (FeS<sub>2</sub>) powder. Two alloys contained Ti additions to determine if these additions would have any beneficial effect on the corrosion behavior of the alloys. Light optical microscopy was used to observe the scales that formed during exposure. Measurements were made of the substrate corrosion layer thickness to quantify the extent of corrosion that occurred. Electron probe microanalysis (EPMA) line scans were used to determine the corrosion layer compositions on selected alloys. Critical alloying contents required to prevent significant substrate corrosion from occurring during the test time were determined. It was found that 19 at% Al was required to prevent significant substrate corrosion from occurring during 100 h of exposure.

**KEY WORDS:** chromium additions, high temperature, iron-aluminum, iron disulfide, oxidation, pyrite, sulfidation

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## INTRODUCTION

Traditional high-temperature corrosion testing has been carried out in simulated corrosive environments where an alloy is directly exposed to a corrosive gas at elevated temperatures.<sup>1-9</sup> Although these tests are extremely valuable when determining potential corrosion-resistant coatings, it has been shown that sulfur-rich slag deposits can accumulate on components in low NO<sub>x</sub> furnaces, such as boiler tube walls, and can accelerate the corrosion rates as well.<sup>10</sup> Flame impingement from the burner in a fossil-fired boiler can increase the amount of ash and unburned coal reaching the furnace walls, which can accelerate the waterwall wastage further.<sup>11</sup> The accumulation of these deposits transforms into a layer of slag, which can contain various reactive species. For example, slag can contain chlorine and sulfur from the unburned coal as well as excess carbon from ash.<sup>10</sup> In contrast, furnaces prior to low NO<sub>x</sub> retrofitting were found to have slag accumulations that consisted mainly of ash deposits and contained little or no chlorine and sulfur.<sup>10</sup>

Slag collections from actual boilers have shown that troilite (FeS) can be found in areas where corrosion losses were high.<sup>12</sup> FeS can form due to incomplete decomposition of pyrite (FeS<sub>2</sub>) during the combustion process. FeS<sub>2</sub>, which is a component of coal, may not be able to oxidize completely to an iron oxide, due to a low amount of oxygen in the atmosphere, and instead form FeS.<sup>12</sup> In an unpublished study performed at Lehigh University, it has been

TABLE 1

Alloy Compositions Used for Corrosion Testing<sup>(A)</sup>

Alloy Designation	Fe	Al	Cr	Ti
Fe-14.5Al	Bal.	14.2	—	—
Fe-14.5Al-1Cr	Bal.	14.3	1.0	—
Fe-14.5Al-2Cr	Bal.	14.5	2.1	—
Fe-14.5Al-5Cr	Bal.	14.8	5.0	—
Fe-14.5Al-2Cr-1.5Ti	Bal.	14.9	2.2	1.8
Fe-19Al	Bal.	18.8	—	—
Fe-19Al-1Cr	Bal.	18.8	1.0	—
Fe-19Al-2Cr	Bal.	19.0	2.1	—
Fe-19Al-5Cr	Bal.	19.9	5.0	—
Fe-19Al-2Cr-1.5Ti	Bal.	19.2	2.1	1.7

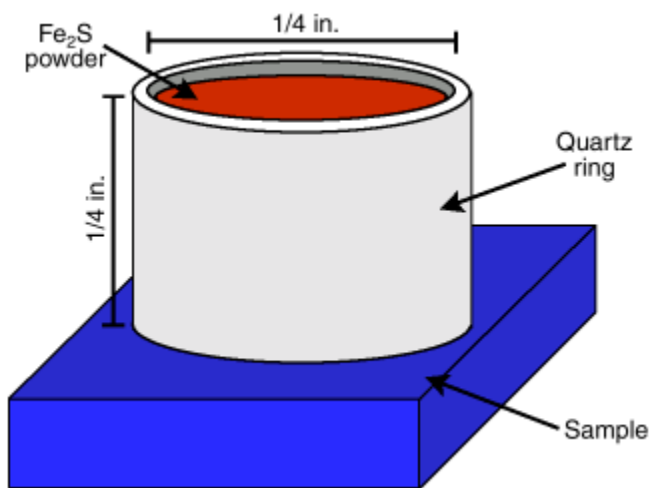
<sup>(A)</sup> All values are in at%.

FIGURE 1. Schematic diagram of the gas-slag-metal experimental setup.

shown that  $\text{FeS}_2$  converted to  $\text{FeS}$  during exposure to a moderately sulfidizing atmosphere and when exposed to an oxidizing atmosphere,  $\text{FeS}_2$  oxidized to form hematite ( $\text{Fe}_2\text{O}_3$ ). Upon combustion, flame impingement possibly could deposit all three of these compounds:  $\text{FeS}_2$ ,  $\text{FeS}$ , and  $\text{Fe}_2\text{O}_3$  on the boiler tube walls. To make matters worse, the local corrosion environment around the powder potentially may be more sulfidizing than the gaseous environment alone because the  $\text{FeS}_2$  powder released sulfur into the environment during the transformations to  $\text{FeS}$  and  $\text{Fe}_2\text{O}_3$ .

