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January 1985

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ELEVATED TEMPERATURE EROSION CORROSION OF 9CRI MO STEEL

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ABSTRACT

The combined erosion-corrosion behavior of 9Cr1Mo steel was determined at 850°C using a blast of air and 130µm dia Al₂O₃ particles from a nozzle at a number of velocities from 10-70 m/s. The mechanism of surface degradation and the rates of sound metal loss were determined. Corrosion occurred at all velocities tested. It was established that the sound metal loss increased with increasing velocity and that there was a change in the loss mechanism at about 30 m/s. Below this velocity, scale loss occurred by an erosion enhanced corrosion mechanism. Above 30 m/s the impacting particles consolidated and densified the scale, eliminating the micro-cracks that occurred at the lower velocities. Elimination of the micro-cracks increased the stresses in the scale, resulting in periodic spalling of larger pieces of scale. The spalling increased the sound metal loss rates at the higher velocities, resulting in a classic transition rate "S" curve.

INTRODUCTION

The impact of small, solid particles on the containment surfaces of such process equipment as entrained coal gasifiers and fluidized bed combustors can result in unacceptable levels of surface degradation by combined erosion-corrosion.¹,² The purpose of this investigation was to study the erosion-corrosion mechanism of a chromium containing steel that is used in this type of equipment. A laboratory scale, nozzle type erosion tester was used that can operate at test temperatures up to 900°C using a number of different particles, carrier gases and operating conditions.
Test conditions were selected that simulated portions of the operating environments which can occur in the equipment used to convert or utilize energy from pulverized coal. The alloy selected for testing was 9Cr1Mo steel, a commercial alloy that is used in elevated temperature process equipment. It was selected because it contains enough Cr to form a protective Cr\(_2\)O\(_3\) scale layer at some test conditions, but not enough to form a protective scale layer at other conditions. By using such a marginal chromium content alloy at one elevated temperature, 850°C, and varying the gas-solid particle stream velocity, it was possible to observe the role that erosive particle impacts had on the scale formation, adherence, and performance as an erosion-corrosion barrier. The 850°C test temperature is above those for which 9Cr1Mo steel is normally used in process equipment in order to obtain surface degradation rates that occurred within a reasonable test time. Test temperatures below 850°C did not have reproducibly measurable metal recession rates for the maximum exposure time used.

**TEST CONDITIONS**

All of the experiments were carried out in the elevated temperature erosion tester shown in Figure 1. It can use a variety of erodents and either air, argon or nitrogen carrier gases. Temperatures from 20°C to 900°C are achievable with a temperature variation not exceeding 15°C over the test range. 850°C was used in the investigation. Particle velocities from 10 to 70 m/s were used at a solids loading of 2.5 g/min. Particle velocities were established by setting a pressure drop across the nozzle using a metering system that was connected to a shop air supply. The air pressures were determined
using a computer calculation developed in Reference 5 to take elevated temperatures into account.

Test times of 30 min and 5 hr were used in this study. The erosion of the steel reported herein was carried out at an impingement angle of $\alpha = 90^\circ$. Studies were also carried out at $\alpha = 30^\circ$, they will be reported in a subsequent report. The erodent particles were angular shaped, 100-150µm, Al$_2$O$_3$ with an average particle size of 130µm.

Thickness changes of the base metal as the result of combined erosion-corrosion were made by measuring the cross section of the metal after testing using an optical micrometer observing a cross section through the primary erosion zone. The back side of each specimen was protected from the environment by sitting on a solid surface cradle. Even so, a temper color thin oxide scale that was not measurable did occur on the protected side of the specimen.

The 9Cr1Mo steel used was cut from a 1" thick, 5" ID pipe. Its composition was nominally 0.15C, 0.75Si, 0.45Mn 9Cr, 1.0Mo, balance Fe. It was flattened in a rolls and annealed at 925°C. Specimens whose final size was 17.5 X 17.5 X 2mm were milled and polished to a 600 grit finish.

To prevent oxidation of the test surface prior to the test, nitrogen was passed through the erosion tester until the specimen reached the test temperature. After the test, the specimen was quickly removed from the furnace section of the tester and placed under a protective flow of nitrogen until it had cooled to approximately 300°C to prevent further oxidation. Some spalling of the scale on the test surface occurred during cooling. Optical and scanning electron
microscopes (SEM) were used to observe the specimens' surface and cross sections. KEVEX and x-ray diffraction were used to determine the compositions of the scales.

