**Corrosion Analysis of Boiler Tubes from
a 100-hp Boiler Burning an Oil Coal Slurry**

R. H. Cooper

MASTER

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CORROSION ANALYSIS OF BOILER TUBES FROM A 100-hp BOILER
BURNING AN OIL COAL SLURRY

R. H. Cooper

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ABSTRACT

A corrosion analysis was performed on segments of ASTM A 178 steel boiler tubes removed from a 75-kW (100-hp) boiler operated by the Pittsburgh Energy Research Center. It burned a coal slurry in No. 6 fuel oil and produced 171°C (340°F) steam. The boiler tubes were exposed to this environment for 1000 hr with an operating wall temperature of about 180°C (350°F). A 76- μ m-thick (3-mil) scale formed on the fire side of these tubes. Exposure of the tubes to atmospheric conditions during boiler disassembly and cyclic boiler conditions prevented estimating the contribution of the flue gas exposure to the scale buildup. However, analysis of this scale indicated that fly ash deposition accounted for less than 5% of the tube weight of the scale

INTRODUCTION

In 1976, the Oak Ridge National Laboratory was requested to assist the Pittsburgh Energy Research Center (PERC) in performing a corrosion analysis on heat exchanger tubing from a 75-kW (100-hp) boiler that had been had been operated on an oil-coal slurry for 1000 hr. Metallographic analytical tools were used in this work, and the data were reported to PERC. Further studies based on x-ray diffraction measurements, scale weight measurements, and chemical analyses of tube scale were performed on other sections of tubes by PERC. A detailed discussion by PERC of these measurements is available in a formal project report.¹ The data reported here supplement the PERC analysis and serve as a permanent record of the metallographic observations.

¹J. J. Demeter et al., *Combustion of Coal-Oil Slurry in a 100 hp Firetube Boiler*, PERC/RI-77/8 (May 1977).

The boiler was a test facility for development of nozzles and air distribution systems needed to fire boilers on fuel oil-coal slurries and other fuel mixtures that are not in widespread commercial use. The nine tubes examined in this analysis had been exposed for 1000 hr to steam at 171°C (340°F) on the outside and flue gases generated from the combustion of a No. 6 fuel oil-coal slurry on the inside. During this exposure the tube wall temperature was maintained at about 180°C (350°F), and the fuel was burned with approximately 30% excess air. Measurable corrosion scale was observed on the surfaces of these tube segments. Spectrographic analysis of scale from the fire side of the tubes indicated that fly ash deposits accounted for less than 15% of the weight of the scale. Further investigation failed to determine what fraction of this scale was due to flue gas exposure, atmospheric exposure during boiler disassembly, and cyclic conditions associated with boiler operations. In view of these difficulties in interpreting the origin of this scale, recommendations concerning the handling of coupons and the operation of the boiler for future surveillance tests were made.

DESCRIPTION OF BOILER

The Cleaver-Brooks 75-kW (100-hp) boiler is a four-pass design. A conceptual drawing of this type of boiler is included as Fig. 1. During operation, an oil-coal slurry is injected and burned with excess air in the combustion chamber (pass 1). Combustion gases pass through one tube and are redirected in series through three consecutive arrays of tubes — passes 2, 3, and 4. Flue gas temperatures at various locations within the boiler were determined by PERC¹ to be:

Exit Pass 1	1289°C (2353°F)
Pass 2 Plenum	1257°C (2295°F)
Pass 3 Plenum	331°C (628°F)
Pass 4 Plenum	310°C (590°F)
Stack	232°C (450°F)

Fig. 1. Schematic Drawing of the Construction of a Typical Four-Pass Boiler. Taken from J. J. Demeter et al., *Combustion of Coal-Oil Slurry in a 100 hp Firetube Boiler*, PERC/RI-77/8 (May 1977).

During operation of this boiler the outlet steam temperature was maintained at 172°C (341°F). Under these conditions the temperature of the tube wall was measured¹ to be 177°C (350°F).

