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ELEVATED TEMPERATURE COMBINED EROSION–CORROSION OF STEELS

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ABSTRACT

An investigation was made of the effect of erodent particle size and test temperature on the combined erosion-corrosion behavior of a series of chromium containing steels with 2 1/4, 5, 9, 12% and 18% chromium. The particle sizes used were 5μm, 50μm, and 100μm and the test temperatures ranged from 700°C to 1000°C. The tests were made in the oxygen rich exhaust gases of a methane-air burner that propelled the particles at the specimens at 5 m/s for 30 minutes.

It was determined that corrosion was the dominant mechanism at all test conditions for all alloys. The erosion process enhanced the growth rate of multi-layered iron oxide and iron-chromium oxide spinel scales and markedly changed the morphology of the scale surface and the thickness of the various scale layers. These changes were determined to be affected by the particle size of the erodent, the test temperature and the chromium content of the alloy.

INTRODUCTION

The ability of steel alloy surfaces to withstand the combined degradation modes of erosion and corrosion at elevated temperatures is an important consideration in the design of emerging coal conversion and utilization systems. Processes such as coal gasification and fluidized bed combustion can impose harsh environments on metal containment surfaces. Separate studies of the corrosion and erosion of various alloys in environments partially simulating those that occur in process plants have been underway at this laboratory and several others for the past several years. Investigations of combined erosion-corrosion have also been conducted. The subject is an active research
area at the present time.

High temperature corrosion research in both oxidizing and reducing atmospheres, with and without the presence of sulfur in the corrodant gas to simulate coal reaction products, has determined the nature and rates of formation of oxide and sulfide containing scales on chromium containing steel alloys.[1-8] The effects of important variables such as chromium content of the alloy, exposure temperature and time, and the composition of mixed reactant gases on the morphology, composition distribution and rates of formation of scale products have been determined. The effects of coal char reactant products from coal gasifiers in contact with alloy steels on their elevated temperature corrosion rates and mechanisms have been determined for a number of different char compositions and exposure conditions.[9-11]

The erosion of metals in the absence of corrosion to determine the mechanisms of erosion have provided an understanding of how ductile metal surfaces deform and lose material from their surfaces.[12-15] Separate studies of the erosion mechanisms of the scales formed on the surfaces of metals at elevated temperatures prior to carrying out the erosion tests at room temperature have provided some insight into how brittle scales on ductile metals erode in both thick and thin layers. [16-17] Studies have also been undertaken to determine the behavior of alloys subjected to the combined effects of erosion and corrosion in a single test.[18-22]

Work in the field of erosion and corrosion has progressed to the point where observations of combined behavior can be partially interpreted in terms of the separate mechanisms which occur. In the
work reported in this paper, this background has been used in an attempt to gain a more basic understanding of what is happening and how the characteristics of the alloys tested and the test conditions combine to produce the degradation products that are observed. However, the unexpected results of combined erosion-corrosion reported herein raise many questions that cannot be readily answered from the foundation research that is referred to above. It is hoped that more detailed study and interpretation of the corrosion and erosion phenomena reported in the references and further research will result in more detailed understanding of the behavior that will be discussed below.

The test conditions used are representative of those which occur in the beds and free boards of fluidized bed combustors (FBC). The low velocity, 5 m/s, that was used is typical for a FBC as is the range of particle sizes, 5-100μm dia ave. Most erosion tests [1-8] use considerably higher velocities and generally, larger particle sizes. As such they are not as applicable to FBC tube erosion-corrosion as the investigation reported herein. The compromise that had to be made in this investigation was the test temperature for the alloys tested. While FBC’s reach the test temperatures used, generally higher chromium content alloys are used at the higher test temperatures in FBC's.

TEST DESCRIPTION

The test specimens of each alloy (see Table 1 for composition) were exposed in the downstream duct of a methane-air burner operating with excess oxygen in the combustion gas. The exact partial pressure of
oxygen was not determined. The specimens were 1.25cm on a side cubes with a small hole through their center for mounting on an alumina rod that extended into the 10cm dia. duct perpendicular to its wall. The specimens were exposed so that two of their sides were at 45° angles to the exhaust gas and the other two sides were shielded from direct gas-particle impingement, thus providing two surfaces that were eroded-corroded and two surfaces that were only corroded on the same specimen. The ACES exhaust gas simulator at the Sandia Livermore National Laboratory Combustion Research Center was used for the tests. Figure 1 shows two test cubes in place on the rod with smaller alumina washer spacers between them during a test.

The test conditions were:

gas oxygen rich methane-air combustion gas
velocity of particles 15 m/s
particles 5µm dia coal ash ave
50µm, 100µm dia Al₂O₃ ave
solids loading 6g/min
test duration 30 minutes
impingement angle 45°
 specimen temperature 700°-1000°C
temperature differential across specimen <6°C (measured by optical pyrometer during the test)

After the test exposure the specimens were cooled in less than 1 minute after the gas-particle flow was turned off by natural convection and radiation to temperatures <300°C where further corrosion could not occur, removed and the exposed surfaces and cross sections
microscopically analyzed using a scanning electron microscope (SEM) with KEVEX. Because different mechanisms occurred on the front (combined erosion-corrosion) and on the back (quasi-static corrosion) of each specimen at the same time, it was not possible to determine a degradation rate by either weight or thickness loss measurements.

RESULTS

Corrosion was the dominant mechanism of surface degradation at all of the test conditions investigated. The scales that formed and grew on the metal surfaces were affected by the impacting solid particles during their growth in several ways, depending upon the composition of the base alloy, the test temperature and the size of the erodent particles.

