405
8/25/81
e.h.

② DR-297/
B6684

ornl MASTER

ORNL/TM-7734/P2

OAK
RIDGE
NATIONAL
LABORATORY

UNION
CARBIDE

**Corrosion of High-Temperature
Materials in AFBC Environments
Part 2 (4500-h Tests)**

T. G. Godfrey
J. H. DeVan

Fossil
Energy
Program

OPERATED BY
UNION CARBIDE CORPORATION
FOR THE UNITED STATES
DEPARTMENT OF ENERGY

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

Printed in the United States of America. Available from
National Technical Information Service
U.S. Department of Commerce
5285 Port Royal Road, Springfield, Virginia 22161
NTIS price codes—Printed Copy: A05 Microfiche A01

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

ORNL/TM-7734/P2
Distribution
Categories
UC-90e, #h

Contract No. W-7405-eng-26

METALS AND CERAMICS DIVISION

CORROSION OF HIGH-TEMPERATURE MATERIALS IN AFBC ENVIRONMENTS
PART 2 (4500-h TESTS)

T. G. Godfrey and J. H. DeVan

Date Published: August 1981

DISCLAIMER

This book was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37830
operated by
UNION CARBIDE CORPORATION
for the
DEPARTMENT OF ENERGY

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

**THIS PAGE
WAS INTENTIONALLY
LEFT BLANK**

CONTENTS

ABSTRACT	1
INTRODUCTION	1
TEST PROGRAM AND PROCEDURES	2
Description of Combustor	2
Tube Matrix	4
Preparation of Tubes	5
Methods of Examination	8
OPERATING CONDITIONS AND RUN DATA	8
Normal Operations	8
Upset Operations	10
Trimmer Tube Failure	10
Tube Temperatures	11
TEST RESULTS	19
Low-Temperature Tubes	19
High-Temperature Tubes	25
Alonized Specimens of Alloy 800H and Type 310 Stainless Steel	32
Corrosion Response Based on Tube Wall Thickness Measurements	32
Severe Corrosion of Type 316 Stainless Steel	39
SUMMARY AND CONCLUSIONS	41
Low-Temperature Tubes	41
High-Temperature Tubes	42
Types 304 and 316 Stainless Steel	42
Type 310 Stainless Steel	43
Alloy 800H	43
ACKNOWLEDGMENTS	44
REFERENCES	45
APPENDIX A — Analyses of Bed Feed	47
APPENDIX B — Daily Test Conditions	49
APPENDIX C — Adjustment of Feed Rate Data	55
APPENDIX D — Summary of Upset Conditions	61
APPENDIX E — Analysis of Trimmer Tube Failure	65
APPENDIX F — Detailed Fire-Side Corrosion Measurements	73

CORROSION OF HIGH TEMPERATURE MATERIALS IN AFBC ENVIRONMENTS
PART 2 (4500-h TESTS)

T. G. Godfrey and J. H. DeVan

ABSTRACT

Candidate heat exchanger tube materials were tested for times to 4500 h in a small atmospheric-pressure fluidized bed combustor (AFBC) operated by the FluidDyne Engineering Corporation of Minneapolis, Minnesota. The materials included alloy 800H; types 304, 310, and 316 stainless steel; and aluminumized alloy 800H and type 310 stainless steel. These air-cooled tubes were exposed to the AFBC environment with wall temperatures ranging from 820 to 875°C, a Ca/S molar ratio of 3.3 to 5.3, 2.5 to 3.5% excess O₂, and a fluidizing velocity of 0.7 m/s (2.3 fps).

A set of low-temperature tubes was also included in the test for the final 3000-h period. These tubes were composites of 2 1/4 Cr-1 Mo steel and type 304 stainless steel and were air-cooled to temperatures in the range 480 to 590°C.

Tubes were removed at intermediate times for metallographic examination. In general, the materials performed well. With one exception, metal wastage was at the lower limit of detection and intergranular corrosion was not severe. Most of the high-temperature samples, however, contained subsurface specks of metal sulfides, primarily of manganese, extending to depths of about 50 μ m.

The exception to the good performance noted above was a type 316 stainless steel high-temperature tube exposed for the final 3000-h portion of the 4500-h test. This tube suffered severe sulfidation-oxidation over most of its surface. The absence of such attack on eight other type 316 stainless steel tubes indicated that the position of the affected tube in the bed may have been a more significant corrosion factor than the steel composition *per se*.

INTRODUCTION

The atmospheric-pressure fluidized bed combustor (AFBC) offers promise as an environmentally acceptable and cost-effective method of producing heat energy from coal for industrial or utility applications.¹

Depending upon the particular design or application, heat of combustion generated in the fluidized bed is transferred to air or steam through a heat exchanger immersed directly in the bed.

The Coal Combustor for Cogeneration (CCC) Program at ORNL is directed at the development of an AFBC system to heat clean air at temperatures approaching 815°C (1500°F) to drive a gas turbine to generate electricity. A test program designed to evaluate the corrosion performance of candidate heat exchanger materials for the CCC concept has been carried out with an AFBC operated by Fluidyne Engineering Corporation. We previously reported^{2,3} test results obtained under this program for heat exchanger tubes exposed 500 h and 1500 h, respectively. Subsequent tests under the same bed operating conditions have extended the total exposure time of the tubes to 4500 h. In the same tests other tubes were exposed at lower temperatures for 3000 h. The present report describes the operating and test results for these longer term tests and compares the findings with those of the previous 500- and 1500-h exposures.

TEST PROGRAM AND PROCEDURES

Description of Combustor

The combustor used in this test program was a 0.46 by 0.46 m (18 by 18 in.) AFBC constructed and operated by the Fluidyne Engineering Corporation of Minneapolis, Minnesota. In this combustor, bed temperature is controlled by an array of 41 air- and water-cooled tubes, which pass horizontally through the bed, as shown in Fig. 1. This facility operates with a constant combustion air input with coal feed input regulated to maintain the desired excess oxygen in the exhaust flue. The limestone feed is slaved to the coal feed and controlled so as to maintain the desired SO_2 concentration in the flue gas. The coal used was Illinois No. 6 with 3.5% S, ground to -4 mesh. The sorbent was Quincy limestone. Typical analyses are given in Appendix A. The coal and limestone were mixed and pneumatically conveyed into the bed through a single feed lance. Additional details concerning design and operation of this AFBC facility have been presented elsewhere.^{2,3}

OPERATING CONDITIONS

BED TEMPERATURE	890°C (1635°F)
FLUIDIZING VELOCITY	0.70 m/s (2.3 fps)
EXCESS O ₂	2.5-3.5 %
SO ₂	250-400 ppm
Ca/S	3.3

	COLUMN									
	10	9	8	7	6	5	4	3	2	1
ROW	A	+		+		+		+		+
			○	+		+		■		+
	B	+		■		○		○		○
			○		○	+		○		+
C	+		○		○		○		○	
		○		+		+		○		+
D	+		○		+		+		■	
		○		■		○		+		+

+ BED TEMPERATURE CONTROL TUBES
 ○ HIGH-TEMPERATURE TUBES
 ■ LOW-TEMPERATURE TUBES

Fig. 1. The fluidized bed built by FluidDyne, showing the location of the cooling coils in the bed and the location identification. Normal operating conditions are also specified.

Tube Matrix

The initial 500- and 1500-h tests in the combustor, described elsewhere,^{2,3} used 16 of the 41 tubes in the bed as corrosion test specimens. These 16 air-cooled tubes comprised types 304, 310, and 316 stainless steel; alloys 600 and 800H; and Alonized* samples of type 310 stainless steel and alloy 800H.

The current test series used the same 16 tube positions for an additional 3000-h run, which was conducted in sequential segments of 500, 1000, and 1500 h. The specimen tube positions, materials, and time intervals, including the initial 500- and 1500-h tests, are shown in Fig. 2. Note that eight tubes were carried from the initial 1500-h test run into the final 3000-h run and that six of these tubes (2B, 3C, 4B, 4C, 6C, 8D) were exposed for the total test duration, 4500 h. The test alloys in this latter set were types 316 and 310 stainless steel, alloy 800H, Alonized* alloy 800H, and Alonized* type 310. The eight new tubes added at the start of the 3000-h run consisted of types 304, 310, and 316 stainless steel and alloy 800H, and subsequent tube replacements among the 16-tube array were confined to the same alloys. Temperature conditions were approximately the same for each of the 16 tube positions, namely, a tube wall temperature of about 810°C (1490°F) at the air coolant inlet and about 870°C (1600°F) at the air outlet.

Four additional tube positions, not part of the initial 1500-h test matrix, were used for corrosion test specimens for the subsequent 3000-h series. The purpose was to incorporate candidate steam superheater materials, specifically 2 1/4 Cr-1 Mo steel and type 304 stainless steel, into the tube matrix. These materials were used in the form of composite tubes, with the 2 1/4 Cr-1 Mo steel segment operating over a temperature range from 430 to 510°C (800-950°F) and a second segment of type 304 stainless steel operating from 510 to 590°C (950-1100°F).