RESULTS

1. Scale appearance and Morphology

The scale formation that occurred on the 9Cr-1Mo steel after a 5 hour exposure at lower test temperatures is shown in Figure 2. At 250°C a very thin scale occurred with some localized, small craters. At 450°C, a more substantial scale formed with some formation of Fe₂O₃ nodules. However, this scale was still too thin to be studied effectively. At 650°C, the typical Fe, Cr oxide spinel scale developed, but it was still too thin to measure its thickness. It was only at temperatures of 750°C and above that a 5 hour exposure to the eroding Al₂O₃ particles in the air stream produced a metal loss and scale that could be studied as a function of test variables. 850°C was used to assure that enough metal recession occurred to measure at all test velocities.

Figure 3 shows the patterns of erosion-corrosion that occurred at the three upper test temperatures over the range of velocities that were used. The left hand column shows the appearance of the test surface when it was exposed to the air blast only, without any particles in it. The pattern consisted of three areas, a primary erosion zone, a secondary or halo zone and an outer zone where a few particles struck. The distribution of the flow of particles and gas out of the one-dimensional flow nozzle are discussed in Reference 6.

It can be seen that as the conditions got more severe, the upper right
hand corner of Figure 3, more of the specimen surface was affected.

The morphology of each of the three eroded areas is shown in Figure 4 along with KEVEX peaks identifying the principal elements that are present. The areas selected had no evidence of major spallation occurring. In the primary zone, the scale was relatively dense and had some regions of small size nodular scale occurring on top of the base scale. The KEVEX peak indicates that the scale was Fe₂O₃ with some embedded Al₂O₃ erodent material in it. The absence of chromium in the top layer is consistent with observations reported in Reference 7 and 8 for tests performed at lower velocity in a different type of tester.

The middle photo shows the scale in the halo zone. It is different from that in the primary zone in that it does not appear to be as dense and has some distinct, relatively large nodules in the outer layer. Comparing the principal scale layer in the primary zone in the top photo with the scale layer in the halo zone in the middle photo, it appears that the higher mass loading of particles in the primary zone densified the scale as they impacted the surface and prevented large nodules from forming. Some of the large nodules that formed in the halo zone appeared to have been broken off after they reached a critical condition, leaving pits in the scale beneath them. In the deeper pits beneath the top layer in the halo zone some chromium begins to show up in the KEVEX peak analysis. This occurrence also ties in with observations made in the investigations reported in Reference 7 and 8 where Fe, Cr oxide spinels occurred in the scale layer below the top layer and nearer the scale-metal interface.
In the outer zone where only a few particles impacted the surface, the scale is very thin and smooth with a considerable presence of chromium, as indicated by the KEVEX peak analysis. The KEVEX peak may include the base metal composition as the scale is very thin. However, Figure 2 does show visible evidence that some erosion-corrosion had occurred.

In other regions of the primary and halo zone, spalling of layers of the scale occurred during the test exposure and cooling down period. Figure 5 shows an area where both adhered and spalled scale occurred. There is a pattern on the spalled area that is seen at the higher magnification in the lower photo to be a grain pattern in the scale that can be related to the base metal grain size, as can be seen in Figure 6.

The nature of the scale formed on eroding-corroding surfaces near the end of a test when the overscale has spalled off in a sudden occurrence is shown in Figure 6 and 7. Figure 6 shows the eroded-corroded surface and the cross section of the alloy directly beneath the eroded surface. It can be seen that the scale forms in patterns on the surface that outline the grains of the alloy. The scale that forms in the grain boundaries has a different shading than that which forms on the grains and appears to be more susceptible to erosion. This results in preferential erosion of the scale immediately above the grain boundaries of the base metal and can be considered to be an erosion etching process.

Figure 7 shows a lower magnification view of the erosion etched surface and a high magnification view of the scale above and near the
grain boundary of the base metal. The size of the small oxide scale crystals can be seen in the lower photo, as differentiated from the base metals' grain size. The KEVEX peak analyses in Figure 7 show that the scale above the metals' grain boundaries has a considerable higher Cr content than that which forms over the grains. The oxide crystal size appears to be similar for both oxides.