Throughout the 1000-hr exposure, the boiler was fired with excess air levels varying from 20 to 35%. The flue gas composition when the system was operating at 32% excess air was given¹ as: O₂, 4.7%; CO₂, 13.1%; NO_x, 0.04%; SO₂, 0.04%; N₂, 73.6%; and H₂O, 8.6%.

Before this 1000-hr run was started, two each of the original tubes from passes 2, 3, and 4 were replaced with 51-mm-diam (2-in.) standard electric-welded 12-gage ASTM A 178 tubing. The boiler was run in this configuration for 1000 hr on a fuel of 20% coal and 80% No. 6 fuel oil (the compositions of these components are listed in Table 1). On completion of the 1000-hr exposure, one tube from each of the three pairs of replacement tubes was removed, and 0.23-m (9-in.) samples were cut from both ends and near the center of each tube. These 0.23-m sections and one 0.15-m (6-in.) section of as-received tube were shipped to ORNL for corrosion analysis. Figure 2 schematically indicates the location and identification used for the samples discussed in this report.

Table 1. Typical Composition of Coal and Fuel Oil Used in This 1000-hr Exposure

	Pittsburgh Coal	No. 6 Fuel Oil
Content, wt %		
Hydrogen	5.3	12.2
Carbon	74.9	86.6
Nitrogen	1.5	0.2
Oxygen	9.5	0.1
Sulfur	1.9	0.8
Ash	7.0	
Heating value		
MJ/kg	31.3	44.1
Btu/lb	13,443	18,975

Fig. 2. Relative Sample Location and Identity.

SAMPLE PREPARATION AND RESULTS

Each sample was sectioned at ORNL into two 76-mm-long (3-in.) cylindrical portions. One portion from each pair was cut lengthwise to allow convenient inspection of both inside and outside contours. Figure 3 compares the interior and exterior surfaces of coupons from all nine surveillance locations. Figures 4 through 13 are photographs of the interior and exterior of the as-received sample and surveillance tube sections.

The composition of the tube material was confirmed with spectrographic analyses obtained from samples 2 mid, 3 mid, and 4 mid. Table 2 lists the results of these analyses and an analysis obtained by PERC from the tube vendor.

In addition, numerous samples of corrosion scale scraped from the interior contours of the 2, 3, and 4 inlet and outlet samples were analyzed spectrographically. The results of these analyses are summarized in Table 3.

Portions of all ten samples were mounted and polished for metallographic examination. Microhardness traverses across the wall thickness were performed on samples 2 inlet, 3 inlet, and 4 inlet; the results are listed in Table 4. In addition, each of these samples was examined under the optical microscope both "as polished" and etched.

DISCUSSION OF RESULTS

As-Received Tubing

The chemical analyses confirmed that the tubing conforms with the ASTM A 178 requirements (Table 2). The average grain size of this material is between ASTM 7 and 8 (Fig. 14). No unusual conditions in the material were observed at the exterior contour; however, grain structure of the material near the interior surface [Fig. 14(b)] suggests some decarburization and grain growth. Surface decarburization and grain growth may occur during the final annealing step of manufacturing and should not significantly alter the corrosion resistance of the material.

Fig. 3. A 178 Boiler Tubes from 75-kW (100-hp) Boiler.

CYN-2598

Fig. 4. Unused Section of A 178 Boiler Tube.

CYN-2599

Fig. 5. Second Pass Inlet of A 178 Boiler Tube.

Fig. 6. Second Pass Mid of A 178 Boiler Tube.

CYN-2601

Fig. 7. Seccd Pass Outlet of A 178 Boiler Tube.

CYN-2602

Fig. 8. Third Pass Inlet of A 178 Boiler Tube.

CYN-2603

Fig. 9. Third Pass Mid of A 178 Boiler Tube.

CYN-2604

Fig. 10. Third Pass Outlet of A 178 Boiler Tube.

CYN-2605

Fig. 11, Fcurth Pass Inlet of A 178 Boiler Tube.

CYN-2606

Fig. 12. Fourth Pass Mid of A 178 Boiler Tube.

CYN-2607

Fig. 13. Fourth Pass Outlet of A 178 Boiler Tube.