*Trademark of Alon Processing, Inc., Tarentum, Pa.

Fig. 2. Specimen tube position, alloy, and time test matrix for the 4500-h corrosion test in the FluidDyne AFBC. The "A" signifies an Alonized tube section.

Preparation of Tubes

The compositions of the test alloys are listed in Table 1. The austenitic alloys were procured in the form of commercial-grade 12.7-mm-OD by about 1.6-mm-wall (1/2 by 0.06 in.) tubes, and the chromium-molybdenum steel was drawn to size from heavy-wall 22.2-mm-OD (7/8-in.) boiler tubes.

Table 1. Composition of test alloys exposed
in the FluidDyne AFBC^a

Element	Content, wt %				
	2 1/4 Cr-1 Mo	304	310	316	800H
Fe	95.90	69.40	52.31	65.52	43.83
Cr	2.29	18.60	24.13	17.49	20.51
Ni		9.40	20.88	12.40	33.46
Mn	0.47	1.69	1.54	1.76	0.86
Si	0.23	0.53	0.67	0.52	0.37
Mo	0.96	0.05	0.29	2.20	
C	0.11	0.05	0.058	0.06	0.063
P	0.019	0.022	0.010	0.032	0.13
S	0.015	0.006	0.011	0.022	0.003
Co		0.23			
Cu		0.02	0.10		
Ti					0.49
Al					0.41

^aProducer's analyses.

Because the objective of this program was to test typical materials, the specimen tubes were given no special surface or chemical cleaning treatments.

Certain of the tubes in Fig. 2 are designated by hyphenated compositions. In these cases the in-bed regions of the tubes were made up of 0.15-m (6-in.) segments of differing surface or alloy compositions welded to form one continuous tube. The hyphenated alloy designations denote the respective tube segments. Following the initial 1500-h test, any new tube, even if the composition was uniform (single alloy designations in Fig. 2), was initially divided into four segments and then welded. This provided a test of welds and heat-affected zones and also permitted more precise wall thickness measurements along the tube before exposure. After the initial thickness measurements, the sections were gas tungsten arc welded together with the following filler alloys: type 304 stainless steel with type 308L filler, type 310 stainless steel with type 310 filler, type 316 stainless steel with type 316L filler, and alloy 800H with ERNiCr-3 filler. The temperature for each tube was continuously

monitored at the entrance, approximate midpoint, and exit of the in-bed portion with type K thermocouples spot-welded to the inner wall. See Fig. 3 for additional details.

ORNL-DWG 81-4362

Fig. 3. Tube installation in combustor and nominal thermocouple locations.

The low-temperature composite tubes were prepared in a similar fashion to that just described. Parted sections of both halves were measured and then welded as follows: 2 1/4 Cr-1 Mo to 2 1/4 Cr-1 Mo with 2 1/4 Cr-1 Mo, 2 1/4 Cr-1 Mo to type 304 stainless steel with ERNiCr-3, and type 304 stainless steel to type 304 with type 308L. Because these low-temperature tubes were cooled with high-velocity air, a different attachment method was employed for the thermocouples to minimize fin-cooling effects. At entrance, central, and exit locations slant holes and surface slots were machined to accept 0.76-mm (0.030-in.) stainless-steel-sheathed type K thermocouples, which were subsequently fully welded into place. This scheme provided thermocouple immersion depth judged sufficient for good thermometry.

Methods of Examination

At the conclusion of each test period the selected specimen tubes were removed and visually examined by Fluidyne and/or ORNL representatives at the site. Tubes scheduled for destructive examination were sealed in plastic bags with a dessicant and shipped to ORNL. Before the destructive analysis most tubes were photographed for reference. To characterize the entire tube length three to six full cross sections were removed from each tube. When appropriate, sections of weld areas were also obtained.

Standard metallographic methods were used to prepare specimens for examination. All samples were examined metallographically, and requisite scale and corrosion measurements were made by using a measuring eyepiece, usually at 320X. Wall thickness was measured with a toolmaker's microscope. Representative or unusual specimens were microchemically analyzed by energy-dispersive x-ray microprobe techniques.

OPERATING CONDITIONS AND RUN DATA

The 4500-h experiment at Fluidyne was intended to be operated under tightly controlled bed conditions with a minimum number of shutdowns and restarts. In large measure, this objective was met. In this section we will describe the experimental conditions that obtained during the final 3000 h of testing. The first 1500 h of this experiment has been previously reported.^{2,3} It should be noted that eight high-temperature sample tubes were exposed to both the initial 1500 h and final 3000-h test segments (see Fig. 2).

Normal Operations

The final 3000-h portion of the 4500-h test program was divided into segments of 500, 1000, and 1500 h, and the nominal operating conditions for each segment are given in Table 2. More detailed operating data are presented in Appendix B. Test parameters were recorded at 2-h intervals throughout the experiment and were averaged to yield the results in Table 2. The "measured" and "corrected" coal feed rates reflect an air

Table 2. Nominal operating conditions for the final 3000 h of the 4500-h test

	Average value and standard deviation		
	500-h test	1000-h test	1500-h test
Coal rate, g/s (measured)	6.82 $\pm$ 0.18	6.56 $\pm$ 0.26	5.61 $\pm$ 0.19
(corrected)	5.73	5.93	
lb/h (measured)	54.1 $\pm$ 1.4	52.1 $\pm$ 2.1	44.5 $\pm$ 1.5
(corrected)	45.5	47.1	
Sulfur content, %	3.4	3.5 $\pm$ 0.2	3.4
Sorbent rate, g/s (measured)	2.44 $\pm$ 0.29	3.01 $\pm$ 0.35	2.43 $\pm$ 0.25
(corrected)	2.12	2.84	
lb/h (measured)	19.4 $\pm$ 2.3	23.9 $\pm$ 2.8	19.3 $\pm$ 2.0
(corrected)	16.8	22.6	
Ca/S mole ratio	3.3 ^a	4.2 ^a	3.8 $\pm$ 0.5
Fluidizing velocity, m/s (fps) (based on combustion air)	0.76 (2.5)	0.79 (2.6)	0.73 (2.3)
Bed temperature, °C	891 $\pm$ 1.1	891 $\pm$ 3.9	892 $\pm$ 0.7
°F	1636 $\pm$ 2.0	1636 $\pm$ 7.0	1638 $\pm$ 1.3
Oxygen in flue gas, %	3.2 $\pm$ 0.2	3.4 $\pm$ 0.2	3.4 $\pm$ 0.2
SO ₂ in flue gas, ppm	344 $\pm$ 35	349 $\pm$ 30	340 $\pm$ 25

^aBased on corrected feed rates.

leakage problem in the coal feed system during the first two segments of the test, as discussed in Appendix C. The rate was corrected to account for the elutriation of most of the fines (-100 mesh) from the -4 mesh coal and -16 mesh limestone from the feed. These fines were then captured in the baghouse. Thus, during these first two test segments only the coarser fraction of the coal was actually fed to the combustor.

The air leakage problem also precluded the accurate determination of the ratio of coal and limestone transport air to fluidizing air, the best estimate is 0.15 or lower. The significance of this ratio has been discussed previously.^{2,3} The air leakage was eliminated during the final 1500-h segment by the substitution of a compressed air ejector for the rotary valve, and the air ratio was maintained at 0.15.

The standard deviations of the primary variables in Table 2 (temperature, oxygen, and SO₂) are a clear indication that the fluidized bed was well controlled and generally stable.

Upset Operations

Some unscheduled upsets were experienced during the test program. Those upsets, which required the use of the propane ignition burner to recover bed temperature, are summarized in Appendix D. In each case, oxidizing conditions were maintained in the bed during recovery, and the rate of recovery was closely controlled to avoid any possibility of overheating.

During the first 500-h segment, the otherwise steady operation was hampered by frequent coal feed interruptions (coal plugs), about one to three per 24-h period. These were typically minor inconveniences, causing a momentary drop in bed temperature; recovery was easily achieved without the propane ignition burner once the plug was cleared.

The cause of these interruptions was traced to a deteriorating rotary valve used in the solids feed system. As the test progressed, wear of the rotor increased the tip clearance between the rotor and the housing. This increase in clearance caused a gradual loss in air used to transport the coal and sorbent, resulting eventually in particle saltation and plugging. To avoid this problem, the transport air was adjusted manually to the minimum amount required to avoid slugging flow in the feed line.

A replacement airlock was installed at the beginning of the 1000-h test. Fewer coal feed interruptions were experienced; however, the performance of the airlock deteriorated as in the previous test. After approximately 300 h, the airlock was no longer an effective seal, and the solids transport air had to be adjusted manually as in the previous test period. The feeding problems were successfully solved before the start of the final 1500-h test by using a compressed air ejector, eliminating the need for a rotary valve entirely.