Evidence that the spalling took place prior to the end of the test is shown in Figure 8. The upper photo shows the scale that formed after the spalling had occurred. The lower photo shows a scale area in the outer region of the specimen where spalling had not occurred. The oxide grain size in the outer area is larger than that which formed in a spalled area of the primary zone near the end of the test because the grains had a longer time to grow in the outer region where they were continuously exposed to the air atmosphere.

2. Erosion Rates

Erosion rates were determined by measuring the remaining sound metal thickness after the erosion exposure. The micrometer stage on an optical microscope was used for these measurements. The amount of thickness reduction after the 30 min. tests was not sufficient to obtain reproduceable measurements so 5 hour exposures were made which did result in reasonably consistent measurements. Two or three five hour tests were run for each of a range of particle velocities. Table 1 lists the before and after measurements. It can be seen that the presence of the erodent particles increased the thickness losses compared to the dynamic corrosion tests run without particles (tests 1 and 2.)
Figure 9 plots the erosion of the 9Cr1Mo steel as a function of the particle velocity. The 0 velocity data points are those for dynamic corrosion with no particles in the gas. There is a distinct division between the low velocity particle flows and the higher velocity flows in the amount of thickness loss which occurred. Two regimes of thickness loss occurred. All of the specimens tested formed a scale on the surface as will be presented the next section. Therefore, the division in erosion regimes does not specifically relate to corrosion dominated and erosion dominated regions of the velocity spectrum. The shape of the curve in Figure 9 in relation to the morphology of the scales that formed at the different test velocities indicated that a change in the material loss mechanism had occurred.

3. Effect of Particle Velocity on Scale Morphology

Increasing the velocity of the erodent particles causes distinct changes to occur in the morphology of the scale's surface. Figure 10 shows how the surface of the scale changes over a range of particle velocities after a 5 hr exposure. The most notable difference occurs between a particle velocity of 30 m/s and 45 m/s, which corresponds to the sharp increase in erosion rate seen in Figure 9.

At \( V = 10 \) and 30 m/s the scale appears to be more segmented and cracked and has a larger oxide crystallite size. At \( V = 30 \) m/s the beginning of consolidation or densification of the scale can be seen. It becomes more pronounced at \( V = 45 \) and 70 m/s. At 45 and 70 m/s the scale is essentially continuous, no division or cracks are discernible, and individual crystallites cannot be observed at the magnification.
used. At 70 m/s, considerable Al₂O₃ can be seen embedded in the surface.

DISCUSSION

Test Temperature

The selection of 850°C as the test temperature at which to carry out this investigation was a compromise between service condition simulation and an acceptable duration laboratory test. The recommended continuous service temperature for 9Cr1Mo steel is 700°C. The modification of oxide scale formation by the impact of small, solid particles significantly modifies the kinetics of the reactions. It was felt that the degree to which scale formation kinetics are changed by the erosion-corrosion mechanism compared to static corrosion made it acceptable to use an accelerated rate test at a higher temperature. The combined mechanism appears to be independent of such considerations as parabolic rates and is a simpler function of combined chemical and physical occurrences that would reach a steady state condition where time and temperature could be traded off.

Erosion-Corrosion Zone Morphology

The primary, halo and outer erosion-corrosion zones that were observed on the test surfaces are related to the fluid mechanics of the essentially one dimensional flow of a 2 phase system out of a nozzle. The flow characteristics are discussed in Reference 6. The formation of a halo eroded zone around a center, primary zone on metal surfaces eroded at room temperature are discussed in Reference 9.

The outer two zones have a significant reduction in erosivity, especially for brittle materials such as scales, because the effective
angle of impingement is reduced, the velocity of the particles is less and the solids loading of particles is lower than in the primary zone. The effect of the different flow areas on the morphology of the scale and its propensity to spall during the test was significant. In metals, only the rate of erosion is different from one zone to the next.

