

Corrosion of carbon steel tubes in the mid-furnace of kraft recovery boilers – environment characterization

PREET M. SINGH, VIKAS R. BEHRANI, AND JAMSHAD MAHMOOD

ABSTRACT: Carbon steel tubes in the mid-furnace of kraft recovery boilers have experienced accelerated corrosion in recent years. Gaseous environments in the mid-furnace of kraft recovery boilers were generally considered to be oxidizing and not very corrosive to the carbon steel waterwall tubes. However, mid-furnace corrosion has occurred above the cut line where composite tubes that have a stainless steel outer layer used in the lower furnace are welded to the carbon steel waterwall section. This study characterized the local environments in three boilers to find the reasons for accelerated corrosion of waterwalls in the mid-furnace. Gas sampling ports were installed in the mid-furnace with high and low corrosion rates of carbon steel waterwall tubes. During boiler operation, gases were sampled and analyzed using a specially designed gas chromatograph. In each boiler, the areas with higher corrosion rates had higher concentrations of reducing gases and sulfur-containing gases at the waterwall surface than in the low-corrosion areas. Results from this study indicate that accelerated mid-furnace corrosion likely results from changes in the boiler operation, such as decreases in the air supply at the lower air port levels of the furnace. These changes also might result in higher local gas temperatures and local thermal cycling in the mid-furnace of boilers.

Application: The results in this paper will help boiler owners, manufacturers, and operators to better understand and mitigate corrosion problems in this area of recovery boilers.

Accelerated corrosion of carbon steel tubes in the mid-furnace areas has become a problem for a number of kraft recovery boilers in recent years. Mid-furnace corrosion has occurred in boilers in which significant operational changes, including increased liquor solids and decreased air supply at the lower furnace levels, were introduced. Accelerated corrosion in mid-furnace (also referred to as cut-line corrosion) was more noticeable above the cut line where the stainless steel waterwall tubes are welded to the carbon steel tubes. A significant amount of work has been published on the gaseous environment in boilers and the role of gas composition and thermal conditions on the kinetics of corrosion reactions in recovery boilers [1-13]. However, very little information was available on the gaseous environments at the waterwall surface in mid-furnace areas of recovery boilers.

Recent changes in operating processes, such as limited air supply at the lower level, seem to have caused significant change in the local environment at the tube surface. The mid-furnace area separates areas with reducing and sulfidizing environments in the lower furnace from the oxidizing environment of the upper furnace areas. Significant instability in the gas flow in a boiler furnace can lead to cycling between an oxidizing and a sulfidizing environment in the mid-furnace area [14]. Previous work [14-19] on the corrosion behavior of alloys in the oxidizing and sulfidizing environments with thermal cycling have shown spallation of scale due to thermal stresses. Other high-temperature commercial processes, such

as oil refining, coal gasification, and fossil-fuel conversion, produce complex gaseous environments that can be oxidizing or sulfidizing to alloy components. The resulting multi-oxidant process environments often are not at equilibrium and can vary in the degree of aggressiveness with changes in operating conditions. Corrosion behavior of metals and alloys in oxidizing-sulfidizing environments has been studied by a number of researchers [23-28] to simulate the industrial environments, but no systematic work has been done to understand the environment in the mid-furnace of kraft recovery boilers.

The main objective of this study was to characterize gaseous environments in the mid-furnace areas of different boilers to develop an understanding of the possible corrosion mechanisms operating in the mid-furnace of kraft recovery boilers. Data from three recovery boilers with mid-furnace corrosion problems revealed similarities and differences in the mid-furnace gaseous environment of different boilers.