In summary, the upsets described do not appear significant in their potential effects on corrosion performance, and test conditions were generally recovered in 15 to 40 min in each case. The more important concern was the error in measured solids feed rates.

Trimmer Tube Failure

During the 1000-h test, it was necessary to remove one of two type 304 stainless steel water tubes used for gross bed heat extraction,

approximately 300 h into the test. The decision to interrupt the test and remove a water-cooled tube was based on a desire to restore full bed temperature control to the trimmer circuit. With the previous tube arrangement the heat balance was marginal and slight variations in heat extraction during the 1000-h test due to increased moisture in the coal, moisture in the combustion air, and variations in the sorbent feed rate caused the bed temperature to fluctuate between 880 and 890°C (1620 and 1635°F). This problem was not experienced during the 500-h test.

The water-cooled tube was not physically removed but was switched from water to air service after the combustor had been cooled to a safe working temperature. After 700 h into the test, this tube failed at midspan and had to be replaced. The tube was sent to ORNL for a detailed metallographic examination, and structural analyses were performed by both ORNL and FluidDyne personnel to ascertain the probable cause of failure. A summary of the analysis is presented in Appendix E. The conclusion of this analysis was that the constraints on the ends of the tube did not slip as designed to accommodate thermal expansion, and the resultant stresses led to eventual rupture of the tube. There was no evidence that eventual failure of this tube was caused by the earlier water service.

The failed tube was replaced with a new type 304 stainless steel tube of the same composition with the same metallurgical history. This tube performed satisfactorily over the remaining 1800 h under the same operating conditions experienced by the tube that failed. The appearance of this tube at the conclusion of the test showed no evidence that would distinguish it from the other air trimmer tubes exposed to the fluidized bed environment.

The other upset conditions listed in Appendix D were from a variety of causes, the most common being feed interruptions due to plugging. These longer upsets required use of the above-bed propane burner to reignite the bed. This was always done with the bed fluidized to prevent direct flame impingement on the in-bed tubes.

Tube Temperatures

Twenty-eight 12.7-mm (1/2-in.) thin-wall metal tubes were supplied by ORNL. Twenty of these tubes (high alloy) were to be tested with a hot-end

temperature not to exceed 870°C (1600°F), while the remaining eight tubes (low alloy) were to be tested with a hot-end temperature not to exceed 595°C (1100°F). Eight of the high-alloy tubes had been tested during the previous 1500-h test,^{2,3} while the other twenty were new. Each tube was 0.97 m (38 in.) long and typically consisted of shorter segments welded together. Three type K (Chromel vs Alumel) thermocouples were either spot-welded to the inner wall of each tube or inlaid in the tube wall, as shown in Fig. 3. The dimensions shown are referenced from the cold end of the sample tube (the left end).

Of the 20 higher temperature tubes used in the test, 6 were instrumented with thermocouples inlaid in the tube wall, and the remainder had thermocouples spot-welded to the inside surface of the tube as in previous tests.² The thermocouples on three of the tubes remaining from the earlier 1500-h test (tubes 4B, 4C, and 8D) had become detached and could not be rewelded to the tube inner wall because of the oxide scale. However, since air to all tubes was supplied from a common manifold and the amount of cooling was slight for these high-temperature tubes, temperatures for these tubes could be estimated with reasonable accuracy. Each of the eight lower temperature tubes tested was instrumented with thermocouples inlaid in the tube wall.

Tube wall temperatures for the high-temperature and low-temperature tubes were controlled by metering the cooling air flow to each tube from a common manifold, which was maintained at a constant pressure.

Thermocouple outputs were recorded automatically every 2 h with a Vidar 5402 digital data acquisition system. Millivolt outputs were recorded on punch tape and then converted to temperature by use of a polynomial curve fit equation. Average tube temperatures for the high- and low-alloy sample tubes are presented in Figs. 4 through 9 and in Tables 3 through 5. Some of the thermocouples failed in service or were found unreliable and the data rejected. The tube temperatures plotted in Figs. 4 through 9 show variations at a given axial position of about 50°C, with extreme variations up to 90°C. The variation of temperature with time, as indicated by a given thermocouple, was quite small and these variations are summarized in Tables 3 through 5 as values of standard deviation.

Fig. 4. Average temperatures for the high-temperature tubes over the 500-h period from 1500 to 2000 h. Specimen identification is keyed to Figs. 1 and 2.

Fig. 5. Average temperatures for the high-temperature tubes over the 1000-h period from 2000 to 3000 h. Specimen identification is keyed to Figs. 1 and 2.

Fig. 8. Average temperatures for the low-temperature tubes over the 1000-h period from 2000 to 3000 h. Specimen identification is keyed to Figs. 1 and 2.

Fig. 9. Average temperatures for the low-temperature tubes over the 1500-h period from 3000 to 4500 h. Specimen identification is keyed to Figs. 1 and 2.

Table 3. Average tube temperatures over the 500-h period from 1500 to 2000 h^a

Tube	Temperature and standard deviation, °C (°F)		
	Cold end	Middle	Hot end
<u>High-temperature tubes</u>			
2C	853 ± 2.6 (1568 ± 4.7)	865 ± 3.7 (1589 ± 6.7)	
5D	806 ± 2.2 (1482 ± 3.9)	837 ± 1.6 (1538 ± 2.8)	865 ± 0.7 (1589 ± 1.2)
9D		847 ± 2.1 (1557 ± 3.7)	856 ± 6.2 (1752 ± 11.1)
3C	842 ± 1.7 (1548 ± 3.0)	806 ± 6.2 (1482 ± 11.2) ^b	859 ± 3.3 (1578 ± 5.9)
3B	817 ± 2.9 (1503 ± 5.2)	848 ± 1.5 (1558 ± 2.7)	863 ± 1.5 (1585 ± 2.7)
6C	853 ± 4.9 (1568 ± 8.9)		853 ± 2.8 (1568 ± 5.1)
7B			863 ± 2.9 (1586 ± 5.2)
8C	776 ± 4.7 (1429 ± 8.5) ^b	848 ± 3.6 (1558 ± 6.5)	866 ± 1.4 (1591 ± 2.6)
9C		856 ± 2.5 (1573 ± 4.5)	871 ± 3.6 (1599 ± 6.4)
9B	828 ± 1.6 (1522 ± 2.8)	858 ± 3.8 (1577 ± 6.9)	866 ± 2.3 (1590 ± 4.1)
2B		847 ± 8.3 (1556 ± 14.9)	
6B	828 ± 2.9 (1523 ± 5.3)	825 ± 3.3 (1517 ± 5.9) ^b	883 ± 1.2 (1622 ± 2.2)
9A	828 ± 1.2 (1522 ± 2.1)	862 ± 1.6 (1583 ± 2.8)	866 ± 1.2 (1590 ± 2.2)
<u>Low-temperature tubes</u>			
2D	456 ± 11.1 (852 ± 20.0)	485 ± 12.7 (950 ± 22.9)	
3A	456 ± 11.3 (853 ± 20.3)		526 ± 12.9 (978 ± 23.2)
8B	514 ± 9.6 (957 ± 17.2)	521 ± 10.3 (970 ± 18.6)	551 ± 10.9 (1023 ± 19.7)
7D	436 ± 9.0 (817 ± 16.2)	484 ± 9.0 (904 ± 16.2)	

^aTubes 4B, 4C, and 8D not instrumented.

^bMeasured temperature appears low compared with expected value.

Table 4. Average tube temperatures over the 1000-h period from 2000 to 3000 h^a

Tube	Temperature and standard deviation, °C (°F)		
	Cold end	Middle	Hot end
<u>High-temperature tubes</u>			
2C	831 ± 6.5 (1527 ± 11.7)	817 ± 17.9 (1502 ± 32.2) ^b	866 ± 3.6 (1591 ± 6.4)
5D	799 ± 4.7 (1470 ± 8.5)	838 ± 3.2 (1540 ± 5.7)	864 ± 3.1 (1587 ± 5.5)
9D		841 ± 4.6 (1546 ± 8.2)	849 ± 5.3 (1560 ± 9.4)
3C	846 ± 4.6 (1554 ± 8.3)		855 ± 5.1 (1571 ± 9.1)
3B	818 ± 3.9 (1504 ± 7.0)	852 ± 3.2 (1566 ± 5.7)	863 ± 2.9 (1586 ± 5.2)
6C	854 ± 3.3 (1569 ± 5.9)		847 ± 3.5 (1557 ± 6.3)
7B			857 ± 4.2 (1574 ± 7.5)
8C	763 ± 7.7 (1406 ± 13.8) ^b	854 ± 4.1 (1570 ± 7.3)	862 ± 3.3 (1583 ± 5.9)
9C		853 ± 4.4 (1568 ± 8.0)	861 ± 3.6 (1582 ± 6.6)
9B	819 ± 11.0 (1506 ± 19.8)	848 ± 7.6 (1559 ± 13.7)	865 ± 3.0 (1589 ± 5.4)
2B		835 ± 9.8 (1535 ± 17.7)	
6B	814 ± 4.2 (1497 ± 7.6) ^b	823 ± 6.7 (1514 ± 12.0) ^b	
9A	812 ± 8.8 (1493 ± 15.9)	867 ± 3.9 (1592 ± 7.0)	873 ± 3.2 (1603 ± 5.7)
<u>Low-temperature tubes</u>			
2D	439 ± 10.2 (823 ± 18.3)	487 ± 10.7 (909 ± 19.1)	
3A	472 ± 7.0 (882 ± 12.6)	520 ± 8.6 (968 ± 15.4)	557 ± 9.2 (1035 ± 16.6)
8B	441 ± 16.1 (825 ± 29.0)	502 ± 7.5 (936 ± 13.4)	543 ± 8.2 (1010 ± 14.8)
7D	432 ± 10.2 (809 ± 18.3)	506 ± 8.3 (942 ± 15.0)	

^aTubes 4B, 4C, and 8D not instrumented.