Therefore, to better simulate actual burner conditions, a relatively new test combining gaseous and solid-state corrosion was performed on several Fe-Al-based alloys. Fe-Al-based alloys were tested because they recently have been considered as weld overlay coatings for the corrosion protection of boiler tubes in coal burning atmospheres. The corrosion tests in this study made use of both gaseous and solid-state corrosion environments. The gas-slag-metal experi-

ments were carried out using  $\text{FeS}_2$  powder as the simulated slag and exposures were carried out in two gaseous corrosive environments: a mixed oxidizing/sulfidizing atmosphere and an oxidizing atmosphere. The purpose of this study was to determine alloy compositions that demonstrated excellent corrosion resistance in simulated gaseous corrosion environments while in contact with a reactive slag.

## EXPERIMENTAL PROCEDURES

Alloys were cast by arc-melting high-purity components under an argon atmosphere and drop cast into a water-cooled copper mold. Although weld overlays were the coatings of interest, cast alloys were used because it was shown previously that the high-temperature corrosion behavior of weld claddings could be explained by using cast alloys of equivalent composition.<sup>13</sup> It has been reported since that the corrosion behavior between weld overlays and cast alloys are similar due to the lack of microsegregation within the welds and both having coarse-grained microstructures.<sup>14</sup> Binary Fe-Al alloys, ternary Fe-Al-Cr alloys, and quaternary Fe-Al-Cr-Ti alloys that were used for this study are listed in Table 1. Quartz ring corrosion experiments were conducted using a high-temperature horizontal tube furnace. The samples were ground to 600 grit and cleaned using acetone ( $\text{CH}_3\text{COCH}_3$ ). The corrosion test setup consisted of a quartz ring containing  $\text{FeS}_2$  powder in contact with the alloy surface. A predetermined amount (1,680 mg) of  $\text{FeS}_2$  powder was poured into the quartz ring without being packed, and this setup was placed into the furnace. A schematic for the gas-slag-metal samples can be seen in Figure 1. Either the mixed oxidizing/sulfidizing gas or the oxidizing gas then was introduced into the furnace at a flow rate of 300 mL/min. The samples were heated at a rate of  $50^\circ\text{C}/\text{min}$  and were held at  $500^\circ\text{C}$  for 100 h. Water vapor present in both gases was injected into the furnace at a controlled rate via a capillary tube and a syringe pump. The components of the mixed oxidizing/sulfidizing and oxidizing gases used for exposure can be seen in Table 2. The sulfur and oxygen partial pressures were calculated using thermodynamic modeling software.<sup>15</sup> Samples were mounted carefully in cold setting epoxy and their polished cross sections were observed using light optical microscopy (LOM). LOM images were taken with a camera integrated with a light optical microscope and digital imaging system. The thickness of the internal corrosion products observed in nonprotective alloys were measured using this digital imaging system and a quantitative image analysis system. Quantitative chemical analysis data was obtained using electron probe microanalysis (EPMA) at 20 keV. A phi( $\rho z$ ) correction scheme was used to correct for absorption and fluorescence of x-rays that occur during EPMA.

## RESULTS AND DISCUSSION

### Mixed Oxidizing/Sulfidizing Environment

Polished cross sections of alloys containing 14.5 at% Al (all values reported in at%) exposed to the mixed oxidizing/sulfidizing gas can be seen in Figure 2. It can be seen from this figure that alloys containing 14.5% Al and <2% Cr all formed significant substrate and external corrosion scales after 100 h of exposure. The substrate corrosion products formed on these alloys were uniform and appeared to be made up of multiple corrosion layers. Fe-14.5Al-5Cr formed a significant substrate scale, similar to the aforementioned alloys, but formed a very thin external corrosion layer. All alloys containing 19% Al tested in the mixed oxidizing/sulfidizing environment showed no signs of corrosion after 100 h of exposure (Figure 3[b]).

The external corrosion layers formed on the 14.5% Al alloys were fairly nonuniform and appeared to be several microns thick. These external corrosion layers on these alloys looked identical to the powder imbedded in the epoxy above. It has been determined previously at Lehigh University that FeS<sub>2</sub> powder will convert to FeS during exposure to the mixed oxidizing/sulfidizing gas. The powder imbedded into the epoxy was identified as FeS based on previous reports and EDS analysis. Because both the morphology and contrast between the external scale and the powder appeared to be identical, the external scale adjacent to the metal surface was most likely FeS. It is safe to say that this layer is an external corrosion product and not powder on the metal surface because of the following:

- the external corrosion product was uniformly present across the exposed surface, and
- the external layer was not present on samples that were protective during 100 h of exposure (Figure 3).