EXPERIMENTAL PROCEDURES

Kraft recovery boilers selected for this study had experienced accelerated mid-furnace corrosion of waterwall tubes. Mid-furnace areas with high and low corrosion rates of waterwall tubes were identified based on the waterwall thickness data from their annual inspections. Corrosion was detected above the last level of air supply (air ports) and cut line (where the composite tubes used in the lower furnace are welded to the carbon steel tubes). During a boiler shutdown, air-tight stain-

CORROSION

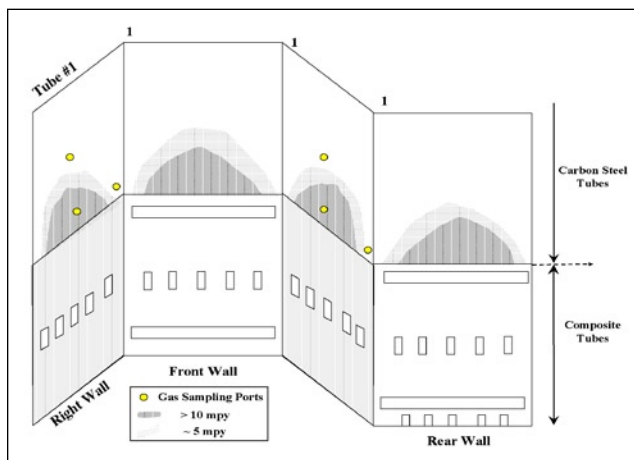
less steel gas-sampling ports were installed in each selected area by drilling a hole through the web membrane and welding a specially designed stainless steel sampling port to support the ceramic tube used to take local gas samples from inside the boiler.

Gas samples from the recovery boiler mid-furnace were collected from the waterwall surface in the areas of interest. Gas samples were taken from the waterwall tube surface by keeping the sampling tube flush with the waterwall surface. To prevent chemical reactions and changes in the gas composition due to high temperature corrosion of metallic gas lines, we used ceramic tubes to sample gases from these ports. Multiple samples were collected from each port. Gases were analyzed using a PerkinElmer gas chromatograph with two thermal conductivity detectors, for the light gases and a flame photometric detector for the sulfur-bearing gases. Typical gas sampling included light gases such as oxygen, nitrogen, hydrogen, carbon dioxide, methane, and carbon monoxide, and sulfur-bearing gases such as hydrogen sulfide, carbonyl sulfide, methyl mercaptan, dimethyl sulfide, and sulfur dioxide. The gas chromatograph was calibrated using standard gases. For this study, we did not quantify water content in the recovery boiler gases, and all gas compositions given in this paper are given on the dry-gas basis.

RESULTS AND DISCUSSION

Results from boiler 1

Boiler 1 was operating at 6.0 MPa (875 psig) steam pressure, with a steam temperature in the mid-furnace tubes of approximately 274°C. This boiler had two levels of air supply. Secondary air was supplied at each corner of the boiler (four ports), at the load burner level. The distance between the liquor gun elevation and the secondary air ports was approximately 20 ft. **Figure 1** shows the corrosion pattern in the mid-furnace of this boiler. Figure 1 also shows the locations of gas sampling



1. Schematic of boiler 1 showing corrosion patterns and gas sampling port locations on the waterwall surface in mid-furnace area (above the cut line). This boiler had two air levels. High corrosion rates were experienced at higher elevations in the middle of each wall compared with corrosion in the corners.

Average % Gas Species Concentration -Right Wall			
Gas Species	Low-corrosion area (near cut line)	High-corrosion area (near cut line)	10 ft above cut line (low-corrosion area)
H ₂	6.67	12.04	5.16
CO ₂	17.71	14.23	17.87
O ₂	0.52	0.27	0.55
CH ₄	1.82	4.42	1.59
CO	13.57	27.95	13.98
N ₂	58.62	40.60	59.74
H ₂ S	0.31	0.91	0.16
COS	0.10	0.20	0.12
CH ₃ SH	0.023	0.030	0.013
(CH ₃) ₂ S	0.028	0.033	0.033
CS ₂	0.0018	0.0012	0.0024
(CH ₃) ₂ S ₂	0.0012	0.0019	0.0009