^bMeasured temperature appears low compared with expected value.

Table 5. Average tube temperatures over the 1500-h period from 3000 to 4500 h^a

Tube	Temperature and standard deviation, °C (°F)		
	Cold end	Middle	Hot end
<u>High-temperature tubes</u>			
2C	825 ± 6.7 (1517 ± 3.1)	839 ± 3.3 (1542 ± 5.9)	867 ± 1.6 (1592 ± 2.9)
5D	798 ± 2.6 (1469 ± 4.6)	841 ± 4.1 (1545 ± 7.3)	859 ± 2.0 (1578 ± 3.6)
9D		837 ± 2.8 (1539 ± 5.0)	853 ± 1.3 (1567 ± 2.4)
3C	846 ± 2.4 (1554 ± 4.4)		
3B	818 ± 1.9 (1505 ± 3.4)	849 ± 3.1 (1560 ± 5.6)	863 ± 1.6 (1585 ± 2.9)
6C	849 ± 1.5 (1560 ± 2.7)		844 ± 1.3 (1551 ± 2.3)
7B	738 ± 12.3 (1361 ± 22.1) ^b	815 ± 2.7 (1501 ± 4.8)	840 ± 5.7 (1544 ± 10.3)
8C	812 ± 3.3 (1494 ± 6.0)	862 ± 4.4 (1583 ± 8.0)	865 ± 2.2 (1589 ± 4.0)
9C		845 ± 2.3 (1554 ± 4.1)	856 ± 1.3 (1573 ± 2.4)
9B	783 ± 6.4 (1442 ± 11.5) ^b	836 ± 5.3 (1536 ± 9.6)	861 ± 1.6 (1582 ± 2.8)
2B		821 ± 4.4 (1510 ± 7.9)	
9A	837 ± 9.9 (1538 ± 17.8)		867 ± 2.2 (1592 ± 3.9)
<u>Low-temperature tubes</u>			
2D	435 ± 7.6 (815 ± 13.7)	537 ± 15.1 (998 ± 27.2)	597 ± 8.1 (1106 ± 15.1)
3A	463 ± 6.4 (865 ± 11.5) ^c	5C1 ± 7.9 (933 ± 14.2)	578 ± 7.5 (1072 ± 13.4)
8B	493 ± 2.7 (919 ± 14.1)	511 ± 5.8 (951 ± 10.4)	580 ± 6.3 (1076 ± 11.3)
7D	440 ± 14.1 (824 ± 25.3)	510 ± 13.4 (950 ± 24.2)	

^aTubes 4B, 4C, and 8D not instrumented.

^bMeasured temperature appears low compared with expected value.

^cThermocouple burned out after about 200 h.

The temperature variation among tubes is most likely due to a combination of effects: local variations in bed-side heat transfer coefficient caused by local flow patterns in the bed, local variations in bed temperature, slight differences in cooling air flow, and differences in thermocouple installation.

Local variations in bed-side heat transfer coefficient, which could be as much as $\pm 10\%$, can be expected to produce variations in tube temperature for the high-temperature tubes of up to $\pm 11^\circ\text{C}$ and up to $\pm 22^\circ\text{C}$ for the low-temperature tubes. Variations in bed temperature and cooling air flow are of secondary importance and can be expected to produce variations in tube temperature of not more than 6°C for either case.

The variations that would result from the differences in thermocouple installation cannot be easily assessed since the measured temperature depends totally on the thermal connection between the thermocouple and the tube wall and the location of the thermoelectric junction with respect to that thermal connection because of the fin-cooling effect. Errors could easily reach 30°C (54°F) [but always to lower temperatures] due to fin-cooling.

Although these temperature variations appear to be large on the expanded scales of Figs. 4 through 9, they are representative of the control and accuracy that can be reasonably achieved at the temperature levels involved for an AFBC experiment outside a research laboratory. Furthermore, as will be discussed, even with the consistent increase in temperature from the inlet to the outlet end of each tube, the change in corrosion response along the tube was no more significant than changes resulting from other effects.

TEST RESULTS

Low-Temperature Tubes

Composite tubes of type 304 stainless steel and 2 1/4 Cr-1 Mo steel were tested at four positions in the bed. The wall temperature of the chromium-molybdenum tube increased from 430°C to 510°C in the direction of air flow, and the type 304, from 510 to 590°C . Visual examinations of

these tubes after 500, 1000, 1500, and 3000 h showed the exterior surface to be covered with a tenacious, nearly black, generally smooth deposit of calcium sulfate. In isolated areas this deposit had spalled, exposing oxide scale and occasionally the base metal. There was no visual evidence of any unusual corrosion damage.

Detailed examination of metallographic specimens removed from the low-temperature composite tubes revealed generally good performance of the materials in 3000 h of testing. Typical microstructures for the high temperature end of the 2 1/4 Cr-1 Mo steel tube after 3000 h are shown in Fig. 10. The inside tube surface (air side) shows a uniform three-layered oxide film, which in 3000 h reached a thickness of about 50 μm . Under the same temperature and time conditions, the external (fire-side) surface shows only two mixed oxide layers, but their total thickness is about twice that of the air-side oxide. The interface between the inner oxide layer and the metal is noticeably more irregular on the fire side than on the air side.

The type 304 stainless steel showed only a superficial oxide film on the inner and slightly thicker on the outer surface (Fig. 11). However, grain boundary oxidation on the fire side reached a maximum depth of 20 μm , and occasional subsurface sulfide inclusions were observed to depths of 25 μm [Fig. 11(b)]. Microprobe analyses indicate that these inclusions are manganese rich. The ERNiCr-3 transition weld joint between the chromium-molybdenum steel and the stainless steel was also examined. No attack of heat-affected zone or weld metal was found. Also, it should be stressed that corrosion did not vary with circumferential position for these low-temperature tube samples.

Results of the measurements of the scale thickness and intergranular attack on the full set of low-temperature tubes are shown in Tables 6 and 7. The values reported are averages of eight measurements of the maximum of each feature around the circumference of the specimen, the standard deviation, and the maximum of each set of eight measurements. For the 2 1/4 Cr-1 Mo specimens, the outside scale thicknesses for both fire side and air side are shown; however, no indications of intergranular corrosion or oxide finger penetration were found. The air-side oxide scale on the

50 μm

Fig. 10. Specimen 7D of 2 1/4 Cr-1 Mo steel exposed for 3000 h at about 510°C in the Fluidyne AFBC. (a) Air side. (b) Fire side. Note the three-layer oxide on the air side as opposed to the two layers of oxide under the CaSO_4 deposit on the fire side.

50 μ m

Fig. 11. Type 304 stainless steel specimen 7D exposed for 3000 h at 590°C in the FluidDyne AFBC. (a) Air side. (b) Fire side. Note the very thin oxide film barely visible on the air side. The dark band is a gap caused by shrinkage of the mounting material. On the fire side, the interface between the oxide scale and the sulfate deposit is difficult to discern.

Table 6. Corrosion measurements on the 2 1/4 Cr-1 Mo steel portions of low-temperature composite tubes exposed in the FluidDyne AFBC

Sample ^a	Exposure (h)	Removal time ^b (h)	Oxide scale thickness, ^c μm						Wall thickness, μm	
			Fire side			Air side			av	s.d.
			av	s.d.	max	av	s.d.	max		
3A-1	500	2000	20	5	28	20	10	30	1747	41
-2			40	13	68	30	10	43	1723	54
8B-1	500	2000	15	7	27	24	7	31	1763	14
-2			28	9	47	40	7	48	1764	7
3A-1	1000	3000	74	46	113	37	10	54	1726	21
-2			82	41	152	54	14	76	1727	37
8B-1	1000	3000	23	12	44	19	7	25	1774	12
-2			31	4	34	40	4	45	1763	13
8B-1	1500	4500	42	12	60	28	5	35	1761	19
-2			37	7	45	27	9	36	1761	20
-3			56	29	101	56	6	66	1749	30
7D-1	3000	4500	91	48	166	68	18	95	1729	28
-2			50	14	66	27	8	36	1747	23
-3			58	30	110	31	8	43	1759	14

^aThe tube is designated by the row and column in the test bed. For each tube the sample number increases from the inlet end; the temperature increases smoothly from about 430°C at the inlet to about 510°C at the transition to the stainless steel.