5µm Coal Ash Particles

The results of the tests that used 5µm dia, ave coal ash particles will be presented as a function of the chromium content of the alloys tested, starting with 1018 plain carbon steel with a 0% Cr content. Further, the results will be ordered by the test temperature, starting with the lowest temperature, 700°C. The patterns of behavior of the alloys appeared to follow these two variables in a somewhat orderly manner. The test temperatures, which ranged from 700°C for the 1018 to 1000°C for the 304SS, are generally above the top service temperatures of the various alloys tested. They were selected to achieve oxidation rates which could be readily studied after the short, 30 minute exposures in the burner duct. For all of the alloys tested, corrosion was the dominant mechanism.
Figure 2 shows the surfaces of the corroded and the eroded-corroded sides of a single specimen after exposure to the oxygen rich, methane-air combustion gases and 5\(\mu\)m dia coal ash particles at 700°C. The observed surface on both sides consisted of iron oxide, Fe\(_2\)O\(_3\).\[^6\] The morphology of the scale on the corroded side consisted of a fine network of small needles with nodules of Fe\(_2\)O\(_3\) penetrating up through them. A few coal ash particles from eddys in the flow behind the impingement sides of the specimen were also identified (the larger, light nodules). The eroded-corroded side had the same fine needle network as the corroded side, but the larger Fe\(_2\)O\(_3\) nodules were not present. As can be seen, corrosion was the dominant mechanism on both sides. The fine needles that occurred on the eroded side did not appear to be vulnerable to the impact force of the impinging coal ash particles.

\(\text{1/4Cr1Mo}\)

Figure 3 shows the surfaces of this alloy after the 700°C test. The appearance of the surface is essentially the same as was seen on the 1018 steel specimen. Figure 4 shows the same alloy after the 800°C test. The morphology of both surfaces has changed somewhat. On the corroded side the Fe\(_2\)O\(_3\) needles have formed into an interlaced network with some individual needles of larger diameter also occurring. Very few nodules of Fe\(_2\)O\(_3\) are observed along with a very few coal ash particles (the larger, light grey particles in the lower photo).
On the eroded-corroded side, the morphology is somewhat different. The fine needle network appears to have condensed and a number of quite long comparatively large diameter, individual needles of oxide are seen growing out of the scale. Again, a few coal ash particles can be seen in the lower right photo. The delicacy of the long, individual needles on the eroded-corroded side is quite remarkable, considering that this side is being struck by coal ash particles. Note that the two lower photos are at the same magnification. The dynamic corrosion plus erosion that is occurring on the exposed side appears to be enhancing the growth of the oxide needles at this higher test temperature.

Figure 5 shows the SEM-KEVEX analysis of the surface scale on the eroded-corroded side of the specimen tested at 800°C. It can be seen that only iron oxide is present. The occurrence of only iron oxide on the surface with no chromium was observed for all of the alloys tested, except for the 304SS.

5Cr 1/2Mo

As the chromium content of the alloys increased the morphology of the oxide scale surface changed. The beginning of dramatic differences between the corroded side and the eroded-corroded side began with the 5Cr 1/2Mo Steel. Figure 6 shows the corroded (above) and eroded-corroded (below) surface at two test temperatures, 800°C and 875°C.

At the 800°C test temperature, the needle-like network of the scale can be seen on both the corroded and eroded-corroded sides. A few nodules appear on the corroded side; none on the eroded-corroded side. The same thing occurred on the 1018 and 2 1/4Cr1Mo steels at the lower, 700°C temperature (Figures 2 and 3). It appears that test
temperature and chromium content trade off in the resultant oxide structures. As the chromium content increases, the surface morphology reverts back to that which occurred in the lower chromium content alloys at lower test temperatures.

At the 875°C test temperature, the next major change in the morphology occurred. The needles are almost non-existent on the corroded side (upper right photo) and they are replaced by nodules and columns of Fe₂O₃. The columns are different from the nodules in that their tops appear to be somewhat pointed and porous or wrinkled on the corroded side. The great difference in the morphology between the corroded and eroded-corroded sides first occurs on the eroded-corroded surface of this alloy. It consists entirely of crystalline columns of significantly greater cross section than the pointed-porous topped columns on the corroded side and their tops do not show any porosity. This behavior was observed on the higher chromium content alloys at higher test temperatures to an even greater degree, as will be seen.

9Cr1Mo

Figure 7 shows the extent to which the great difference occurred in the morphology of the 9Cr1Mo steel between the corroded and eroded-corroded sides of the same test specimen. On the left side of the figure is the corroded side with a myriad of small diameter pointed-porous or wrinkled topped columns extending up perpendicular from the metal surface. The few gray globules are coal ash particles. On the right photo is the eroded-corroded surface. The well defined crystalline columns of iron oxide (no chromium was determined) are many
times the size of the columns on the corroded side and are no where near as numerous. Many of their tops have been chiselled away to form more or less points and no porosity or wrinkling can be observed at the tops of the columns. The action of the eroding coal ash particles can be seen to affect individual columns, but do not result in an overall eroding away of the surface. The relatively low density of the columns and their large size seems to reflect a selective thinning of the columns compared to the density of those that occurred on the corroded only side so that the remaining columns could grow to a much larger size. This phenomenon appears to be similar to the selective thinning of trees in a forest to promote the growth of the remaining ones.