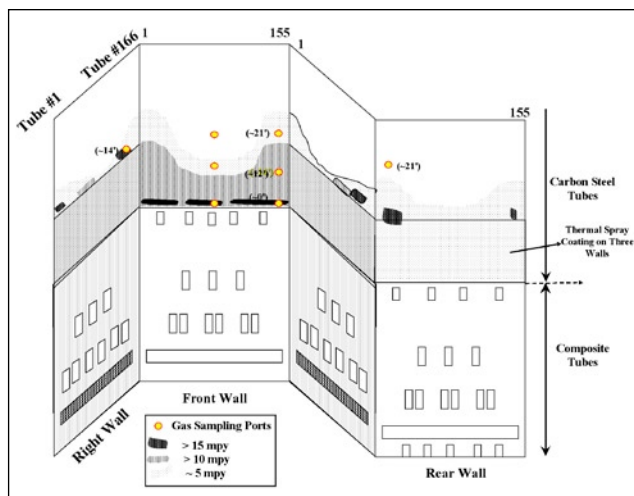
I. Average gas compositions at the right side waterwall in mid-furnace with high and low-corrosion area of boiler 1. Location of ports for these samples was near secondary air port just above the cut line. (This boiler had two levels of air supply.)

Average % Gas Species Concentration -Left Wall			
Gas Species	Low-corrosion area (near cut line)	High-corrosion area (near cut line)	10 ft above cut line (low-corrosion area)
H ₂	2.51	8.7	1.54
CO ₂	14.84	15.9	20.42
O ₂	2.56	0.7	1.20
CH ₄	0.38	2.5	0.32
CO	8.31	33.1	4.70
N ₂	71.53	38.0	72.34
H ₂ S	0.34	0.761	0.00
COS	0.044	0.223	0.000
CH ₃ SH	0.010	0.003	0.000
(CH ₃) ₂ S	0.010	0.005	0.000
CS ₂	0.0002	0.000	0.0000
(CH ₃) ₂ S ₂	0.0002	0.000	0.0000

II. Average gas compositions at the left side waterwall in mid-furnace with high and low-corrosion area of boiler 1. Location of ports for these samples was near secondary air port just above the cut line. (This boiler had two levels of air supply.)

ports installed on the left and right wall in this boiler.

A minimum of seven gas samples were taken from each sampling port over a period of two days. **Tables I** and **II** list average gas compositions for the boiler 1 at selected mid-furnace locations on left-side and right-side walls. Gas samples from each area varied slightly. However, the gas composition at the waterwall surface did not vary significantly between



2. Schematic of boiler 2 showing corrosion patterns and port location on the waterwall surface in mid-furnace area (above the cut line).

the corrosive and noncorrosive areas (Tables I and II). Nitrogen and oxygen concentrations were significantly higher in the low-corrosion areas compared with gases in the high-corrosion areas. Hydrogen, methane, and carbon monoxide, all of which are produced during pyrolysis and combustion of black liquor, were in higher concentrations in the high-corrosion areas. The sulfur-bearing gases (hydrogen sulfide and sulfur dioxide) were generally at higher concentrations in the high-corrosion area than in the low-corrosion area. Similar differences in gas compositions were found in the south and north wall gas samples.

The nitrogen and oxygen concentrations were higher at low corrosion rate areas in the mid-furnace, whereas, the concentrations of hydrogen, methane, carbon monoxide, and sulfur-bearing gases were lower than in the high-corrosion area.

Gas species	% Gas Species Concentration	
	Average high-corrosion areas (at cut line)	Average low-corrosion areas (12 ft above cut line)
H ₂	4.52	0.49
CO ₂	5.81	10.13
O ₂	5.19	9.87
CO	5.25	0.71
CH ₄	0.11	0.0
N ₂	78.7	78.6
H ₂ S	0.131	0.0030
COS	0.213	0.0197
CH ₃ SH	0.001	0.000
(CH ₃) ₂ S	0.0001	0.000

III. Average gas compositions at the waterwall in mid-furnace with high and low-corrosion area of boiler 2. Location of ports for these samples was near tertiary air port just above the cut line.