Role of Scale Spalling on Metal Loss

As the velocity increased above 30 m/s the appearance of the surface of the scale changed, Figure 10. It appeared to be more continuous and condensed and dimples appeared on its surface from particle indentations. When this happened both the metal thickness loss and the degree of spalling of the scale, especially in the primary zone, increased markedly. It is postulated that the metal's thickness loss rate at the lower velocities is the erosion enhanced oxidation of the metal to form the scale observed in Figure 10. At higher velocities, the marked increase in the thickness loss rate was due to the occurrence of periodic spalling of the scale during the test. Thus, a major change in the erosion-corrosion mechanism occurs when the velocity is high enough to densify the scale.

The spalling could be due to the continuous nature of the condensed scale not having stress reducing cracks in it.\textsuperscript{10,11} In coatings on metals, cracks are induced in the coatings prior to service to increase their thermal fatigue life and prevent spalling. The large numbers of sub-critical micro-cracks reduce the elastic modulus and, therefore, minimize the stresses that can develop in the scale layer.
for a given strain level. This results in a strain accommodation that reduces the spalling tendency of the scale. Spalling of the scale also occurred during the cooling down period from the 850°C test temperature.

Transition From Low To High Metal Loss Rates

The metal thickness loss v.s. particle velocity curve, Figure 9, had a distinct transition in it at about 30 m/s going from low metal loss rates to high metal loss rates. This type of curve shape is indicative of a major change in the mechanism of the behavior that is being plotted. The major change in the mechanism of erosion-corrosion that occurred was the change from the low loss rate mechanism of cracking and chipping of small pieces of the scale to the high loss rate mechanism of periodic spalling of larger pieces of the scale. It is thought that the change in thickness loss rate at the higher particle velocities is due to the change in the manner in which the scale is removed rather than because of a change from primarily corrosion to a synergistic, combined erosion-corrosion mechanism. Corrosion was observed on all surfaces of the specimens after the test at all velocities. The initiation of loss of larger pieces of scale by spalling could account for the increased metal loss. A sharp increase in erosivity of the particles could not occur between 30 and 40 m/s to account for the large increase in erosion rate as the erosivity is a function of the kinetic energy which increases uniformly by the square of the velocity.
Oxide Formation Rates

The formation and loss of scale on areas that underwent spalling provided insight into the relationship between scale growth rates, erosion and the composition of the scale. A higher chromium content iron-chromium oxide spinel formed above the metals' grain boundaries (GB) compared to over the grains (Figure 7). This could be the result of the relative diffusion rates of chromium in the metals' grain boundaries and in the grain bulk which resulted in different growth rates of the scale in each area. The GB scale appeared to grow faster than that over the grain, leaving a higher, protruding scale of a lighter color above the GB's. This scale was more readily eroded than that which formed above the grains, causing an erosion-corrosion etching type phenomena to occur. This phenomena was only observed when the scale growth period was short and the scale formed was thin.

The short growth time of the scale was established by comparing the smaller size of the oxide crystallites in the spalled area of the primary zone to the larger size crystallites which occurred in the outer zone of the specimen where spalling did not occur. It has been observed in other elevated temperature erosion-corrosion testing that erosion-corrosion enhances the growth of oxide scale crystals. Therefore, the only way that the scale in the primary zone could have a smaller grain size than that in the outer zone is if it had considerably less time in which to grow.
CONCLUSIONS

1. A transition in the mechanism of scale loss occurred above 30 m/s when periodic spalling of the scale during the test replaced erosion enhanced corrosion as the primary erosion-corrosion mechanism.

2. Metal thickness loss increased with particle velocity over the range of velocities used.

3. The consolidation and densifying of the scale occurred at the higher velocities, making it prone to spalling.

4. Chromium rich Fe,Cr oxide spinels formed over the grain boundaries of the metal grains which were more prone to removal by particle impacts than the lower Cr content oxide which formed over the metal grains themselves.

5. Three distinct zones of erosion-corrosion occurred on the test specimens as the result of variations in the particle flow out of the nozzle because of fluid mechanics considerations.