Thickness measurements were taken of the substrate corrosion scales present on the alloys containing 14.5% Al to determine if chromium or titanium additions had any significant effect on the corrosion behavior of these alloys. The thickness measurements are shown in Table 3. As can be seen from this table, some measured improvement to the substrate scale thickness was made only when 5% Cr was added. Although no measurements were made of the external corrosion product thickness, similar results were observed. Referring back to Figure 2, it can be seen qualitatively that chromium additions of 1% to 2% may have made a slight improvement on the external corrosion layer thickness of the 14.5% Al alloys, but significant improvement occurred only when 5% Cr was added. Titanium did not seem to make any improvement on the corrosion behavior of these alloys. Unfortunately, as a result of problems with surface roughness within the substrate scale

**TABLE 2**

*Gas Compositions Used for Corrosion Testing (vol%)*

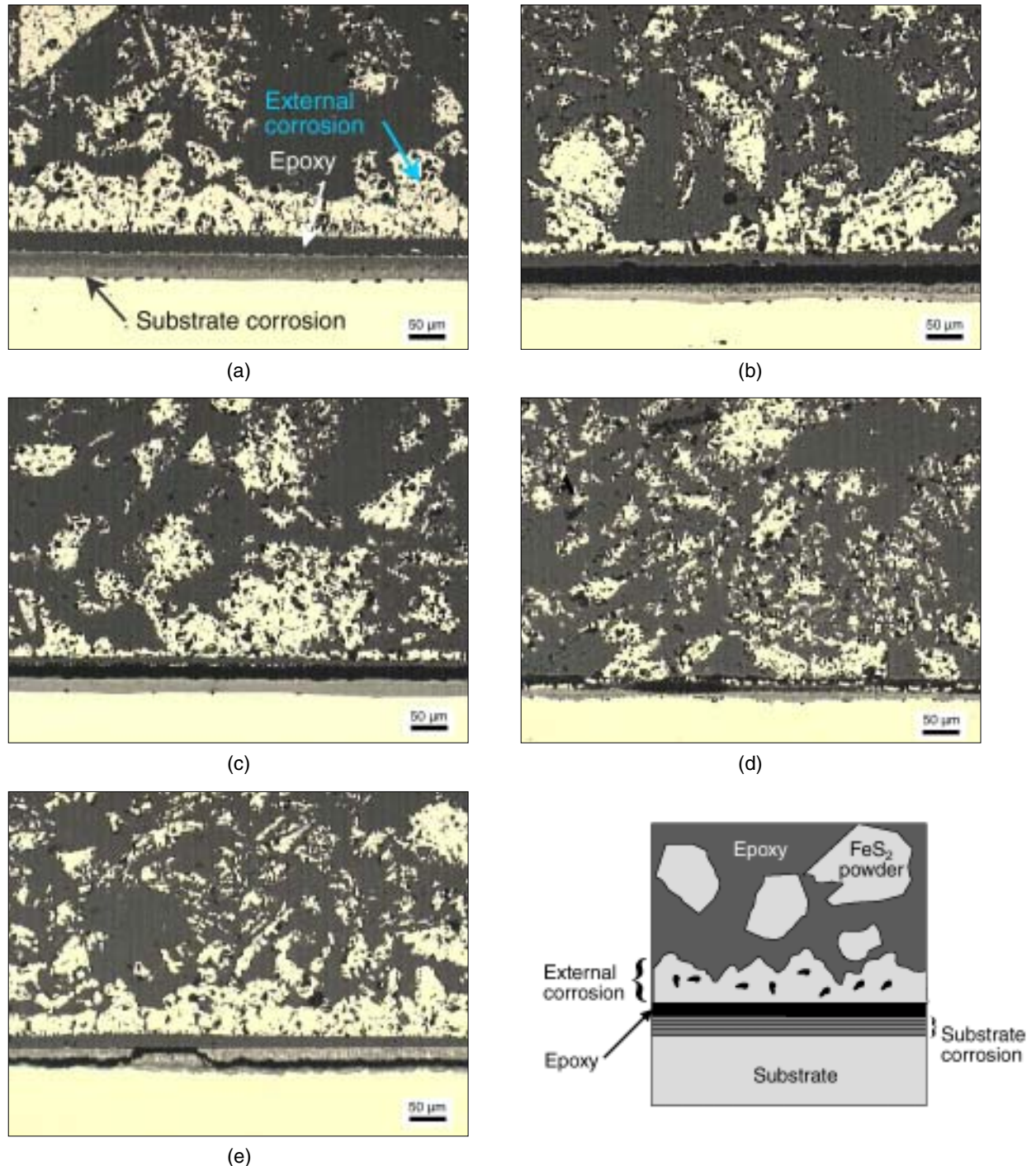
Gas Component	Mixed Oxidizing/Sulfidizing Gas	Oxidizing Gas
O <sub>2</sub>	—	2
CO	10	—
CO <sub>2</sub>	5	15
H <sub>2</sub>	—	—
H <sub>2</sub> O	2	6
H <sub>2</sub> S	0.12	—
SO <sub>2</sub>	—	0.12
N <sub>2</sub>	Bal.	Bal.
Log P <sub>O<sub>2</sub></sub>	-19	-2
Log P <sub>S<sub>2</sub></sub>	-8	-46

area on samples exposed to the mixed oxidizing/sulfidizing atmosphere, quantitative analysis could not be performed to help identify the phases that formed.