Results from boiler 2

Boiler 2 had three air levels, pressure of 10.3 MPa (1500 psig), and a steam temperature in the mid-furnace of ~320°C. Composite tubes extended up to the tertiary air port elevation. The boiler had significant corrosion of the carbon steel waterwalls in the mid-furnace up to 15–23 ft above the cut-line level. The corrosion pattern differed from that of boiler 1; the corners were more corroded than was the middle of waterwalls (**Fig. 2**).

Multiple gas samples from the waterwall surface were taken from the installed ports. **Table III** shows average gas composition for the high-corrosion areas (with corrosion ~15 m/year) and the low-corrosion areas (with corrosion rate of ~5 m/year), just above the cut line. Generally, the areas above the tertiary air port levels are expected only to have oxidizing gases; however, the gas compositions in Table III indicate that the gases in the mid-furnace of boiler 2 contained significant concentrations of reducing gases like hydrogen and carbon monoxide, along with the reduced-sulfur-bearing gases like hydrogen sulfide and methyl mercaptans. The concentration of reducing and reduced-sulfur-bearing gas concentrations in the high-corrosion areas of the mid-furnace were significantly higher compared to the gas composition in the lower corrosion areas at the same elevation.

Gases were analyzed at regular intervals from one high-corrosion area and one low-corrosion area of boiler 2 to check for the variations in the gas composition over time. Gases in the low-corrosion area were generally more oxidizing than in the high-corrosion area. The gas composition detected in the high-corrosion areas also varied considerably. Gas compositions changed significantly, from reducing and sulfur-containing to oxidizing and without any sulfur gases. Variations in the gas composition were less significant in the low-corrosion areas than in the high-corrosion area.

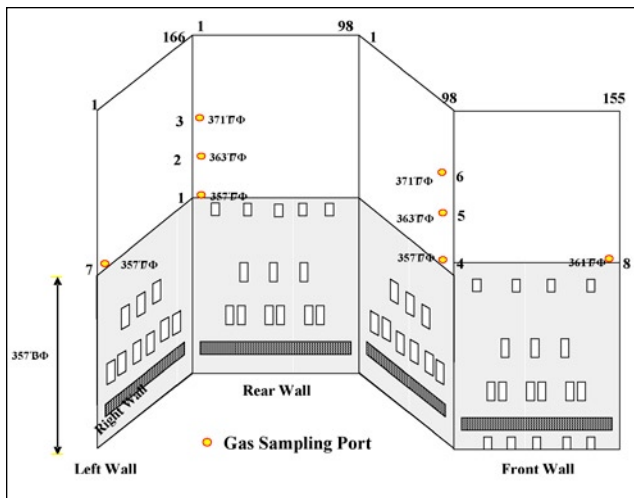
Hydrogen sulfide and other sulfur bearing gases were detected in almost all selected areas near the cut line, but the concentration of oxidizing gases were higher in the low-corrosion area and about 10 ft above the cut line on both walls. Accelerated corrosion in mid-furnace areas in boiler 2 could be attributed to a higher local concentration of reducing and sulfidizing gas compositions. However, gas composition fluctuations also might have been a factor in producing an unstable scale on the waterwall tube surface.

Results from boiler 3

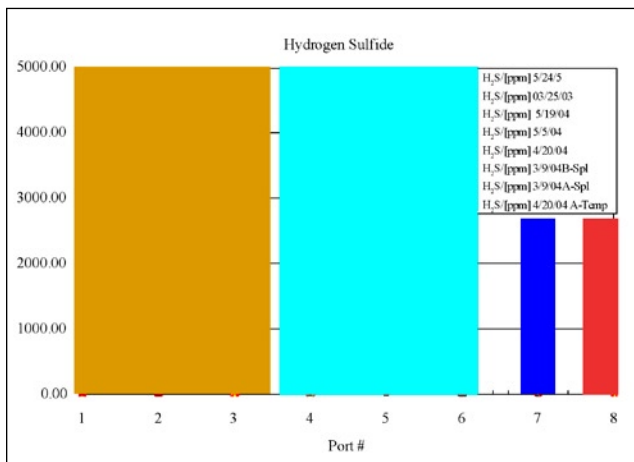
Boiler 3 had three air levels and was operated at 10.3 MPa (1500 psig) pressure with a steam temperature around 320°C. Composite tubes in this boiler extended up to the tertiary air port elevation. The boiler experienced significant corrosion of the carbon steel tubes in three corners. The front wall and the rear wall had accelerated corrosion on the carbon steel tubes extending almost 12 ft above the cut line (**Fig. 3**).