^bTime during 4500-h test when sample was removed.

^cFor each side of each section, the average of eight measurements taken around the circumference, the standard deviation of these measurements, and the greatest measurement are given.

Table 7. Corrosion measurements on the type 304 stainless steel portions of low-temperature composite tubes exposed in the Fluidyne AFBC

Sample ^a	Exposure (h)	Removal time ^b (h)	Fire-side corrosion measurements, ^c μm						Wall thickness, μm	
			Oxide scale thickness			Intergranular corrosion			av	s.d.
			av	s.d.	max	av	s.d.	max		
3A-3	500	2000	4.0	1.4	4.6	9.0	5.3	18.6	1673	37
-4 ^d			5.0	1.3	6.5	0	0	0		
-5 ^d			3.6	0.8	4.6	5.4	3.8	11.1		
-6			4.4	0.7	5.5	7.2	2.8	11.2	1696	44
8B-3	500	2000	6.0	0.8	6.8	4.6	0.8	5.7	1700	24
-4 ^d			4.0	1.1	6.0	3.5	0.8	4.2		
-5 ^d			6.4	1.2	8.3	5.2	1.6	7.4		
-6			3.5	1.0	5.7	4.1	2.0	6.9	1703	17
3A-3	1000	3000	3.7	0.8	4.7	6.1	2.1	9.5	1690	40
-4 ^d			4.4	1.0	6.2	2.6	1.5	3.7		
-5 ^d			4.4	0.4	4.9	6.8	2.1	10.8		
-6			4.7	0.9	6.3	7.7	3.8	10.7	1700	52
8B-3	1000	3000	5.2	1.9	7.3	9.9	2.9	14.9	1697	51
-4 ^d			7.2	3.4	15.3	0	0	0		
-5 ^d			5.3	1.2	7.0	7.5	1.1	8.9		
-6			6.1	1.2	7.9	11.8	8.0	19.6	1707	51
8B-4	1500	4500	6.2	1.8	10.4	7.5	2.4	10.4	1692	49
-5 ^d			3.5	1.4	5.5	4.4	1.8	8.1		
-6 ^d			5.6	1.3	7.3	4.1	2.1	7.1		
-7			4.5	0.5	5.2	4.0	2.1	7.9	1700	52
7D-4	3000	4500	5.6	2.2	9.8	9.6	3.8	15.4	1683	46
-5 ^d			5.1	1.6	7.9	6.6	2.3	10.7	1567	12
-6 ^d			5.7	1.2	7.0	6.9	2.7	13.0	1575	12
-7			6.4	3.3	14.2	9.9	3.9	16.0	1677	36

^aThe tube is designated by the row and column in the test bed. For each tube the sample number increases from the transition joint midway between the inlet and the outlet. The temperature increases smoothly from 510°C at the midpoint to about 590°C at the outlet.

^bTime during 4500-h test at which sample was removed.

^cFor each feature the average of eight measurements taken around the circumference, the standard deviation of these measurements, and the greatest measurement are given.

^dSurface machine ground before exposure in AFBC.

type 304 stainless steel samples was so thin and spotty that its measurement was not meaningful. The second and third of the four entries for each of the type 304 stainless steel tubes were on sections of the tube that had been machine-ground before exposure in the AFBC and were located during the test about 15 mm from the companion first and fourth specimens.

The results in Table 6 indicate significant variation in oxide scale thickness on both air side and fire side of the 2 1/4 Cr-1 Mo steel. Except for the 3000-h 7D specimen, there is a general trend toward thicker oxide on the hotter portion of the tube (higher numbered sample). The inconsistent average and maximum fire-side values for tube 7D probably indicate scale spallation. Though not shown in Table 6, measurements of tube wall thickness before and after exposure indicate that metal loss is consistent with the observed maximum scale thicknesses within the precision of the respective measurements and wall variability. Within the limits of these data, a general increase in scale thickness with time can be noted; however, the variability of the data obscures the time dependence.

For the low-temperature type 304 stainless steel samples the results in Table 7 show that oxide scale thickness and intergranular corrosion penetration are so low that no time-dependent relationship would be resolved for times from 500 to 3000 h. The oxide scale was moderately uniform, as indicated by the small standard deviations, but intergranular corrosion was more varied, though still slight. In general, the specimens with machine-ground surfaces experienced slightly lower intergranular corrosion attack than did the unground ones. As with the 2 1/4 Cr-1 Mo steel samples, wall thickness measurements indicated that no significant metal loss occurred during the 3000-h test.

High-Temperature Tubes

Under normal well-controlled operating conditions, tubes from the 16 high-temperature positions maintained a similar visual appearance from position to position, and the appearance did not change noticeably from 500 to 4500 h. These tubes, which operated at metal temperatures between 810 and 870°C (1490 and 1600°F), were covered by a tenacious, paintlike

surface deposit of calcium sulfate. In contrast with the deposit on the low-temperature tubes, these deposits ranged in color from dark olive through red to tan. Also, the deposit appeared to be in discontinuous layers, suggesting a cyclic buildup or spallation and re-formation. On some areas the surface deposit had spalled, probably on cooling, exposing the oxide scale and, on occasion, the underlying base metal. The existence of this sulfate deposit strongly indicates that erosive wear by the bed matrix was not a significant factor in the corrosion of the in-bed tubes.

Typical microstructures at the external surface of type 316 stainless steel and alloy 800H are shown in Fig. 12. The calcium sulfate deposits on these tubes are built up of numerous concentric layers or bands, which could have formed only by deposition from a gas or liquid phase. The banded structure is obviously associated with periodic changes in bed operating conditions, such as startup and shutdown. Typically, both the 300 series stainless steels and alloy 800H showed grain boundary attack to depths of 20 to 40 μm beneath the oxide scale. Beneath the intergranular attack were inclusions of manganese-chromium sulfide, which seemingly decorated grain boundaries.

The complete set of corrosion measurements for the high-temperature tubes from the 4500-h test is listed in Appendix F. Table 8 lists the averages of the individual measurements from Appendix F. It must be emphasized that the individual measurements vary widely from place to place on a single specimen and from specimen to specimen (ranging from 0.5 to 2 times the reported average). The results are still being studied in an attempt to determine whether these variations follow a pattern or are totally random. It should also be stressed that for each microscopic field of view, the deepest or thickest single feature was selected for measurement; thus, the values reported are the averages of all "worst cases" and would therefore overestimate corrosion damage compared with some other methods of measurement, such as descaled weight loss. The results in Table 8 are grouped by alloy and exposure time, and, as in the previous tables, the third column indicates the time at which that specimen tube was removed and examined. This table reports the average adherent oxide scale thicknesses, the average of the maximum subscale

50 μm

Fig. 12. Extent of fire-side corrosion after 4500-h exposure in AFBC at 810 to 870°C. (a) Sulfide inclusions are conspicuous in the stainless steel. (b) However, they are less evident in the alloy 800H.

Table 8. Fire-side corrosion measurements on high-temperature tubes exposed in the FluidDyne AFBC over the range 810 to 870°C (1400–1600°F)^a