ACKNOWLEDGEMENT


REFERENCES


FIGURES

1. Sketch of elevated temperature erosion tester.
2. Eroded-corroded surfaces of 9Cr1Mo steel at 250°, 450°, 650°C.
3. Surfaces of test specimens at temperatures and velocities in test grid.
4. Surface of scale in three erosion zones.
5. Surface showing both adhered and spalled scale areas.
6. Surface and cross section of scale beneath spalled area in primary zone.
7. Morphology and composition of scale beneath spalled area in primary zone.
8. Morphology of Scale beneath spalled area and in outer zone.
9. Effect of particle velocity on erosion-corrosion of 9Cr1Mo Steel at 850°C.
10. Effect of particle velocity on scale morphology in 5 hour tests.
TABLE 1
THICKNESS LOSS MEASUREMENTS AFTER 5 HOURS EXPOSURE AT 850°C

<table>
<thead>
<tr>
<th>Test No.</th>
<th>Test Conditions</th>
<th>Thickness of Specimen (mm)</th>
<th>Thickness Loss (mm)</th>
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<tr>
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<td>Before Test</td>
<td>After Test</td>
</tr>
<tr>
<td>1</td>
<td>Dynamic Corrosion</td>
<td>45</td>
<td>1.870</td>
</tr>
<tr>
<td>2</td>
<td></td>
<td>1.890</td>
<td>1.884</td>
</tr>
<tr>
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<td></td>
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<td></td>
<td></td>
<td>1.871</td>
</tr>
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</table>
1. Sketch of elevated temperature erosion tester.
250°C

10μm

9 Cr 1 Mo Steel
Nozzle Tester
Erosion-Corrosion
Air
130μm Al₂O₃ Primary Zone

V = 25 m/s
t = 5 Hrs.
α = 90°

2. Eroded-corroded surfaces of 9Cr1Mo steel at 250°C, 450°C, 650°C.
Velocity = 60 10 30 45 70 M/S

Dynamic Velocity of Particles
Oxidation no
Particles

9 Cr 1 Mo Steel

Nozzle Tester $\alpha = 90^\circ$
Erosion-Corrosion Time = 30min.
Air
130$\mu$m Al$_2$O$_3$

3. Surfaces of test specimens at temperatures and velocities in test grid.

XBB 8311-10381
Primary Zone

Halo Zone

Outer Zone

9 Cr 1 Mo Steel

Nozzle Tester $V = 35 \text{ m/s}$
Erosion-Corrosion $T = 900^\circ \text{C}$
Air $\alpha = 90^\circ$
$130\mu \text{m Al}_2O_3$ $t = 5 \text{ Hrs.}$

4. Surface of scale in three erosion zones.
9 Cr 1 Mo Steel

Nozzle Tester \( V = 35 \text{ m/s} \)
Erosion-Corrosion \( T = 850^\circ\text{C} \)
Air \( \alpha = 90^\circ \)
\( 130\mu\text{m} \) \( \text{Al}_2\text{O}_3 \) \( t = 5 \text{ Hrs.} \)

5. Surface showing both adhered and spalled scale areas.
Grain size on eroded surface

Grain size of cross section of eroded surface

Eroded surface

9 Cr 1 Mo Steel

Nozzle Tester
Erosion-Corrosion
Air
130μm Al₂O₃

α = 90°
Time = 5 Hrs.
T = 850°C
Velocity = 45 m/s

XBB 847-5134

6. Surface and cross section of scale beneath spalled area in primary zone.
9 Cr 1 Mo Steel

Nozzle Tester \( \alpha = 90^\circ \)
Erosion-Corrosion Time = 5 Hrs.
Air \( T = 650^\circ C \)
130\( \mu \)m \( Al_2O_3 \) Velocity = 45 m/s

7. Morphology and composition of scale beneath spalled area in primary zone.
9 Cr 1 Mo Steel

Nozzle Tester \quad \text{V} = 25 \text{ m/s}

Erosion-Corrosion \quad \text{T} = 850 ^\circ \text{C}

Air \quad \alpha = 90 ^\circ

130 \mu \text{m} \text{ Al}_2\text{O}_3 \quad t = 5 \text{ Hrs.}

8. Morphology of scale beneath spalled area and in outer zone.
9. Effect of particle velocity on erosion-corrosion of 9Cr1Mo steel at 850°C.

T = 850°C
α = 90°
t = 5 hr
750 g of 130 μm Al₂O₃
Gas = Air
10. Effect of particle velocity on scale morphology in 5 hour tests.
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