To understand the extent of gas composition variations in the mid-furnace areas, gas samples from the mid-furnace of boiler 3 were collected and analyzed seven times over a two-year study period. During this period, the boiler operation

CORROSION



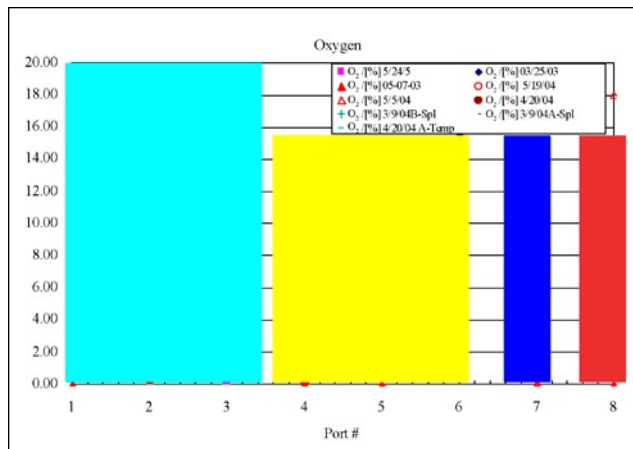
3. Schematic of boiler 3 showing port location in mid-furnace area (above the cut line).



4. Variation in the concentration of hydrogen sulfide in gas samples, during a 2-year period, taken from different ports in the mid-furnace, shown in Figure 4. Locations at Port #s 1 and 4 had highest corrosion rate (~15mpy) at the waterwall surface whereas Port #s 3 and 4 had the lowest corrosion rates (<5 mpy).

parameters were not changed, so the data represent “normal boiler operation” conditions. Multiple samples were taken from each port during each visit. The average of the gas composition for one particular day was used to determine a day-to-day variation in the boiler gas composition.

Hydrogen sulfide and other sulfur bearing gases were detected in almost all selected areas near the cut line, but the concentration of oxidizing gases was higher in the low-corrosion area at the cut line level and about 10 ft above the cut line, on both walls. Gas compositions at the high-corrosion areas on both walls were generally sulfidizing and reducing, with higher concentrations of hydrogen and carbon monoxide gases. **Figure 4** shows hydrogen sulfide concentrations in each sample taken from each port over the two-year period. Hydrogen sulfide in port 1 varied from 0 ppm to ~3500 ppm. The variation in gas composition was smaller at ports 2 and 3, which were at higher elevations in the same corner of the



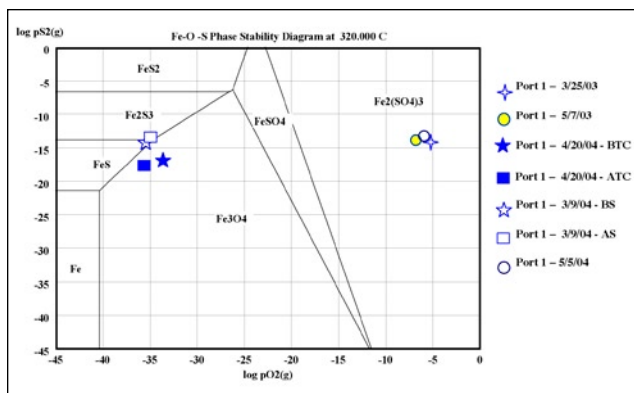
5. Variation in the concentration of oxygen in gas samples, during a 2-year period, taken from different ports in the mid-furnace, shown in Figure 4. Locations at Port #s 1 and 4 had highest corrosion rate (~15mpy) at the waterwall surface whereas Port #s 3 and 4 had the lowest corrosion rates (<5 mpy).

rear wall (Fig. 4). These results indicate that the gas composition is less stable at the boiler’s lower elevations, near the tertiary air port and cut line. Similar trends were found for the hydrogen and carbon monoxide composition in the gas samples. **Figure 5** shows a large variation in the oxygen concentration at the waterwall surface at different ports. The oxygen concentration decreased as elevation increased, as might be expected because oxygen is consumed in combustion and other reactions. Instability in the boiler gas flow and occasional black liquor spray at the waterwall surface near cut line also contribute to large variations in gas compositions.