The determination of the composition of the columns on both the corroded and eroded-corroded sides of the 9CrlMo steel specimens was made by SEM-KEVEX analysis. Figure 8 shows the analyses of the scales that formed on the 9CrlMo steel. It can be seen that both types of columns are iron oxide with no chromium present. The presence of some coal ash is shown by the presence of such elements as Si, Ca, Al and Mg. The reason that the corroded side has more coal ash present is that the velocity of the particles on the back side of the specimens in the flow eddy is much slower than on the front side that directly receive the impact of the coal ash at the full 5 m/s. The wafting particles on the back side can more readily get caught in the forest of columns that exists there.

The effect of test temperature on the morphology of the scales formed on the 9CrlMo steel are shown in Figure 9. On the left side are shown the surfaces after the 800°C test. Essentially the surface is a
continuous \( \text{Cr}_2\text{O}_3 \) scale with some nodules of \( \text{Fe}_2\text{O}_3 \) and a few coal ash particles on the surface. The eroded-corroded side shows a slightly eroded \( \text{Cr}_2\text{O}_3 \) surface. At this lower temperature, compared to the 950°C exposed specimen shown in Figure 8, the chromium is present in sufficient quantity to form a typical protective barrier-type scale. The eroded-corroded side is still a mild enough overall condition to enable the alloy to maintain the protective scale.

When the test temperature is increased to 950°C (photos on the right side of Figure 9) the same type of behavior as was shown in Figure 7 is evident. In Figure 9 the magnification is higher than in Figure 7 and the nature of the columns can be more readily seen. Also, in the lower right photo another aspect of the erosion process is shown. In some isolated areas the columns had been eroded away, leaving a more continuous scale showing beneath them. The contours appearing on the scale layer is direct evidence of the erosion process. The overall differences between the surface morphologies of both the corroded and eroded-corroded sides of the specimens at the two test temperatures are pronounced.

**410SS**

The 410SS specimens showed the same type of behavior as the lower chromium content, ferritic steels that formed columns. Figure 10 shows the two surfaces after testing at 950°C. The chiselled tops of the eroded-corroded side columns toward pointed shapes are clearly evident as are their greatly increased size over the corroded side columns. Note the difference in magnification at both the lower and the higher magnifications. At least one of the columns (lower right side photo)
is cracked, providing a short circuit diffusion path.

It is possible that the large diameter, dense columns that form on the eroded-corroded side could retard metal loss once they form. The basis for the retardation of metal loss is contained in Reference 16 where it was reported that dense, columnar NiO scale on a nickel substrate eroded at substantially lower rates than equiaxed, porous NiO. On the other hand, the voids between the large columns and the increased diffusion rates in the dense columns could enhance corrosion and scale formation.

Figure 11 shows the SEM-KEVEX peak analyses. Even with 12% Cr content in the alloy, the columns of oxide at the surface were determined to be iron oxide with no chromium. It is interesting to note, however, that the No. 2 peak on the eroded corroded side shows some chromium in a crystallite that appears to be below the general level of the columns. Some coal ash was retained on the corroded side of the specimen, peak 2, where the velocities are very low. The coal ash is composed of oxides of magnesium, aluminum, silicon, potassium and calcium as well as some additional, minor oxides and sulfides. Coal ash is known to be corrosive of itself. The ash particles are slowed down considerably in the wakes on the back sides of the specimens and can get caught in the columns of scale.

The 410SS specimens were cross sectioned to determine the nature of the scale down to the base metal. Figure 12 shows the morphology of the scales and the distribution of iron and chromium through them. Analysis of the photos indicates that combined erosion-corrosion not only changes the surface morphology of the scale compared to straight
corrosion, but also the cross section morphology and composition.

On the corroded side (left side photos) the secondary electron image indicates that there is a duplex scale present with a line of porosity separating the two phases. Analyzing the x-ray maps shows that the top layer is iron oxide with no chromium in it, as has been discussed above. However, the second layer of the scale has substantial chromium content and is either Fe₂O₃ with chromium dissolved in it or an iron-chromium oxide spinel.[6] This layer has a scalloped, intimate bonded interface with the base metal. The cross sections of the small, pointed columns of surface scales described earlier can be readily seen in the upper left photo.

The morphology of the scale cross section on the eroded-corroded side of the specimen (right side photos in Figure 12) is quite different from that on the corroded only surface. The surface of the eroded-corroded side is made up of relatively large diameter columns of Fe₂O₃ with void areas between them that extend down to the iron-chromium oxide layer. It is for this reason that there is a chromium peak in Figure 11 on the eroded-corroded side. The cross sections of the columns on the surfaces of the corroded and eroded-corroded sides of the same specimen verify the observations made earlier concerning the morphological differences between the surfaces of the corroded and eroded-corroded scales.

Referring to the x-ray maps, it can be seen that the scale thicknesses of each of the constituents on the corroded and eroded-corroded sides as well as the distribution of the two phases on each side are markedly different.
On the corroded only side of the specimen, both the Fe$_2$O$_3$ and iron-chromium oxide layers have approximately the same thickness. The contact area between the lower scale layer and the substrate metal is intimate with no voids present. The outer, Fe$_2$O$_3$ scale is much thinner on the erosion-corrosion side, extending only to the base of the columns. The Fe-Cr oxide layer is much thicker on the eroded-corroded side, essentially constituting the main body of the scale. Also, the interface between the lower layer of the duplex scale and the base metal is quite porous on the erosion-corrosion side. The large increase in the iron-chromium oxide layer thickness apparently has depleted the substrate surface in chromium sufficiently to cause some Kirkendall type voids to occur at the scale-metal interface. The amount of voids between the upper and lower scales is near the same for both sides. Thus the occurrence of erosion in conjunction with corrosion, with the corrosion process dominant, changed the morphology and the composition distribution of the scale that was formed.