### Oxidizing Environment

Polished cross sections of alloys containing 14.5% Al exposed to the oxidizing gas (Figure 4) showed that all of these alloys developed thick external corrosion layers (at least 100 μm thick) as well as uniform substrate corrosion scales. The external corrosion scales seemed to consist of two distinct layers. The external layer directly adjacent to the metal substrate was observed to contain a large amount of porosity while the outer layer of the external scale was a thick, solid layer that contained very little porosity. The substrate corrosion products observed on these alloys seemed to consist of multiple layers, and cracks (arrows) could be seen to run through the substrate corrosion products perpendicular to the substrate/external corrosion scale interface. Some samples, namely Fe-14.5Al and Fe-14.5Al-2Cr-1.5Ti, showed cracks that penetrated into the base metal as well. Corrosion products were observed within some of these cracks (see box in Figure 4[e]). This would indicate that the cracks formed sometime during exposure where corrosive species could penetrate into the substrate. Penetrative cracks such as these can act as fast pathways for corrosion to take place and therefore are very detrimental to the corrosion resistance of the alloy. The only alloy containing 14.5% Al that did not show significant cracking of the internal scale was Fe-14.5Al-5Cr, which contained the smallest substrate corrosion layer, and could indicate that the perpendicular cracks observed in these samples are related to the thickness of the substrate corrosion scales.

Thickness measurements again were taken of the inward growing substrate corrosion scales formed on these alloys to determine if the alloying additions had any significant effect on the corrosion behavior. The substrate corrosion scale thickness

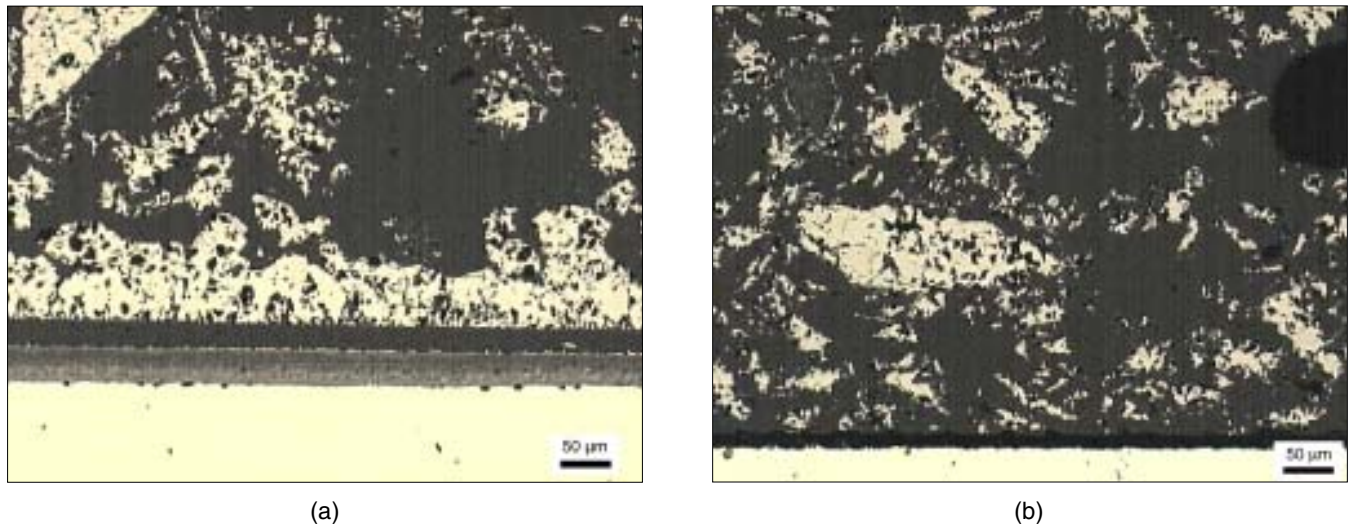


**FIGURE 2.** Samples containing 14.5 at% Al exposed to  $\text{FeS}_2$  powder and the mixed oxidizing/sulfidizing environment at  $500^\circ\text{C}$  for 100 h: (a) Fe-14.5Al, (b) Fe-14.5Al-1Cr, (c) Fe-14.5Al-2Cr, (d) Fe-14.5Al-5Cr, (e) Fe-14.5Al-2Cr-1.5Ti.

measurements for these alloys exposed to the oxidizing gas can be seen in Table 4. Similar to the mixed oxidizing/sulfidizing gas results, there was no significant effect of chromium on the substrate scale thickness until 5% Cr was added. It also was observed that cracking of the substrate corrosion scale occurred in all samples containing thickness layers in excess of approximately  $45\ \mu\text{m}$ . The external corro-

sion layer thickness seemed to qualitatively decrease slightly with the addition of chromium, but the scale/slag interface was not flat enough to obtain relevant thickness data. Again, the addition of titanium to Fe-14.5Al-2Cr did not have any beneficial effect on the corrosion resistance of this alloy.