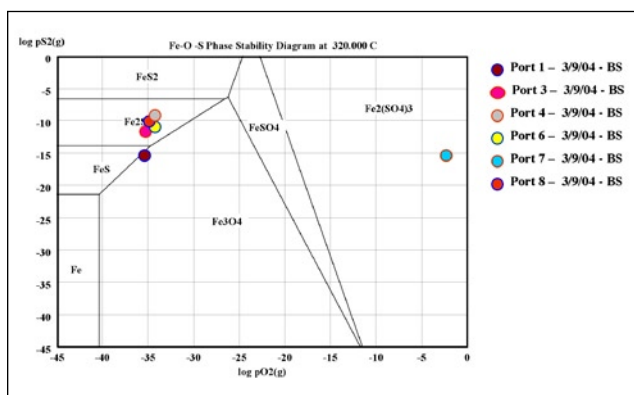
To understand the reason for the variation in the gas composition, operation records for the boiler were compared with analyzed gas composition data. We looked for any changes in the liquor temperature, liquor pressure, air temperature, or air distribution that might explain the variation in local gas composition. However, we found no correlation between the changes in the local gaseous environment in the mid-furnace and the boiler operating parameters. Based on this we concluded that the gas composition variation might result from the instable gas flow in the mid-furnace of this boiler.

The type of scale that develops on metal surfaces strongly depends upon the gas composition. Sulfide scales develop when the gases are reducing and sulfidizing, whereas an oxide scale develops when the gases contain higher concentrations of oxidizing gases. We used average gas composition data from each port to calculate the partial pressure of oxygen and sulfur. Overall scale composition will depend on the thermodynamic stability and on the kinetics of formation and rate of conversion reactions.

We calculated the partial pressures of sulfur and oxygen in a given gas mixture and at a given temperature by assuming a thermodynamic equilibrium, which is more certain at higher temperatures. **Figure 6** shows regions of thermodynamically stable phases as a function of oxygen and sulfur



6. Phase stability diagram for an iron-oxygen-sulfur system at 320°C. Diagram shows partial stability regions for different possible scale compositions as a function of oxygen and sulfur partial pressures.



7. Phase stability diagram for an iron-oxygen-sulfur system at 320°C. Diagram shows partial stability regions for different possible scale compositions as a function of oxygen and sulfur partial pressure in contact with iron.

partial pressures in an iron-oxygen-sulfur system at 320°C. The average gas composition of samples taken from this port on each day was used to calculate the partial pressures of oxygen and sulfur and was plotted on the phase-stability diagram for the iron-oxygen-sulfur system at 320°C. Gas samples for Fig. 6 were taken from the same port location (i.e. port 1) in the boiler, but on different days. Data indicate that the gaseous environment at this port varied from very oxidizing to sulfidizing under the so-called normal boiler operation conditions during different periods. These results clearly indicate that the gaseous environment fluctuated in the mid-furnace of this boiler. Instability in the boiler gas-flow in the mid-furnace area might explain the difference in the local gas composition at different times under otherwise similar operating conditions (Fig. 6).

Data superposed on the phase stability diagram in **Fig. 7** show the difference in the gas composition at different port locations in the mid-furnace on a particular day. On this day, the average gas composition in all selected areas, except for port 7, was rich in sulfidizing gases, which thermodynamically favors the formation of iron sulfide (Fe_2S) on the carbon

steel tube surface. On other days, the gas compositions for the ports at the higher elevation were more oxidizing than the lower ports. Figures 6 and 7 show that gaseous environments in local areas can change from completely oxidizing to completely reducing. This was more prominent in the lower areas, near the cut line, and generally associates with areas with higher corrosion rates.