**304SS**

The 304SS, an austenitic alloy with a significantly higher chromium content than any of the chromium containing ferritic alloys tested, behaved in a somewhat different manner than the other steels at temperatures higher than were used to test the other steels but still using 5μm coal ash particles. Figure 13 shows that the surfaces developed a continuous Cr$_2$O$_3$ scale on both the corroded and eroded-corroded surfaces and that the barrier scale was not penetrated by the eroding particles.
On both the corroded and eroded-corroded surfaces, nodules of iron-chromium spinel have penetrated up through the Cr$_2$O$_3$ scale layer. The size of the nodules is considerably larger on the erosion-corrosion surface than on the corroded surface. This follows the general trend throughout the test series of having combined erosion-corrosion enhance the growth of individual crystallites on the scale outer surface. Some of the nodules present on the surface are coal ash.

Laser Raman spectroscopy was used to identify the phases that were present on the surface of the 304SS test samples. Figure 14 shows the Raman spectra for the corroded surface. The peak at 570 is Cr$_2$O$_3$, the peak at 660 is spinel and the small peak at 1010 represents the compounds in coal ash. Figure 15 shows the Raman spectra of an area on the surface of the eroded-corroded side. The additional peaks over and above those that occurred in Figure 14 are due to coal ash particles adhering to the surface. These spectra indicate that individual nodules of iron-chromium oxide spinel formed on the iron-chromium-nickel 304SS rather than the iron oxide nodules that formed on the straight iron-chromium alloys.

**Effect of Particle Size**

In order to determine what type of scale morphology would occur on the eroded-corroded surfaces when larger eroding particles were used which could transfer a greater force to the surface upon impact, 50µm and 100µm Al$_2$O$_3$ particles were used to erode 410SS. All of the other test conditions were the same as were used in the 5µm coal ash tests. It was thought that while the small, 5µm coal ash particles used in the earlier tests were too small to result in erosion being the dominant
mechanism, the larger particles could change that circumstance.

As the particle size of the erodent increased, major changes occurred in the morphology of the iron oxide scale surface. Figure 16 shows the surfaces of the erosion-corrosion side of 410SS eroded by 5μm flyash and 50μm and 100μm Al₂O₃. As the particle size increased from 5μm to 50μm diameter particles, the diameter of the iron oxide columns decreased and the number increased. The type of column caused by the 50μm particles reverts to that which occurred on the corrosion side of the specimens in the 5μm flyash test, Figure 10. A further increase in particle size to 100μm diameter Al₂O₃ caused a further major change in the topography of the scale surface, as can be seen in the lower photo in Figure 16. There are a minimum number of small nodules rising out of a cracked continuous iron oxide scale layer.

The variation in surface morphologies as a function of erodent particle size followed a pattern found in all of the other chromium containing steels tested. For example, Figure 17 shows the effect of erodent particle size on the surface scale morphology of the 5Cr 1/2Mo steel. Note that the magnification of the scale on the 100μm Al₂O₃ is one-third that of the two photos above it. A major change in the diameter and shape of the needles or blades formed on the erosion-corrosion surface occurred between the 5μm flyash and the 100μm Al₂O₃ tests. The 50μm diameter particles resulted in a surface morphology that was similar to that found on the 410SS when it was eroded with 50μm diameter particles, see Figure 16. This small diameter column morphology was found on almost all of the alloys that were eroded with 50μm diameter particles.
The cross sections of scales on two alloys with significantly different chromium contents from tests using 100μm diameter Al₂O₃ particles are shown in Figures 18 and 19. The scales were still multi-layered but differed significantly from those which occurred when 5μm flyash was used. It can be seen that the overall thickness of the scale on the eroded-corroded side of both the 2 1/4 Cr1Mo steel (Figure 18) and 410SS (Figure 19) specimens is much greater than that which formed on the corrosion only side of the specimens (note the differences in magnification of the photos). The erosion process markedly enhanced the growth of the scale.

Figure 18 shows the cross sections of the scales formed on the much lower chromium content 2 1/4 Cr1Mo steel when 100μm particles were used at a lower test temperature than was used for the 410SS. The scale is essentially iron oxide. The chromium has concentrated in a thin iron-chromium oxide spinel layer at the scale-metal interface. The scale on the eroded-corroded side is significantly thicker than that on the corroded side, as occurred on 410SS. The scales that formed on both sides of the specimen were almost completely separated from the substrate, indicating a strong susceptibility to spalling.

The distribution of the iron oxide and iron-chromium oxide scale layers on the 410SS was changed between the eroded-corroded scale in the 5μm flyash test, Figure 12, and the 100μm Al₂O₃ test shown in Figure 19. The iron oxide, upper layer, is now the thicker layer with not as much iron-chromium oxide spinel forming, relatively, as occurred in the 5μm flyash test. Small diameter voids occurred throughout the scale layers on the eroded-corroded side but did not occur on the much
thinner scale on the corroded only side.

There is also an internal scale layer that has formed on the eroded-corroded side, below the voids at the scale-metal interface that does not contain chromium. Based on evidence in Reference 7, it is probably inward growing Fe$_3$O$_4$. The much smoother surface of the 100µm particle test scale compared to the 5µm particle test scale is also indicated in Figure 19.

The consistent effect observed on all of the alloys for the eroding particles to increase the scale thickness on the erosion-corrosion side of the test specimen compared to the corrosion only side was checked against the dynamic corrosion obtained by testing specimens in the flowing gases of the ACES burner without injecting any particles. Figure 20 compares the scales formed and their thicknesses for a number of test conditions. It can be seen by comparing the top two photos that static corrosion on the back side of the specimen and dynamic corrosion on the front side (gas flow without particles) results in nearly the same scale thickness with approximately a 10% increase on the dynamic corrosion side. This compares with a 60% increase in the scale thickness on the erosion-corrosion side when 5µm particles are used and a 345% increase when 100µm particles are used, all compared to the static corrosion only side scale thickness.