Table 2. Composition of Boiler Tubes

Elements	Composition, wt %				
	ORNL Analysis			Vendor Analysis	ASTM A 178 Specification
	2 Mid	3 Mid	4 Mid		
Si	0.01	0.01	0.02		
S	0.019	0.018	0.019	0.013	0.06 max
P	0.004	0.004	0.004	0.005	0.05 max
Mn	0.46	0.46	0.46	0.34	0.27-0.63
Cr	<0.01	<0.01	<0.01		
Ni	0.02	0.02	0.02		
Mo	0.01	0.01	0.01		
B	<0.001	<0.001	<0.001		
Nb	<0.01	<0.01	<0.10		
Ti	<0.01	<0.01	<0.01		
Co	0.005	0.005	0.005		
Cu	0.04	0.04	0.04		
Al	0.059	0.058	0.058	0.088	
N	0.005	0.007	0.007		
V	0.001	0.001	0.001		
W	<0.01	<0.01	<0.01		
As	0.004	0.004	0.004		
Sn	0.002	0.002	0.002		
Zr	<0.001	<0.001	<0.001		
C	0.086	0.089	0.090	0.08	0.06-0.18

Table 3. Summary of Analyses of Corrosion Scale on Inner Surface

Elements	Composition, ^a wt %					
	Pass 2		Pass 3		Pass 4	
	Inlet	Outlet	Inlet	Outlet	Inlet	Outlet
Al	0.6	0.4	0.4	0.7	0.8	>5.0
B	≤0.005	0.01	≤0.005	0.01	0.02	0.02
Ca	0.06	0.2	0.08	0.15	0.4	0.2
Co	<0.01	<0.01	0.02	<0.01	<0.01	<0.01
Cr	0.02	0.02	0.02	0.02		0.02
Cu	0.04	0.02	0.03	0.03	0.04	0.03
Hg	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
K	0.02	0.02	0.05	0.04	0.05	0.04
Mg	0.02	0.02	0.03	0.03	0.03	0.05
Mn	0.3	0.4	0.3	0.4	0.3	0.3
Mo	≤0.01	0.01	<0.01	0.02	0.01	0.02
Na	0.02	0.02	0.04	0.03	0.05	0.03
Ni	0.1	0.2	0.2	0.3	0.15	0.2
Pb	0.01	0.02	0.01	0.02	0.04	0.03
S	1.62	1.62	1.62	1.02	1.56	0.84
Si	0.3	0.7	0.3	0.7	1.0	3.0
Ti	0.005	0.01	0.01	0.02	0.05	0.2
V	<0.01	<0.01	≤0.01	0.02	0.01	0.01
Zn	<0.01	<0.01	<0.01	<0.01	0.03	<0.01

^aFe major constituent; Sn, Th, and W none reported.

Table 4. Microhardness Profiles Across Tube Wall

Sample	Location	Microhardness, DPH		
		Location of Indention		
		Outside	Mid	Inside
2 Inlet	A	114	109	108
	B	113	107	118
	C	107	108	118
	D	112	114	120
3 Inlet		115	115	105
4 Inlet		109	101	114

ORNL-DWG 78-6317

Fig. 14. Contour of the Unused Material. 2% Nital etch. 100 $\times$.
(a) Exterior. (b) Interior.

Figure 14 shows the interior and exterior contours of the unused portion of pipe. The black tenacious scale on these two contours is mill scale, which is often formed during the final process annealing step. Also the additional presence of rust on the "as-received" material reflects exposure to atmospheric conditions. It is not clear whether rust was present on the surveillance tubes before the 1000-hr test started. This uncertainty and the presence of rust significantly complicate the interpretation of the data.

Steam-Side Corrosion

Pure steam environments are relatively benign to carbon steel at moderate temperatures, and a protective scale of magnetite usually forms.² Therefore, the 5- to 13- μ m-thick (0.2-0.5-mil) scale found on the outside contour of these tubes [Fig. 15(a)] was expected. However, localized areas of pitting and heavy scale were observed on the steam side [Fig. 15(b)]. Localized attack may have resulted from either exposure to atmospheric conditions during boiler shutdown and disassembly or the temporary loss of chemical control of the boiler water (i.e., increased dissolved oxygen content). It should be noted that prevention of rusting during shutdown is a serious problem in commercial boilers, and rusting is a common cause of premature failure of boiler tubes in industrial boilers during subsequent start-ups.³

Flue Gas Corrosion

A visual inspection of the interior surfaces of the tubes from each of the nine boiler locations, Figs. 5 through 13, revealed an apparent scale buildup. A portion of this scale was removed from samples 2 inlet,

²H. H. Uhlig, *Corrosion Handbook*, Wiley, New York, 1948, p. 524.