Similar to alloys exposed to the mixed oxidizing/sulfidizing gas, all alloys containing 19 at% Al ex-



**FIGURE 3.** Comparison between typical nonprotective and protective samples after exposure to  $\text{FeS}_2$  powder and the mixed oxidizing/sulfidizing environment at  $500^\circ\text{C}$  for 100 h. (a) Samples containing 14.5% Al (Fe-14.5Al) formed both external and substrate corrosion scales (b) while protective samples containing 19% Al (Fe-19Al) did not form any corrosion products.

posed to the oxidizing gas (Figure 5) formed neither a significant substrate nor external corrosion product during 100 h of exposure. From these results, it can be seen that 19% Al is required to prevent significant corrosion from taking place in both the mixed oxidizing/sulfidizing and oxidizing environments when alloys are in contact with  $\text{FeS}_2$ . The additions of <5% Cr had little effect on the corrosion behavior of alloys containing 14.5% Al, but adding 5% Cr helped to decrease the amount of corrosion that occurred on these alloys. Titanium additions again had no significant effect on the corrosion behavior of any of the alloys in either environment.

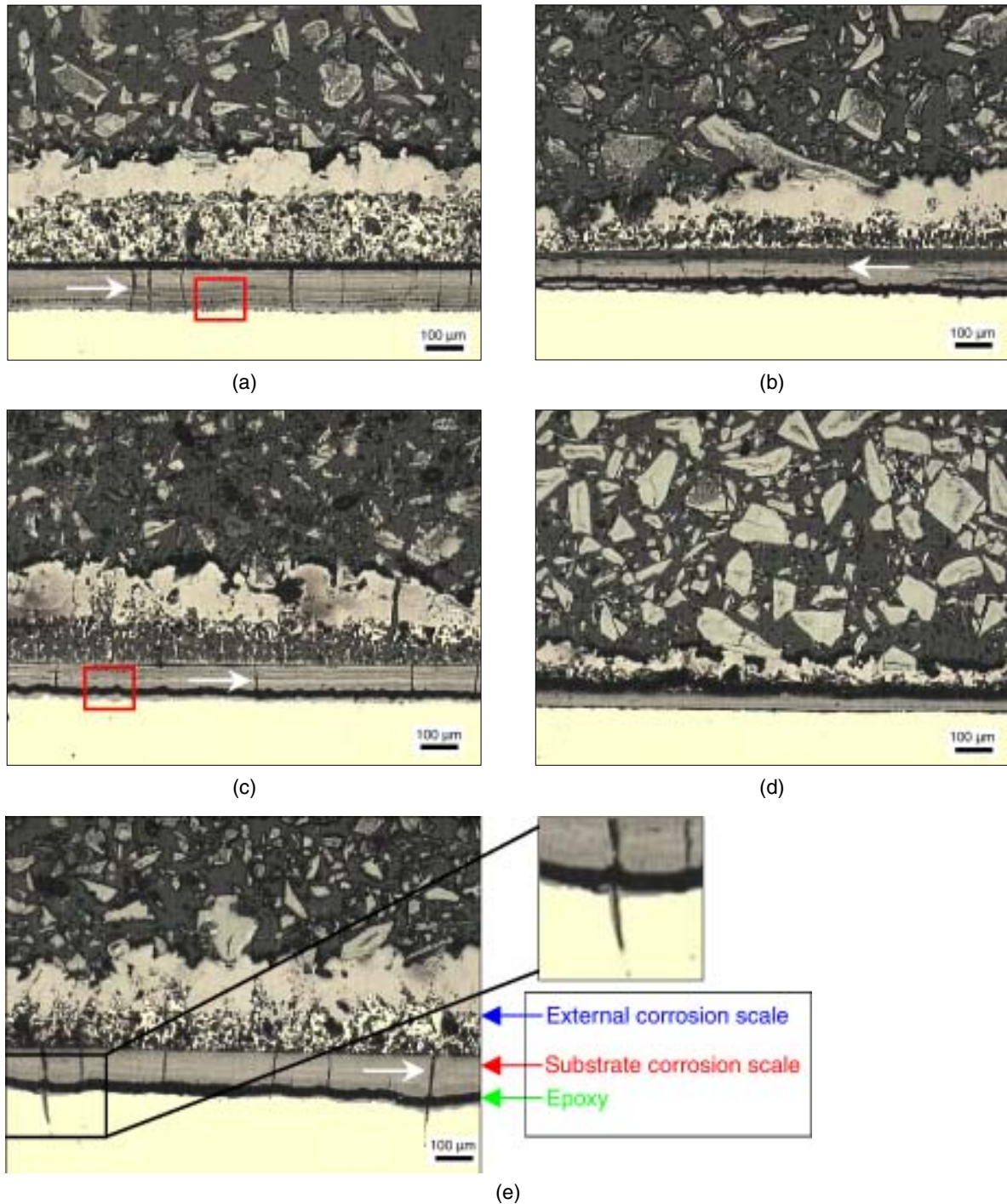
To better understand the types of reactions that took place during the gas-slag-metal experiments, the substrate corrosion scales that formed during exposures to the oxidizing environment were considered in greater detail by obtaining quantitative compositional data on the individual internal corrosion layers. Quantitative compositional data was obtained for two samples exposed to the oxidizing environment, Fe-14.5Al and Fe-14.5Al-2Cr, by using electron microprobe analysis (EPMA). The points of analysis were selected in an attempt to obtain data for the individual corrosion products that comprised the entire substrate corrosion layer.