Cross sections of the erosion-corrosion scales are seen in the lower two photos in Figure 20. While the morphology of the static and dynamic corrosion scales are similar, there is a major difference in the two eroded-corroded scales in addition to their thickness difference. This seemingly anomalous behavior of the more erosive
(larger size) particles causing markedly thicker scales to form was consistently observed for all of the chromium containing steels tested.

The differences in the morphology of the cross section and surface of three of the chromium containing steels tested at the same conditions are shown in Figure 21. These steels were eroded by 100μm \( \text{Al}_2\text{O}_3 \) particles. All three had iron oxide outer scales with the two higher chromium content steels having sizeable inner scales of iron-chromium oxide spinel.

The character of the surface of the outer scale differed for the three steels. The large, light particles on the surface of the 2 1/4Cr1Mo steel are alumina erodent particles that became embedded in the iron oxide scale. Note the difference in magnification between the three photos on the right side of Figure 21. The rosettes of iron oxide that formed on the 9Cr and 12Cr steels are completely different from the iron oxide morphology on the 2 1/4Cr1Mo steel. The rosettes on the 410 steel are much larger than those which formed on the 9Cr1Mo steel (note the difference in magnification between the two photos). Similar rosettes of oxide scale are shown in Reference 8.

In contrast with the differences in the eroded-corroded surfaces that were shown in Figure 21 when the erodent was 100μm \( \text{Al}_2\text{O}_3 \) the use of 50μm \( \text{Al}_2\text{O}_3 \) erodent particles resulted in a very similar surface scale morphology for the three alloys of varying chromium content shown in Figure 22. The columns of iron oxide with wrinkled and porous tops are similar in size and shape to the corroded only side of the 410SS specimen that was tested with 5μm flyash, see Figure 10. The scale morphologies of the three alloys shown in Figure 22 are completely
different from their eroded-corroded surfaces when tested using either 5µm or 100µm size particles, see Figure 10 and 21. The cross sections of the three scales are multi-layered and have generally similar morphologies. The scale thickness of the 9Cr and 12Cr steels was thinner when the 50µm particles were used than when the 100µm particles were used as can be seen by comparing Figures 21 and 22 (note magnification differences).

**Effect of Temperature**

The morphology of the surface iron oxide scale was also affected by the test temperature when 5µm flyash and 100µm Al₂O₃ erodents were used, but not when 50µm Al₂O₃ particles were used. Figure 23 shows the changes that occurred on the 9Cr1Mo steel specimens when the temperature was changed from 800°C to 950°C and 100µm size particles were used. The large diameter needles or blades that grew at 950°C were very similar to those which grew at 800°C on the 5Cr1/2Mo steel shown in Figure 17. The increase in the size of the microstructural elements with increasing temperature was expected, but the delicacy of some of the iron oxide crystals on surfaces that were being impacted by 100µm size particles as they were growing was unexpected.

Figure 24 shows the surfaces of the same alloy, 9Cr1Mo steel, at three different test temperatures when 50µm Al₂O₃ particles impacted the surface. As in the case of varying the chromium content shown in Figure 22, varying the test temperature resulted in essentially the same type of microstructure for all three temperatures. The morphology was the same wrinkled, porous topped columns that were shown in Figure 22.
The effect of test temperature on the surface morphology of the alloys tested with the 5μm flyash particles was generally more dramatic in the differences that occurred. Figure 25 shows the surface of 410SS tested at 850° and 950°C on both the corroded and eroded-corroded surfaces. At the lower test temperature on the corroded side, some of the needle type crystals observed on the surface of the lower chromium content steel occurred. On the eroded-corroded side at the 850° test temperature, the needles have been removed and the remaining structure is essentially unaltered. Increasing the test temperature by 100°C markedly changed the morphology of the surface of the iron oxide on both the corroded and eroded-corroded surfaces as can be seen on the right side photos in Figure 25.

DISCUSSION

Qualifications

The major changes in the surface and cross section morphologies and compositions of the iron oxide base scales that formed on the chromium containing steels were unexpected. While there is some work in the literature that analyzes the growth of iron oxide and iron-chromium oxide spinels on the surfaces of chromium containing alloys, [6,8] the gross changes in morphology that were observed in this work, especially those that were promoted by the erosion process, have not been reported previously. Studying the high temperature corrosion literature referenced in the introduction only begins to account for the behavior reported therein.

Much work of interpretation and determination of mechanisms of the scales that formed must still be done. Hence, this discussion of the
observed behavior will not be in depth at this time. Primarily, the work to date has identified phenomena that require more work to explain adequately.

The predominance of corrosion over erosion on the surfaces of ferritic steels with chromium contents up to 12% is due, at least in part, to the relatively high test temperatures used. These temperatures were selected to assure that scale would form at a rate that was compatible with a reasonable test time. Running the ACES burner at lower temperatures for extended periods of time was not technically or economically feasible. Generally, the test temperatures used were above the recommended service temperatures for the alloys tested. The erodent particle sizes and test velocities were representative of those which occur in coal gasification processes.

The dominance of the corrosion process at all conditions of temperature, alloy composition, particle size and other erosion conditions was unexpected. It was thought that when at least the 100μm Al₂O₃ particles were used, the erosion process would be able to sweep oxide off the surface as it formed. The mechanism of elevated temperature corrosion when the oxidizing surface is being mechanically affected during the process by impacting particles must follow different rate laws than those that have been determined to be applicable to static corrosion. Parabolic and linear rates of corrosion are probably replaced by accelerating rates of some type which are not definable at this time.