We determined that black liquor might be sprayed unintentionally in certain areas of mid-furnace waterwall surface. Thermodynamics calculations indicate that at a pyrolysis temperature of 700°C (much above the waterwall surface temperature) or lower, sodium sulfide and potassium sulfide are less likely to form, thus methyl mercaptan and hydrogen sulfide are more stable and are likely to be higher in concentration near the tubes than in the bulk of the flue gas, particularly away from the air ports. When black liquor is sprayed on tube surfaces and undergoes direct pyrolysis, the reduced sulfur gases remain in reduced form because of the low temperature and the low oxygen partial pressures. This can lead to a change in the ratio of methyl mercaptan and dimethyl sulfide to hydrogen sulfide [12]. This can locally change the oxygen-to-sulfur ratio and favor a sulfide corrosion product rather than an oxide. These small variations in the sulfur partial pressure can result in catastrophic corrosion conditions [13].

Oxide scale on the iron surface, in the boiler tube temperature range, is much more protective than sulfide scale. If the gas composition cycles between the sulfidizing to the oxidizing compositions, the resulting scale might not be very protective and can result in a higher corrosion rate [29]. Previous work on the lower furnace corrosion has shown that the higher corrosion rate of carbon steel might result from the following:

- organo-sulfur compounds in gases,
- alternating oxidizing and reducing conditions,
- cyclic temperatures, and
- higher temperatures.

The differences in corrosion rates in the high and the low-corrosion areas largely depend upon the stability of the scale. Conditions favoring sulfide scale or an unstable scale will lead to accelerated waterwall corrosion. Analysis of the gaseous environments in the mid-furnace areas of the three kraft recovery boilers indicate that accelerated corrosion in recent years might be attributed to higher concentrations of sulfidizing and reducing gases and to fluctuating gaseous environments in these areas caused by air-flow instability.

CONCLUSIONS

Based on the results from the three boilers studied, we can conclude the following:

- In each boiler, areas with higher corrosion rates in the mid-furnace had higher concentrations of reducing gases and sulfur-containing gases at the waterwall surface than in the low-corrosion areas of the same boiler.
- Reducing/sulfidizing environments in the local areas of the mid-furnace might result from operational changes such

as air-split and firing of high solids black liquor.

- Data from boiler 3 indicate that the cyclic (oxidizing/reducing) environments in the mid-furnace might result from gas-flow instability and cause an unstable or less protective scale on the tube surface.

- Local gas flow instability can produce fluctuations in the tube-surface temperature and might result in scale damage, depending upon the magnitude of thermal excursions. **TJ**

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INSIGHTS FROM THE AUTHORS

We choose this topic to research because corrosion in kraft recovery boilers is an important concern in the pulp and paper industry. Our previous research was related to understanding the environment in the lower furnace areas of kraft recovery boilers. This paper has used some of the same techniques to understand cut-line corrosion.

The most difficult aspect of this study was to install the gas-sampling ports in the selected areas and to get gas samples during boiler operation. This was accomplished with tremendous support from the mill personnel and other colleagues.

It has been assumed that the mid-furnace areas are noncorrosive due to oxidizing gases in this area. However, we found that the local gas compositions can be reducing and sulfidizing. This work also showed that the gas compositions fluctuate in a general area of the boiler.

The results of this study help us understand the reasons for corrosion in the mid-furnace. Mills can either use this information to change their operations to change local environments, or use these results to select better materials in these areas to mitigate corrosion.



Singh



Behrani



Mahmood

Our next step is to develop or select better coating materials for in-situ repair of old tubes and to develop or select better, more cost-effective tube materials for new boilers.

Singh is with the School of Materials Science and Engineering and the Institute of Paper Science and Technology; Behrani is with the School of Materials Science and Engineering; and Mahmood is with the Institute of Paper Science and Technology, Georgia Institute of Technology, Atlanta, Georgia, USA. Email Singh at Preet.Singh@mse.gatech.edu.



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