Tube	Exposure (h)	Removal time ^b (h)	Oxide scale thickness, μm			Intergranular corrosion depth, μm			Depth, μm, of sulfide inclusions		
			av	Pooled s.d.	max	av	Pooled s.d.	max	av	Pooled s.d.	max
Type 304 stainless steel											
9A	500	500	5.6	2.0	10.6	18.2	5.6	34.5	28.1	4.1	41.5
2C	1000	3000	10.3	4.0	23.7	30.3	19.8	104.6	48.1	21.0	126.4
2C	1500	4500	9.3	5.2	30.9	47.2	29.0	105.3	72.5	35.7	145.3
9A	3000	4500	9.5	5.6	26.9	32.7	18.8	79.4	66.3	23.4	121.6
9D	4000	4500	8.0	2.9	15.4	29.6	6.5	37.4	55.0	10.2	74.9
Type 310 stainless steel											
9B	500	500	5.1	1.7	7.8	15.6	6.3	33.7	26.58	5.8	38.4
9D	500	500	5.4	1.7	9.2	13.5	4.7	21.4	30.8	7.9	45.0
9C	1000	1500	5.8	1.9	10.2	23.6	10.1	40.7	36.5	12.2	55.0
6B	1500	1500	6.1	1.5	10.7	19.0	7.1	31.0	37.0	11.2	52.8
6B	3000	4500	6.7	2.7	15.8	27.0	13.3	86.1	44.7	13.5	94.5
9C	3000	4500	6.2	2.3	13.0	29.9	12.2	57.2	57.2	9.0	70.8
3C	4500	4500	12.4	3.4	21.5	39.1	19.5	99.0	61.7	17.2	81.7
8D	4500	4500	7.1	1.3	9.1	32.6	12.2	82.9	58.4	18.8	102.2
Type 316 stainless steel											
8C	500	2000	5.4	2.1	11.5	13.5	8.1	29.7	19.0	8.7	48.2
8C	1000	1500	10.4	4.8	28.0	31.0	12.4	48.9	47.7	11.1	73.5
8C	1000	3000	11.2	4.6	32.9	34.3	17.8	100.3	46.5	20.3	119.3
3B	1500	1500	8.5	1.6	12.4	25.4	8.0	50.1	47.0	8.4	62.1
8C	1500	4500	9.6	2.9	14.1	59.7	23.9	111.3	78.0	24.0	137.8
3B	3000	4500	16.9	9.7	46.7	31.9	16.8	68.1	47.0	19.5	87.7
4C	4500	4500	11.3	3.8	22.6	33.1	11.1	60.7	60.0	11.4	92.8
6C	4500	4500	12.3	4.0	22.6	37.7	15.6	89.0	62.2	14.6	104.6
Alloy 800H											
2C	500	500	5.8	1.8	8.5	30.3	10.2	46.4	38.9	11.0	80.2
9A	500	500	6.1	1.2	8.2	28.9	8.4	43.9	35.9	10.9	60.2
9D	500	500	5.0	1.8	9.4	19.5	18.0	41.3	26.4	16.6	45.5
9B	1000	1500	7.1	1.2	9.6	39.9	15.1	60.7	52.5	12.0	78.5
5D	1500	1500	6.9	1.9	11.7	47.2	27.4	84.2	58.9	26.5	88.9
2C	1500	2000	8.0	5.9	13.6	80.0	15.3	119.7	86.6	13.2	115.6
8C	1500	4500	5.4	2.2	8.0	61.3	29.5	112.0	86.1	24.8	136.6
5D	3000	4500	7.9	4.0	18.0	77.9	23.9	130.8	92.2	17.6	146.6
7B	3000	4500	10.1	3.0	14.9	90.4	23.5	135.2	102.2	24.0	142.2
2B	4500	4500	10.8	2.5	16.5	77.3	20.9	133.1	92.2	17.6	146.6
4B	4500	4500	11.4	2.2	15.7	101.5	18.8	146.4	117.1	18.1	156.3

^aFor each feature, values reported are averages of eight measurements taken around the circumference of the sample averaged with those of two to five additional samples from the same tube, the pooled standard deviation ($\sqrt{\sum \sigma_i^2/m}$, for m specimens), and the greatest value among all measurements.

^bTime during 4500-h test at which sample was removed.

corrosion penetrations, which occurred primarily as intergranular attack, and the average maximum depth of identifiable sulfide inclusions. Also recorded are the pooled standard deviations and the maximum values found for each feature from all specimens from that sample tube. Although the maximum intergranular corrosion and deepest sulfide inclusion generally occurred at the same point on any tube, such points did not correlate with circumferential or longitudinal position from sample to sample or from tube to tube. Examination of the complete set of data indicates that the corrosion response does not depend uniquely on temperature over the range 810 to 870°C (1490–1600°F). The data suggest that other factors, such as bed design or operation, must act in concert with temperature to determine corrosion response.

The results in Table 8 show that for all alloys tested the oxide scales are moderately thin, considering the test temperature. However, because the variability is rather large and the observed thicknesses do not increase regularly with exposure time, spallation during exposure is likely. Also, a number of samples show areas with no oxide scale, indicating that it had probably spalled on the final cooling. These "zero" thickness areas were not used in computing the average scale thickness because they would bias the results too strongly. It should be noted that pretest wall thickness measurements (by tube wall micrometer) were made on all samples inserted into the experiment at the 1500-h point and later. These measurements were then compared with microscopic measurements after the exposure. However, the differences between the measurements were generally within the combined uncertainties in the measurements. The data suggest a metal loss of less than 50 μm , and such a loss is consistent with maximum thicknesses of oxide scale.

Intergranular corrosion observed in this test generally shows a square-root-of-time dependence, as depicted in Fig. 13, for each alloy class. The area bounded by the solid lines in Fig. 13 represents the minimum and maximum corrosion attack for the austenitic alloys. The dashed lines define the same for the alloy 800H, which contains higher concentrations of chromium and nickel. A comparison of these two regions suggests that alloy 800H is attacked more rapidly along grain boundaries than are the austenitic materials. A similar exercise can be performed

Fig. 13. Average corrosion penetration versus square root of time for alloy specimens exposed in the Fluidyne AFBC at 810 to 870°C. The solid lines encompass the response of the stainless steels, and the dashed lines similarly represent that of alloy 800H.

for each of the individual austenitic materials. This effort reveals that type 310 stainless steel has the lowest variation in corrosion response while showing possibly the lowest rate of attack.

The data in Fig. 13 are plotted without regard to the particular time interval in which the specimen was in the 4500-h experiment. At exposure times of 1000 and 1500 h inconsistencies in the data are evident for types 304 and 316 stainless steel samples, especially. For example, type 316 sample 8C after 1500 h had an average corrosion depth of 60 μm , but samples 4C and 6C, exposed for 4500 h at nominally the same temperature, averaged only about 36 μm of penetration (see Table 8). Similar inconsistencies are found for the other alloys as well. Explanations for

these outliers are still being sought but are not yet clear. However, as will be pointed out subsequently, the bed is not homogeneous and the environment in certain areas appears to be overly aggressive.

An additional important observation is that a linear relationship appears to hold for the depth of sulfide as a function of intergranular corrosion depth, as shown in Fig. 14. For all the alloys, the sulfide inclusions are 20 to 30 μm deeper than the general grain boundary penetrations. These data clearly show that sulfur is penetrating each alloy in advance of the grain boundary corrosion.

Fig. 14. Relationship between average depth of sulfide inclusions and intergranular corrosion depth for all high-temperature specimens in the FluidDyne test.

Alonized Specimens of Alloy 800H and Type 310 Stainless Steel

The behavior of the Alonized samples, which were the middle sections of several of the samples exposed for the full 4500 h, is difficult to assess. The outer aluminum-rich layer tended to crack and spall on both fire side and air side. The aluminum diffusion zone, in general, appeared to be corrosion resistant but had been penetrated by sulfur, as evidenced by sulfide inclusions in the base metal. In localized and random areas the diffusion zone itself suffered attack from sulfidation-oxidation. Figure 15 shows representative microstructures of adjacent samples on an Alonized type 310 stainless steel tube, which show marked differences in the extent of attack. The areas of extensive attack could represent defects in the original coating. Conversely, they may represent incipient breakaway corrosion. In any event, we felt that meaningful corrosion penetration measurements could not be made on these specimens because the original surface could not be identified or located and the attack was so variable and seemingly random. In view of these data, we can neither recommend nor discourage the in-bed use of aluminized coatings.

Corrosion Response Based on Tube Wall Thickness Measurements

Micrometer measurements of the wall thickness were made on all specimens inserted into the experiment at the 1500-h point and later. Measurements were made at eight evenly spaced points around the circumference of the tube section about 6 mm (1/4 in.) from the machined end that would ultimately be welded to form the final tube specimen. This procedure yielded 48 discrete measurements per tube at identified locations. We intended to measure at very nearly the same locations after exposure, but this was not totally successful because of the metallographic procedures employed. Since all tube samples of a given alloy were from a common heat of material, the pretest measurements generally formed a tight grouping about a mean with a standard deviation of about 25 μm or less for each material.

After exposure, tube wall thickness was measured at eight evenly spaced locations around the circumference for each metallographically prepared sample by using a toolmaker's measuring microscope. The thickness

Fig. 15. Fire-side corrosion of Alonized type 310 stainless steel after AFBC exposure for 4500 h. The two areas pictured are nearly adjacent fields of view on a single specimen and illustrate extremes in behavior.

was taken to be the metal beneath the oxide scales on the air side and fire side, *neglecting intergranular corrosion or subsurface sulfides*. Also since the metal-scale interface was generally not smooth, we estimated the average interface in the field of view. Because the attempt to match the results before and after exposure point by point proved unsuccessful, the eight measurements for a given specimen were averaged. The tabulated results are given in Appendix F for each specimen of each tube and the grand averages for the complete tube are plotted in Figs. 16 through 20 for comparison with the calculated mean pretest thickness for each alloy. Also shown in the figures are the wall thickness ranges corresponding to one and two standard deviations, σ , around the pretest mean. Note that the 2σ limit corresponds to a wall thickness change of about $50\text{ }\mu\text{m}$ (2 mils).