One of the problems that is associated with determining the kinetics of the corrosion process is the inability to precisely measure
the metal surface regression rate. The best kinetic work in straight corrosion is carried out using weight change measurements. In the present case where different reactions occur on the front and back surfaces of the same specimen weight change measurements are not appropriate. Efforts are underway to develop an alternative method to measure the rates in a precise enough manner to use for kinetic analysis. The most promising method tested so far involves bonding of a thin strip of platinum on the surface with a Sauereisen cement to protect a portion of the exposed surface. After the test, a cross section of the surface through the protected area shows a difference in metal thickness between the protected and unprotected areas.

A pattern developed in the test series using 5μm coal ash particles that somewhat related the scale morphology to the test temperature, chromium content of the alloy, and whether near static corrosion or combined erosion-corrosion was occurring. Its meaning, if indeed there is one, is not known. As the chromium content, the test temperature, and the severity of the surface environment (corrosion compared to erosion-corrosion) increased, the dominant shape of the iron oxide particles on the surface progressed from nodules to needles to columns. A less than thorough review of the literature failed to shed light on why this pattern occurred.

**Erosion-Corrosion Mechanism**

The occurrence of Fe₂O₃, free of chromium, as the outer scale of a duplex or multi-layer scale structure with iron-chromium oxide spinel beneath the α-Fe₂O₃ relates to several factors. Among them are the
partial pressure of oxygen at the scale-gas interface compared to that nearer the scale-metal interface, the short growth time of the scales, the difference in the outward diffusion rates of iron and chromium and their diffusion paths. Its occurrence on the higher chromium steels in this study as well as on the low chromium steels differs from the work reported in Reference 8. However, the meticulous observations by Mitchell and his co-workers of the nucleation and growth of oxides on chromium containing steels in the temperature range 400°-950° C in Reference 7, 8 and in their other work promises to be a valuable aid in the effort that is being carried out to gain an understanding of how and why the scales reported on herein occurred in the manners that were observed.

The formation of needles, columns and rosettes or nodules of iron oxide on the surface is due to the manner in which iron diffuses upward and outward from oxide nuclei which form early in the oxidation process. It has been observed that the formations of needles are single crystals of $\alpha$-$Fe_2O_3$ as reported in Reference 6. They grow from the tip upward as the result of fast diffusion paths along both their outside surfaces, and along the inner surface of tunnel walls that extend up the columns toward the tip. These surface short circuit diffusion paths are reported to be up to 9 times faster for iron than iron bulk diffusion in the $\alpha$-$Fe_2O_3$ and 4 times faster than grain boundary diffusion.[6]

In Reference 7 Mitchell and co-workers describe the manner in which the oxides nucleate and grow in low (3%) and high (18%) chromium steels. He has postulated that the nuclei of the growth oxide crystals
precipitate between a thin oxide film of uniform thickness that pre­
exists on the metal surface itself. For low chromium content alloys,
the nuclei grow upward and outward to become the iron oxide needles or
columns observed herein. Rosettes are also formed such as those seen
in Figure 21. Similar rosettes are shown in Reference 8. For higher
chromium content alloys, 9, 12, 18% Cr were reported, the nuclei are
iron-chromium spinels and they grow inward.

The proposed mechanism of growth for the iron oxide is diagramed
in Reference 7. It entails surface diffusion of iron along the pre­
existing oxide film to the nuclei sites. Once the nuclei have reached
a critical size, no more nuclei are precipitated and the ones that are
present grow laterally as well as upward until they impinge on adjacent
ones. The keys to the formation of the nuclei which become the needles
and columns of oxide appear to be the fast diffusion path of iron atoms
along the interface of the pre-existing oxide and the protected
location for nucleation between the pre-existing oxide film and the
base metal.

Translating the mechanism in Reference 7 to this study, on the
corrosion side the pre-existing film remains relatively intact and the
iron can rapidly diffuse along it to many protected nuclei growth
locations between the film and the base metal. Hence the columns
formed are relatively small in diameter and many in number. On the
erosion-corrosion side, the pre-existing oxide film is broken up by the
early impacting erodent particles, permitting nucleation of oxide
precipitates at relatively few sites where the oxide film was not
struck and is still intact. These fewer nuclei can then grow to much
larger diameter columns before they impinge on adjacent crystals. This mechanism could be the basis for the large differences between the size of the columns on the corroded side and on the eroded-corroded side of the test specimens.

For higher chromium content alloys, Mitchell included 9, 12 and 18% Cr steels in Reference 7, inward growing iron-chromium oxide spinels are nucleated by the mechanism discussed above. While the study reported on herein found that the outer scale layer was always iron oxide, inner scales of iron-chromium oxide spinels were observed. These spinels, as reported in Reference 7 and 8 could be either \((\text{Fe,Cr})\) \((\text{Fe,Cr})\)_3O_4 or \(\alpha-(\text{Fe,Cr})_2O_3\). The light and dark areas shown in the inner, spinel scale layer for the 410SS in Figure 21 could be evidence that both spinels were present.