A representative compositional trace, taken for Fe-14.5Al exposed to the oxidizing environment, can be seen in Figure 6. It can be seen in detail from Figure 6(a) that the substrate corrosion product actually consisted of individual layers. From the layers it was observed that there were two distinct regions in the substrate corrosion scale. A lighter corrosion region could be seen adjacent to the base metal and a darker region could be seen toward the alloy surface.

**TABLE 3**  
Substrate Corrosion Scale Thickness  
for Samples Exposed to  $\text{FeS}_2$  Powder  
and the Mixed Oxidizing/Sulfidizing Environment

Alloy	Thickness ( $\mu\text{m}$ )	Standard Deviation
Fe-14.5Al	30.5	2.1
Fe-14.5Al-1Cr	19.7	4.3
Fe-14.5Al-2Cr	26.3	2.8
Fe-14.5Al-5Cr	11.3	1.8
Fe-14.5Al-2Cr-1.5Ti	28.6	2.2

From the compositional data it could be seen that the substrate scale formed on Fe-14.5Al was rich in iron, aluminum, oxygen, and sulfur. The oxygen concentration was slightly greater in the lighter scale region than in the darker scale region, while sulfur was greater in the darker region than the lighter scale region. The aluminum content also was greater in the darker scale region than the lighter region. Similar results were found for the Fe-14.5Al-2Cr sample exposed to the oxidizing atmosphere. A representative EPMA line trace that was conducted on the Fe-14.5Al-2Cr sample exposed to the oxidizing environment can be seen in Figure 7. Note that the dark region cutting across the substrate corrosion scale is a crack due to metallographic preparation. Again, it can be seen that the substrate corrosion layer was made up of individual corrosion layers while having a lighter scale region at the scale/substrate interface and a darker region closer to the alloy surface. The substrate scale that formed on the Fe-14.5Al-2Cr sample exposed to the oxidizing environment was rich in iron, aluminum, chromium, oxygen, and sul-



**FIGURE 4.** Samples containing 14.5 at% Al in contact with  $\text{FeS}_2$  powder and exposed to the oxidizing environment at  $500^\circ\text{C}$  for 100 h. The arrows indicate cracks running perpendicular to the substrate/external corrosion scale interface. Note the corrosion product that formed within the penetrating crack in the Fe-14.5Al-2Cr-1.5Ti sample. (a) Fe-14.5Al, (b) Fe-14.5Al-1Cr, (c) Fe-14.5Al-2Cr, (d) Fe-14.5Al-5Cr, (e) Fe-14.5Al-2Cr-1.5Ti.

fur. The EPMA trace showed that the oxygen concentration was slightly greater in the lighter scale region than in the darker region.

It was observed by close examination of Figures 6 and 7 that there was a slight depletion in aluminum in the region adjacent to the substrate corrosion

product. The nominal aluminum concentration of the alloy was approximately 14.5% Al, but the aluminum concentration adjacent to the substrate corrosion product was approximately 10% Al for both alloys. This depleted region may be due to the formation of aluminum-rich compounds in the substrate

corrosion layers. Aluminum and chromium levels also seemed to increase from the base metal toward the alloy surface in both alloys. This indicates that the alloying elements diffused outward from the alloy to form the external and substrate corrosion products.