³P. H. Effertz, "Morphology and Composition of Magnetite Layers in Boiler Tubes Following Long Exposure," pp. 920-24 in *Proceedings of the Fifth International Congress on Metallic Corrosion, Tokyo, Japan, May 1972*, National Association of Corrosion Engineers, Houston, Texas, 1974.

Fig. 15. Exterior Contour of Sample 2 Inlet. As polished.
 (a) Region with thin protective oxide layer. 200 $\times$. (b) Region with localized pitting and heavy scale. 100 $\times$.

3 inlet, and 4 inlet and spectrographically analyzed. The results of these analyses are listed in Table 3.

The maximum amount of fly ash in the scale can be estimated by comparing the contents of the scale (Table 3) from different locations with typical coal ash contents. The compound analysis of ash from the FMC process and the elemental analysis of typical Pittsburgh coal ash are given in Table 5; they have similar proportions of the major elements present. The relatively high amounts of aluminum and silicon found at the Pass 4 outlet suggest that if fly ash is deposited in the scale, it is present in the heaviest concentration at that location. On the basis of the silicon content of the scale and the Pittsburgh coal ash analysis, about 15% of the deposit might be fly ash at the Pass 4 outlet; elsewhere it might range from about 1.5% to about 5% of the total, neglecting contributions from the metal.

Table 5. Ash Analyses for Comparison with
Analyses of Scale from Tubing

Compound	FMC Char Ash ^a Content (wt %)	Pittsburgh Coal Ash	
		Element	Content (wt %)
SiO ₂	43-43.5	Si	22.5
Al ₂ O ₃	19.6-21.9	Al	12.3
Fe ₂ O ₃	25-25.7	Fe	10.2
SO ₃	6.7-7.5		
CaO	2.5-2.7	Ca	2.1
MgO	1.8-1.9	Mg	0.6
P ₂ O ₅	0.02-0.03		
Na ₂ O + K ₂ O	1.5-3.0	K	1.3
		Na	1.0

^aPrivate communication, Argonne National Laboratory, 1977.

The concentrations of Ti, Al, and Si in the scale on the tube surfaces increase with the distance from the combustion zone. To a limited extent a similar trend may exist for B, Ca, K, Na, Pb, and V. Intuitively this trend is not surprising, since the combustion gas and the tube wall surface temperatures decrease with increased distance from the combustion zone, allowing condensation of these species on the tube walls.

Representative photomicrographs of as-polished and polished-and-etched cross sections of the flue gas side of these tubes are shown in Figs. 16 through 19. The flue gas surface has a relatively uniform scale thickness of approximately 80 μm (3 mils). Further investigation showed some evidence of pitting but no indications of intergranular or decarburization attack. The uniformity of the microhardness results (Table 4) also illustrates that flue gas exposure caused no internal degradation of the tube material.

Fig. 16. Interior Contour of Sample 2 Inlet. 100 $\times$, As polished. Local nonuniformity of the scale is apparently due to loss of some scale, perhaps from handling or exfoliation in service.

Fig. 17. Interior Contour of Sample 2 Mid. 100 $\times$, 2% Nital etch. Some of the scale has pulled away from the base metal (lower) and adheres to the mounting material (top). Some scale probably dissolved during etching. Scale thickness was not measured on this sample in the etched condition.

Fig. 18. Interior Contour of Sample 4 Inlet. 100 $\times$, As polished. Local nonuniformity of the scale is apparently due to loss of some scale, perhaps from handling or exfoliation in service.