**Scale Morphology, Thickness, Composition Differences**

The significant increase in the thickness of the scale on the erosion-corrosion side of the test specimens compared to the corroded only side and the change in the distribution of the iron oxide and the spinel thicknesses between the corroded and eroded-corroded sides cannot be explained yet. They were a consistently occurring phenomena for all of the alloys tested with the degree depending on the composition of the alloy and the size of the eroding particles. The effects increased with increasing erodent particle size. Figure 12 shows a comparatively minor difference for 410SS when 5μm flyash was the erodent while Figure 19 shows a major difference when 100μm Al₂O₃ particles were used. An intermediate difference in the thickness is shown for the 2 1/4Cr1Mo steel in Figure 18. Also, the scales on the
eroded-corroded side were thicker when 100µm erodent particles were used than when smaller particles were used. Compare Figures 21 and 22 for the 9Cr and 12Cr steels.

Dynamic corrosion tests were run on all of the test alloys and the results on 410SS, which are typical, are shown in Figure 20. The difference in scale thickness between the corroded and eroded-corroded sides of the same specimen was determined to not be due to dynamic corrosion effects. The ability of the impacting erodent particles to increase the growth rate of the oxide scale without removing it was demonstrated.

In the presence of chromium, the formation of Fe(1-x)O at the metal interface is suppressed and, instead Fe3O4 forms.\[7\] In Figure 19, beneath the iron-chromium spinel scale and next to the base metal, another layer of scale can be seen that does not contain any chromium and, hence, is probably Fe3O4. The Fe3O4 that forms in the nuclei that grow into needles or columns is transformed to α-Fe2O3 sometime during the growth process.\[7, 8\]

The description of the formation of a multi-layer scale on Fe-3% Cr steel by Mitchell in Reference 8 matches perfectly with the scale on 2 1/4Cr1Mo steel on the corroded side shown in Figure 18. He described an inner, thin layer of (Fe,Cr)3O4 spinel, a thicker, compact middle layer of Fe3O4 and a thin outer layer of α-Fe2O3. Since positive identification of each scale layer was made by Mitchell and he identified a distinct layer of Fe3O4, it is assumed that layers of Fe3O4 are present on some of the other lower chromium content steels, in addition to the scale shown in Figure 18, that are reported on.

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herein. To date, only SEM X-ray maps of the principal elements present have been used in this study to aid in the identification of scales.

In Reference 8 Mitchell reported the occurrence of considerable porosity in the oxide scales. He observed that more and smaller diameter pores occurred in the higher chromium content alloys. The same observation was made in this study, but primarily in the scale on the erosion-corrosion side of the specimens. Compare Figure 19 of 410SS with Figure 18 of 2 1/4Cr1Mo steel.

The effect of the test temperature on the surface morphology of the scales on the erosion-corrosion sides of the specimens was very pronounced when 5μm and 100μm particles were used as the erodent, Figures 25 and 23, but caused very little change when 50μm particles were used, Figure 24. The reason for this apparent anomaly is not known.

CONCLUSIONS

1. Corrosion is the dominant mechanism at all test conditions in the erosion-corrosion of chromium containing steels at temperatures from 700⁰-1000⁰C.

2. The erosion process enhances the growth of oxide scales.

3. The erosion process markedly changes the morphology and composition distribution of the oxide scales.

4. The morphology of the surface oxides are changed as the erodent particle size and test temperatures are changed.

5. As the test temperature and the chromium content of the alloys increased, the dominant shape of the iron oxide particles on the surface of the scale progressed from nodules to needles to columns. The reason for this and its meaning are not known.
6. Iron oxide ($\alpha$-Fe$_2$O$_3$) forms as the outer scale in a multi-layer scale on all of the chromium containing steels tested. The inner scale layers consist of iron-chromium oxide spinels and Fe$_3$O$_4$, depending on the chromium content of the alloy.

7. Dynamic corrosion is not a contributor to the increase in the scale thickness on the erosion-corrosion sides of the test specimens, compared to the corrosion side.

8. Larger erodent particles result in thicker scales.

9. The eroded scale surface became smoother as the erodent particle size increased.

10. Additional studies must be performed to gain a clearer understanding of the mechanisms involved.

ACKNOWLEDGEMENT


REFERENCES


FIGURES

1. Two cube shaped specimens in burner duct during testing separated by circular alumina washers. CBB 834-3754

2. Corroded and eroded-corroded surfaces of 1018 steel at 700°C. XBB 833-1825

3. Corroded and eroded-corroded surfaces of 2 1/4CrlMo steel at 700°C. XBB 833-1824

4. Corroded and eroded-corroded surfaces of 2 1/4CrlMo steel at 800°C. XBB 8210-9326

5. SEM-KEVEX analysis of scale surface on eroded-corroded side of the 2 1/4CrlMo steel at 800°C. XBB 837-6076

6. Corroded and eroded-corroded surfaces of 5Cr1/2Mo steel at 800°C and 875°C. XBB 831 590

7. Corroded and eroded-corroded surfaces of 9Cr1Mo steel at 925°C. XBB 820 9329

8. SEM-KEVEX analyses of the oxide column composition of the 9Cr1Mo steel at 950°C. XBB 837 6078

9. Corroded and eroded-corroded surfaces of 9Cr1Mo steel at 800°C and 950°C. XBB 833 1822

10. Corroded and eroded-corroded surfaces of 410SS at 950°C. XBB 820 9325

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20. Cross section of scales formed on 410SS at 950°C at different test conditions. XBB 841 97

21. Cross section and surface morphology of scales of 2 1/4, 9 and 12Cr steels eroded-corroded at 850°C using 100μm Al2O3. XBB 841 96

22. Cross section and surface morphology of scales of 5, 9 and 12Cr steels eroded-corroded at 950°C using 50μm Al2O3. XBB 841 105

23. Surface morphology of eroded-corroded 9Cr1Mo steel at three different test temperatures using 100μm Al2O3. XBB 841 106

24. Surface morphology of eroded-corroded 9Cr1Mo steel at three different test temperatures using 50μm Al2O3. XBB 841 104