Fig. 16. Average wall thickness of 2 1/4 Cr-1 Mo steel tubes after exposure for the indicated time in the FluidDyne AFBC. Also shown are the mean and σ and 2σ below the mean of all pretest measurements. The time at which each specimen was removed from the AFBC is given in parentheses.

Fig. 17. Average wall thickness of low- and high-temperature type 304 stainless steel tubes after exposure for the indicated time in the Fluidyne AFBC. Also shown are the mean, σ above the mean, and σ and 2σ below the mean of all pretest measurements. The time at which each specimen was removed from the AFBC is given in parentheses.

Fig. 18. Average wall thickness of high-temperature type 310 stainless steel tubes after exposure for the indicated time in the Fluidyne AFBC. Also shown are the mean, σ above the mean, and σ and 2σ below the mean of all pretest measurements. The time at which each specimen was removed from the AFBC is given in parentheses.

Fig. 19. Average wall thickness of high-temperature type 316 stainless steel tubes after exposure for the indicated time in the FluidDyne AFBC. Also shown are the mean, σ above the mean, and σ and 2σ below the mean of all pretest measurements. The time at which each specimen was removed from the AFBC is given in parentheses.

Fig. 20. Average wall thickness of high-temperature alloy 800H tubes after exposure for the indicated time in the FluidDyne AFBC. Also shown are the mean, σ above the mean, and σ and 2σ below the mean of all pretest measurements. The time at which each specimen was removed from the AFBC is given in parentheses.

The average wall thicknesses for the low-temperature 2 1/4 Cr-1 Mo steel tubes are shown in Fig. 16. Inspection of the individual measurements in Table 6 suggests that the increasing temperature along a specimen tube had no consistent effect on measured wall thickness, and the specimen averages in Table 6 indicate no significant time dependence. The wall thicknesses after exposure are up to 70 μm less than the pretest mean and are in approximate agreement with losses calculated from the oxide scale thickness.

For type 304 stainless steel, Fig. 17 shows results for both the low-temperature ($\sim 590^\circ\text{C}$) and high-temperature ($\sim 870^\circ\text{C}$) samples. At the higher temperature, the wall thickness of the 3000- and 4000-h specimens was reduced more than the -2σ limit. Tubes exposed for 1000 and 1500 h lie on the -2σ limit. It must be stressed, however, that the values reported make no distinction between fire- and air-side effects, and the 870°C (1600°F) operating temperature approaches the scaling limit for this steel in air. The set of lower temperature tubes shows wall thicknesses closely grouped within about one standard deviation out to the maximum exposure time of 3000 h.

The results for type 310 stainless steel, Fig. 18, show a very tight grouping about the pretest mean value with only tube 4500-3C outside the 1σ band.

Type 316 stainless steel, Fig. 19, shows considerable scatter in post-test wall thickness, both above and below the pretest mean. Samples tested 1000, 1500, and 3000 h lie below the -2σ limit. One 3000-h specimen (sample 9B) was much more severely corroded than the rest of the type 316 stainless steel population, as discussed in the next section. It should be noted that the four samples that lie 1σ or more above the mean pretest value were all prepared at the outset of this program while the five samples that lie below the mean were prepared and inserted at the 1500-h point or beyond. Thus, it is possible that the former, though from the same heat, were from a different draw of tubing and are not fairly represented by the mean. Unquestionably, the latter group is fairly represented by the mean because it provided the sample of pretest values, and the small standard deviation suggests a common population.

The results for alloy 800H, Fig. 20, approximate those for the type 316 stainless steel with respect to scatter. Also, all samples that lie above the mean were from the initial set inserted before the final 3000-h run. Two specimen tubes from the initial set (9B and 2C) exposed 1000 and 1500 h, respectively, have wall thicknesses near the -2σ limit, as does tube 7B from the second tube population, exposed 3000 h. This suggests that these three tubes have indeed experienced slight though measurable metal wastage.

Inspection of all the wall thickness data in Appendix F and that shown in reduced form in Figs. 16 through 20 suggests several conclusions. First, no strong functional relationship appears between wall thickness (or metal wastage) and temperature over the range 800 to 870°C, that is, the range along the in-bed tubes. Though the detailed data are not presented, the eight measurements at points around the circumference of each exposed sample varied smoothly (sinusoidally) as did the pretest ones, indicating that, in general, no localized metal losses had occurred. Furthermore the point of minimum wall thickness of each sample appears to be randomly positioned with respect to the bed.

The data shown in Figs. 16 through 20 indicate that wall thickness did not necessarily decrease in proportion to the total time of exposure, and in almost no case did the minimum wall thickness correspond to the maximum exposure time (4500 h). This suggests that position in the bed was a much more important parameter than the time of exposure. It should be emphasized that this 4500-h test was essentially steady state, with only 5 intentional shutdowns and 14 upsets, most of which were brief and caused by coal feed blockages. We believe that these few thermal cycles would cause only minor scale spallation and, collectively, the results support this view.

A final point may be made regarding various locations within the bed. Only five specimens showed wall thicknesses significantly beyond the 2σ band (see Figs. 17 and 19). Two of these occupied 8C positions and one was at 9B (see Fig. 19). The other two were at 9A and 9D. The fact that these four positions are almost in line vertically suggests that some spatial inhomogeneity existed in that region. However the attack in this region was manifested only in the case of types 304 and 316 stainless steel

and not in the case of type 310 stainless steel [see 3000-9C (4500) in Fig. 18]. Though not totally conclusive, these observations suggest that type 310 is less susceptible to local "inhomogeneities" in the bed than are types 304 and 316 stainless steel.

The ranking of alloy 800H relative to type 310 stainless steel cannot be clearly established from the data discussed in this section. Although the wall thickness measurements of the former alloy were generally within the -2σ scatter band, samples of the alloy were not exposed for a significant period to the more aggressive zone of the bed discussed above.

Severe Corrosion of Type 316 Stainless Steel

Type 316 stainless steel demonstrated a significant variation in corrosive response during the final 3000 h of testing. Of nine tubes of type 316 stainless steel exposed for periods varying from 500 to 4500 h, eight showed relatively light surface and grain boundary attack with a time dependence comparable to that of type 304 stainless steel (Fig. 12). However, a ninth tube, sample 9B, behaved quite differently in terms of the extent and manner of attack. As shown in Fig. 2, this tube was installed at the 1500-h point and was removed at the conclusion of the 4500-h test. Figure 21 illustrates the extensive corrosion and metal wastage that occurred on this sample. The maximum depth of corrosion reached approximately one-third of the 1.6-mm wall thickness. The mode of attack was severe sulfidation-oxidation, with sulfide forming slightly below a general network of grain boundary oxides.

We have no obvious explanation for the heavy attack of tube 9B. The tube was of the same composition and metallurgical condition as the eight companion type 316 stainless steel tubes. An air trimmer tube failure discussed under "Operating Conditions and Run Data" briefly resulted in an air-rich condition in the vicinity of tube 9B and could have been a contributing circumstance in the heavy attack. However, none of the other tubes in the vicinity of the air leak showed similar effects, and we do not know of a credible mechanism whereby extra air admitted by the leak could induce attack of the type observed.

Fig. 21. Cross section of deeply corroded type 316 stainless steel specimen exposed during the final 3000-h portion of the 4500-h AFBC test.

One property of type 316 stainless steel that may have been a factor in the attack relates to the presence of 2.5% Mo in the alloy and the propensity for molybdenum to induce catastrophic oxidation.⁴ During the general oxidation of type 316 stainless steel in high-temperature air, the molybdenum concentration of the alloy increases ahead of the oxidation front. Given sufficient oxidation and attendant molybdenum enrichment, a film of MoO_2 can form below a precursory iron and/or chromium oxide layer. If a defect in the latter oxide layer then occurs, the MoO_2 will convert to molten MoO_3 . Catastrophic oxidation then ensues as iron and chromium react with the molten oxide, transport across it, and deposit as a porous oxide without passivating the underlying metal.

The presence of sulfur atoms, evidenced by the CrS precipitate below the metal surface, could have exacerbated this mode of attack, for example, by increasing the extent to which general oxidation and molybdenum enrichment produced defects in the protective outer oxide. The process of catastrophic oxidation is highly stochastic, since it requires a sequence of events, each with only low probability. Thus its occurrence in only one of nine tubes is not necessarily irrational, although the possibility that the affected tube saw an environment different from the other eight tubes also cannot be dismissed.