Fig. 19. Interior Contour of Sample 4 Mid. 100 $\times$, 2% Nital etch. Some of the scale has pulled away from the base metal (lower) and adheres to the mounting material (top). Some scale probably dissolved during etching. Scale thickness was not measured on this sample in the etched condition.

One should note that General Electric, as a contributor to the report by Demeter et al.,⁴ reported only 25 to 50 μm (1–2 mils) of scale as a result of a separate metallographic analysis of a different set of tube samples. Losses of scale in handling or differences in samples from location to location might explain the variance in the results. Nevertheless this variation in observed scale thickness was not reflected in the corrosion analyses portion of the project report.⁴

Since the wall thickness of this tube stock was not accurately determined before exposure, a precise metal loss rate cannot be calculated. However, from the available information a worst case estimate of the apparent metal loss rate can be made. In this calculation the surface scale is assumed to be 100%-dense Fe_3O_4 ; more precise calculations are not warranted with the uncertainty listed above. The 80- μm -thick (3-mil) scale formed during a 1000-hr exposure would represent a calculated metal loss rate of 0.3 mm/year (12 mils/year) if the rate of oxidation is constant. This estimated corrosion rate is higher than would be expected for this material when exposed to the environment

⁴J. J. Demeter et al., *Combustion of Coal-Oil Slurry in a 100 hp Firetube Boiler*, PERC/RI-77/8 (May 1977).

described by PERC. A list of factors that could promote excessive scale growth on these tubes and errors associated with the assumptions used in this calculation follows:

1. Corrosion and oxide growth generally follow a parabolic rate law, rather than the linear rate assumed for the calculations above. However, with only a single datum, the conservative approach is to use a linear approximation, which yields a higher corrosion rate.

2. The analysis by PERC showed that the flue gas contained 8% water vapor. On each of the many shutdown cycles experienced by this system, water vapor saturated with SO_2 and CO_2 would condense on the tube walls as an acidic solution. Such a condition is significantly more hostile than the high-temperature flue gas environment. Therefore, a large fraction of the scale observed on the tube walls could be the result of exposure to an acidic condensate rather than to the high-temperature flue gas.

3. A significant portion of the scale observed may have formed as a result of exposure of the tubes to atmospheric conditions following their removal from the boiler.

4. According to PERC, the flue gas from this boiler is made up principally of CO_2 , H_2O , and O_2 , with trace levels of SO_2 . Scale formed in this environment could be made up of magnetite, hematite, ferrous sulfide, and probably numerous other compounds. Analysis of the tubes with the optical microscope does suggest a mixture of compounds within this scale. (See x-ray analyses in ref. 4.) This investigation also revealed evidence of porosity in the scale. Both of these deviations from the assumptions used to calculate a corrosion rate would cause the calculated values to be too high.

RECOMMENDATION AND CONCLUSION

Examination of tube segments from PERC 75-kW (100-hp) boiler revealed a fire side corrosion scale about 80 μm (3 mils) thick. The fraction of this corrosion resulting from exposure to the flue gas environment cannot be determined. In view of this fact, continued analysis of the tubes is not expected to provide additional useful information.

This boiler design does not permit a practical method of exposing accurately characterized surveillance coupons to the flue gas environment while maintaining these coupons at temperatures similar to the wall temperature of the tubes. Destructive analysis of boiler tubes appears to be the only method for obtaining additional corrosion data. It must be emphasized that this type of testing will produce only measurements of scale thickness and not precise metal loss data. However, this information can be valuable in assessing the relative magnitude of corrosion in this environment. The following recommendations are made for future tests:

1. Surveillance tubes should be initially as free of corrosion as possible and well-characterized dimensionally at locations where post-test measurements are to be made.
2. A portion of as-received material should be sealed in an inert environment and held as a reference sample.
3. During subsequent boiler runs, an effort should be made to minimize the number of shutdown and start-up cycles.
4. The significance of data obtained from surveillance samples generally increases with increased exposure time; therefore, the sample tube should be exposed for a minimum of 1000 hr. Furthermore, the test matrix should involve more than one exposure time.
5. On termination of the run, the surveillance tubes should be promptly removed from the boiler, sectioned into samples, and sealed in an inert environment for shipment to the laboratory.

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