As previously mentioned, iron-aluminum alloys have been shown to be protective only when the aluminum content of the alloy is above a critical amount.<sup>1-2,16-20</sup> The critical aluminum content required for corrosion protection varies depending on the corrosion environment, temperature, and other elements present in the alloy.<sup>1-2,16</sup> For example, authors have reported that in oxidizing environments within the range of 600°C to 800°C, critical aluminum concentrations range from 14.5% Al to 22% Al (7.5 wt% to 12 wt%), depending on the testing temperature.<sup>19-22</sup> It also has been reported that in mixed oxidizing/sulfidizing environments that >21% Al was required for protection in isothermal testing conditions, but >25% Al was needed for protection in thermal cycling conditions.<sup>23-24</sup> Although these critical aluminum concentrations have been reported for a variety of environments, they may not apply to the present study because the previously mentioned studies were conducted at temperatures >500°C and the alloys were not exposed to FeS<sub>2</sub> powder. However, even if the aluminum content of the alloy is above the critical content needed for protection, outward diffusion of oxidizing elements can be problematic when the alloy becomes depleted of aluminum and chromium below the corrosion scale. When the Al and Cr compositions drop below the critical values, corrosion products can form into the alloy causing

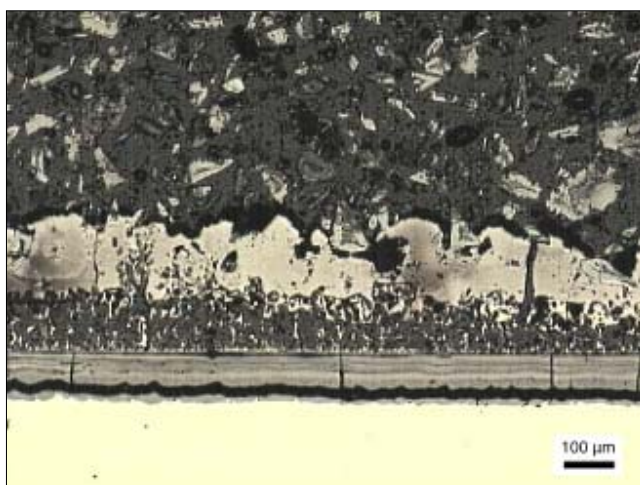
**TABLE 4**

*Substrate Corrosion Scale Thickness for Samples Exposed to FeS<sub>2</sub> Powder and the Oxidizing Environment*

Alloy	Thickness (μm)	Standard Deviation
Fe-14.5Al	103.1	12.5
Fe-14.5Al-1Cr	69.2	2.0
Fe-14.5Al-2Cr	91.3	1.6
Fe-14.5Al-5Cr	43.3	2.0
Fe-14.5Al-2Cr-1.5Ti	88.4	19.3

the inward growing substrate corrosion layers to form.

Therefore, from these substrate corrosion measurements, it was found that alloys containing 19% Al were required to prevent substrate corrosion from occurring during 100 h of exposure. These results are in good agreement with gaseous corrosion results that showed that 19% Al was required to suppress the formation of nodules and rapid corrosion rates.<sup>1-2</sup> Chromium additions seemed to help improve the corrosion resistance of the iron-aluminum alloys. Additions of chromium to alloys containing 14.5% Al helped improve the corrosion resistance of these alloys. Specifically, 5% Cr seemed to have the most significant effect on the amount of corrosion that occurred during 100 h of exposure. Chromium additions did not have much of an effect on the corrosion behavior of alloys containing 19% Al, due to the fact that the binary Fe-19Al alloy was protective in both environments. Titanium additions seemed to have no significant effect on the corrosion behavior of Fe-Al-Cr alloys.

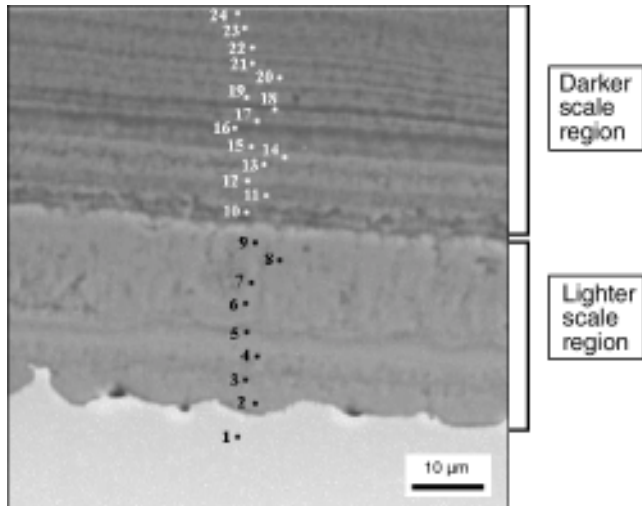


(a)

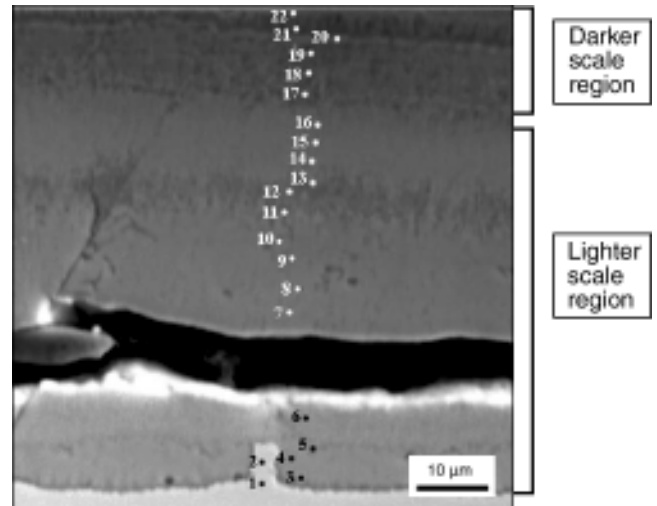


(b)