25. Surface morphology of eroded-corroded 410SS steel at two test temperatures using 5μm flyash. XBB 841 107
TABLE 1

ALLOY COMPOSITION (NOMINAL)

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<tr>
<th>ALLOY</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Si</th>
<th>Mn</th>
<th>C</th>
<th>P/S max</th>
<th>Fe</th>
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<td>1018</td>
<td>0.5</td>
<td>0.2</td>
<td>0.09</td>
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<td></td>
<td></td>
<td></td>
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<td>1 1/4Cr</td>
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<td>0.3</td>
<td>0.4</td>
<td>0.2</td>
<td>0.02</td>
<td>bal</td>
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<tr>
<td>1Mo</td>
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<td>0.02</td>
<td>0.5</td>
<td>0.1</td>
<td>0.02</td>
<td>bal</td>
<td></td>
<td></td>
</tr>
<tr>
<td>5Cr</td>
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<td>0.02</td>
<td>0.5</td>
<td>0.1</td>
<td>0.02</td>
<td>bal</td>
<td></td>
</tr>
<tr>
<td>1/2Mo</td>
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<td>0.5</td>
<td>0.5</td>
<td>0.15</td>
<td>0.02</td>
<td>bal</td>
<td></td>
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<tr>
<td>9Cr 1Mo</td>
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<td>1.0</td>
<td>0.2</td>
<td>0.1</td>
<td>bal</td>
<td></td>
<td></td>
</tr>
<tr>
<td>410SS</td>
<td>18</td>
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<td>1.0</td>
<td>2.0</td>
<td>0.1</td>
<td>0.1</td>
<td>bal</td>
<td></td>
</tr>
<tr>
<td>304SS</td>
<td>18</td>
<td>9</td>
<td>1.0</td>
<td>2.0</td>
<td>0.1</td>
<td>0.1</td>
<td>bal</td>
<td></td>
</tr>
</tbody>
</table>
Fig. 1. Two cube shaped specimens in burner duct during testing separated by alumina washers.
Fig. 2. Corroded and eroded-corroded surfaces of 1018 steel at 700°C.
Fig. 3. Corroded and eroded-corroded surfaces of 2 1/4 Cr 1Mo steel at 700°C
Fig. 4. Corroded and eroded-corroded surfaces of 2 1/4 Cr 1Mo steel at 800°C

2 1/4 Cr 1 Mo Steel
Erosion Tester
3.6 ml/s
Methane-air
Temp. = 800°C
Vel. = 5 m/s
Time = 10 min

XBB 8210-9326
Fig. 5. SEM-KEVEX analysis of scale surface on eroded-corroded side of the 2 1/4 Cr 1Mo steel at 800°C
Fig. 6. Corroded and eroded-corroded surfaces of 5 Cr 1/2Mo steel at 800°C and 875°C
Fig. 7. Corroded and eroded-corroded surfaces of 9Cr1Mo steel at 925°C

9 Cr 1 Mo Steel

Emersion Tester: Temp. = 925°C
5μm flyash: Vel. = .5m/s
Methane-air: α = 45°
Time = 30min.

XBB 820-9329
Fig. 8. SEM-KEVEX analyses of the oxide column composition of the 9 Cr 1Mo steel at 950°C
Fig. 9. Corroded and eroded-corroded surfaces of 9 Cr 1Mo steel at 800°C and 950°C.
Fig. 10. Corroded and eroded-corroded surfaces of 410SS at 950°C

410 SS
Erosion Tester
S.m flyash
Methane-air
Temp. = 950°C
Vel. = 5m/s
α = 45°
Time = 30min

XBB 820-9325
Fig. 11. SEM-KEVEX analyses of columns of scale on 410SS at 950°C
Fig. 12. Cross section of corroded and eroded-corroded surfaces of 410SS at 950°C
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Fig. 14. Raman spectra of corroded surface of 304SS at 1000°C
Fig. 15. Raman spectra of eroded-corroded surface of 304SS at 1000°C
Fig. 16. Effect of eroding particle size on the surface morphology of the eroded-corroded surface of 410SS at 950°C

XBB 837-6248

410 SS
Emersion Tester
Erosion-Corrosion
Methane air
Temp. = 950°C
Vel. = 50/s
θ = 45°
Time = 30 min.
Fig. 17. Morphology of surface scale on 5Cr1/2Mo steel eroded by 5μm, 50μm, 100μm particles.
Fig. 18. Cross section of scales on 2 1/4 Cr1Mo steel from 100μm Al₂O₃ tests at 850°C.
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Fig. 20. Cross section of scales formed on 410SS at 950°C at different test conditions.
Fig. 21. Cross section and surface morphology of scales of 2 1/4, 9 and 12Cr steels eroded-corroded at 850°C using 100μm Al₂O₃.

XBB 841-96
Fig. 22. Cross section and surface morphology of scales of 5, 9 and 12Cr steels eroded-corroded at 950°C using 50μm Al₂O₃.

Emersion Tester
Erosion-Corrosion
Methane air

Temp. = 950°C
Vel. = 15m/s
α = 45°
Time = 30min.

XBB 841-105
Fig. 23. Surface morphology of eroded-corroded 9Cr1Mo steel at three different test temperatures using 100μm Al₂O₃.
Temp. = 850°C

Temp. = 900°C

Temp. = 950°C

Fig. 24. Surface morphology of eroded-corroded 9Cr1Mo steel at three different test temperatures using 50μm Al₂O₃.

XBB 841-104
Fig. 25. Surface morphology of eroded-corroded 410SS at two test temperatures using 5μm flyash.

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