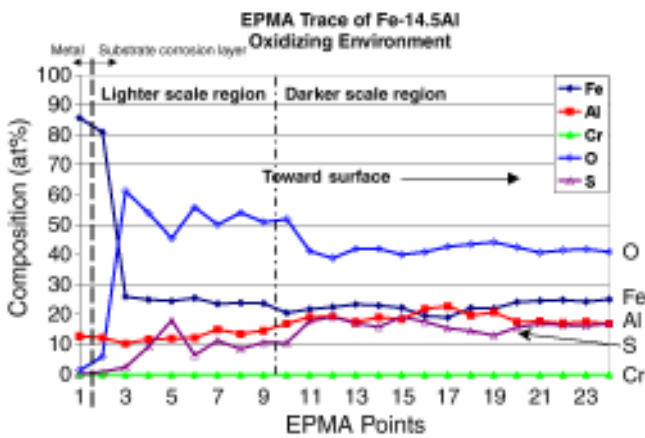
**FIGURE 5.** Comparison of typical nonprotective and protective samples exposed to FeS<sub>2</sub> powder and the oxidizing environment at 500°C for 100 h. (a) Samples containing 14.5% Al (Fe-14.5Al-2Cr) were observed to form both external and substrate corrosion scales and (b) protective samples containing 19% Al (Fe-19Al-2Cr) formed no corrosion products during exposure.



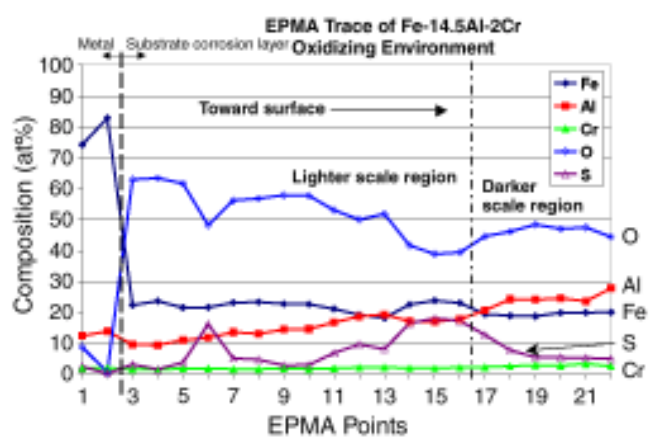
(a)



(a)



(b)



(b)

**FIGURE 6.** Typical EPMA trace across the substrate corrosion scale for Fe-14.5Al exposed to the oxidizing environment while in contact with the  $\text{FeS}_2$  powder (see box in Figure 4[a]).

**FIGURE 7.** Typical EPMA trace across the substrate corrosion scale for Fe-14.5Al-2Cr exposed to the oxidizing environment while in contact with the  $\text{FeS}_2$  powder (Figure 4[c]).

## CONCLUSIONS

- ❖ Ten Fe-Al-based alloys were exposed to two corrosive environments while in contact with  $\text{FeS}_2$  powder at 500°C for 100 h.
- ❖ In the mixed oxidizing/sulfidizing environment, alloys containing Al concentrations of only 14.5 at% were not protective, regardless of Cr and Ti additions. These alloys all formed substrate corrosion layers, which were approximately 20  $\mu\text{m}$  to 30  $\mu\text{m}$  thick, along with significant external corrosion layers.
- ❖ In the oxidizing environment, it was found that alloys containing 14.5 at% Al again were not protective and formed both internal and external growing corrosion products. The substrate corrosion layer showed cracks that protruded into the base metal as well. Additions of 5 at% Cr helped to slightly reduce the amount of corrosion that occurred during 100 h of exposure.

- ❖ Alloys containing 19 at% Al performed well in the mixed oxidizing/sulfidizing environment, as they formed no significant corrosion products. Increasing the aluminum concentration to 19 at% also improved the corrosion resistance in the oxidizing environment dramatically. It therefore was concluded that 19 at% Al was required for protection from substrate corrosion in both environments during 100 h of exposure at 500°C.

- ❖ EPMA results on samples exposed to the oxidizing environment showed that the substrate corrosion scale that formed on gas-slag-metal samples consisted of various layers that varied slightly in composition. A region adjacent to the substrate corrosion scale was slightly depleted in aluminum and these levels increased within the scale toward the surface of the alloy. This indicated that the alloying elements diffused outward from the sample to form both external and substrate corrosion scales.

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