SUMMARY AND CONCLUSIONS

Low-Temperature Tubes

Exposure of 2 1/4 Cr-1Mo steel for up to 3000 h at 430 to 540°C in the Fluidyne AFBC bed environment caused a generally uniform oxidation of the fire-side surfaces, with no intergranular attack or sulfide precipitate. The air-side and fire-side oxidation processes accounted for a total reduction of 50 to 75 μm in wall thickness over the 3000-h test period. One potentially important finding concerned the difference in the nature of the oxide scales produced on the two sides of the tubes. On the fire side, the oxide was overlaid by a striated coating of CaSO_4 and was devoid of the Fe_2O_3 outer layer seen on the air side. Furthermore, the depth of the oxide was much more uneven from point to point around the

circumference than that of the air side. These tests give evidence of a solid-state reaction between the oxide and the CaSO_4 coating, although longer term tests will be required to ascertain the rate of this reaction and its effect on the underlying oxide and metal.

Type 304 stainless steel tubes exposed at about 540 to about 610°C, as was expected, showed only superficial oxidation after 3000 h, and the extent of tube wall reduction could not be resolved within the normal manufacturing tolerances. Unlike the chromium-molybdenum steel, which showed heavier oxidation but no sulfide reaction products, the type 304 stainless steel showed some internal sulfide precipitate immediately below the fire-side oxide scale. The extent of sulfide penetration is extremely superficial over the 3000-h time span of these tests, but the effect needs to be addressed in the context of superheater tubes, whose expected service life is 20 to 30 years. This will require tests of considerably longer duration than those reported here.

High-Temperature Tubes

Types 304 and 316 Stainless Steel

Both the 304 and 316 stainless steel types, which nominally contain 18% Cr, have limited scaling resistance in air above 900°C (1650°F). Nevertheless, with one important exception noted below, the rate of metal loss due to scaling of these steels under our test conditions (maximum temperature ~870°C) was less than 75 μm in 4500 h. Furthermore the depth of selective attack (intergranular penetration and internal sulfidation) was generally below 90 μm .

The one notable exception to the pattern described above was a single type 316 stainless steel tube, which sustained unusually heavy oxidation and sulfidation during a 3000-h exposure. The heavy attack occurred over most of the in-bed region of the tube and reduced the sound metal wall thickness by as much as 530 μm . No other instance of this unusual corrosion was detected in eight companion type 316 stainless steel tubes. (The latter were in fact equivalent in behavior to the five type 304 stainless steel tubes tested.) Thus it is not clear whether the heavy attack is

attributable to alloy compositional effects (specifically molybdenum concentration) or to environmental conditions in the bed that were unique to the affected tube. In view of the attractive high-temperature creep properties of type 316 stainless steel, it is premature to rate this steel as unsuitable for AFBC applications on the basis of the isolated instance of poor performance noted here. Obviously more testing should be completed to define the effect of molybdenum content on corrosion under AFBC conditions.

Type 310 Stainless Steel

Type 310 stainless steel, containing 25% Cr, affords excellent resistance to scaling in air up to about 950°C. In the present tests, in which the temperatures were up to 870°C, there was no marked difference in scaling behavior between the type 310 and austenitic steels containing 18% Cr (e.g., type 304). Also the type 310 stainless steel showed essentially the same depth of grain boundary oxidation as the 18% Cr grades. However, the better scaling resistance of the type 310 may have proved it superior had the tests been carried out for a longer duration or at slightly higher temperatures.

Alloy 800H

The high-temperature oxidation properties of alloy 800H make it uniquely suited to operation in oxygen-containing environments at temperatures as high as 980°C. The chromium in the alloy, at a level of 21%, promotes the formation of a highly protective surface oxide, and the nickel, at a level of 32%, enhances the retention of the oxide during cyclic exposure to high temperatures. Furthermore, the performance of alloy 800H in environments simulating conventional utility coal-fired steam boilers has indicated good resistance to wastage up to 900°C (1650°F).⁵ Nevertheless, the generally poor performance of nickel-based alloys under AFBC conditions^{2,3} has created some skepticism that alloy 800H, containing 32% Ni, will prove susceptible to sulfidation and/or hot corrosion in the AFBC coal-ash environment.

In our test program, the alloy 800H tubes showed no evidence of deposit-induced wastage (hot corrosion), and the degree of scaling

resistance was of the same high order as the 300-series stainless steels. However, the alloy 800H showed more attack in the form of grain boundary oxidation and subsurface sulfide than did the other test alloys. The depth of this localized attack appeared to be controlled by a solid state diffusion process, since it subscribed generally to a $1/2$ -power time dependence. Extrapolating the present test results using the latter time dependence predicts that the depth of localized penetration would reach about 140 μm in one year and about 310 μm in five years. Although these rates are obviously based on an extremely limited set of data and an imprecise understanding of the rate-controlling mechanisms, they do not appear to be an overriding concern in the use of alloy 800H for AFBC applications. Additional longer term testing of the alloy will be required to assess the role of nickel in the resistance of alloy 800H to attack by SO_2 and CaSO_4 over the relatively long exposure periods (one to two decades) germane to AFBC power plants.

ACKNOWLEDGMENTS

The authors gratefully acknowledge the efforts of the staff of the Fluidyne Engineering Corporation of Minneapolis in the operation of the AFBC for these tests. Most especially, we wish to thank R. H. Cooper, Jr. of the Metals and Ceramics Division for his significant contributions to the first phase of this program (1500-h Tests) and helpful discussions throughout the balance of the investigation. During the course of this investigation, extensive thermodynamic calculations were performed by T. B. Lindemer and R. L. Pearson of the Chemical Technology Division in an attempt to establish phase assemblages that might exist on alloy steels under fluidized bed conditions. Their results were especially useful in the interpretation of Inconel 600 behavior discussed in Part 1 of this report. We also wish to thank R. S. Crouse, M. D. Allen, and J. R. Mayotte for their excellent metallographic and microanalytical work; J. I. Federer and P. J. Ficalora for reviewing the manuscript; Sig Peterson for technical editing; and P. T. Thornton for preparation of the final report.

REFERENCES

1. A. P. Fraas and R. S. Holcomb, "Atmospheric Fluidized Bed Combustion Technology Test Unit for Industrial Cogeneration Plants," pp. 55-69 in *The Proceedings of the Fifth International Conference on Fluidized Bed Combustion*, Vol. III, The MITRE Corporation, McLean, Va., 1978.
2. M. R. Carroll, T. G. Godfrey, K. R. Drake, and R. H. Cooper, "Fireside Corrosion of Austenitic Alloys at High Temperatures in a Fluidized-Bed Combustor," Paper 79-GT-121 presented at the ASME Gas Turbine Conference and Exhibit and Solar Energy Conference, San Diego, California, March 12-15, 1979.
3. R. H. Cooper, T. G. Godfrey, and J. H. DeVan, *Corrosion of High-Temperature Materials in AFBC Environments - Part 1 (1500 h tests)*, ORNL/TM-7734 P1 (June 1981).
4. J. H. DeVan, *Catastrophic Oxidation of High-Temperature Alloys*, ORNL-TM-51 (Nov. 10, 1961).
5. A. L. Plumley, J. I. Accortt, and W. R. Roczniak, "Evaluation of Boiler Tube Materials for Advanced Power cycles," pp. 623-79 in *Proc. Corrosion Erosion of Coal Conversion Systems Materials Conference* (Berkeley, Calif. Jan. 24-26, 1979), National Association of Corrosion Engineers, Houston, Texas, 1979.

46

THIS PAGE
WAS INTENTIONALLY
LEFT BLANK

APPENDIX A

Analyses of Bed Feed

Table A1. Typical analyses of Illinois No. 6 coal
used for the 4500-h test at FluidDyne^a

Analysis	Value for each sample ^a				
	1	2	3	4	5
Heating value					
GJ/kg	25.529	25.385	25.099	24.653	24.951
Btu/lb	10,983	10,921	10,798	10,606	10,519
Proximate, %					
Moisture	8.04	10.17	11.01	10.41	13.17
Ash	14.57	12.31	13.15	13.55	12.68
Volatile	34.89	34.70	34.52	32.94	33.71
Fixed C	42.50	42.82	41.32	43.10	40.44
Ultimate, %					
Moisture	8.04	10.17	11.01	10.41	13.17
C	61.25	60.84	60.33	60.20	58.19
H	4.33	4.41	4.32	4.10	4.12
N	1.09	1.04	1.04	0.61	1.06
Cl	0.03	0.07	0.04	0.01	0.09
S	3.64	3.41	3.34	3.42	3.26
Ash	14.57	12.31	13.51	13.55	12.68
O (difference)	7.05	7.75	6.77	7.70	7.43
Ash, ^b %					
SiO ₂	45.57				
Fe ₂ O ₃	15.80				
Al ₂ O ₃	17.99				
TiO ₂	0.74				
CaO	6.88				
MgO	1.09				
SO ₃	4.22				
K ₂ O	2.20				
Na ₂ O	4.84				
P ₂ O ₅	0.15				

^aSamples selected at random.^bAs equivalent oxides.

Table A2. Typical analysis of Q20-60 granular Quincy limestone
in the 4500-h test at FluidDyne

CaCO ₃	97.73 wt %
MgCO ₃	1.47
SiO ₂	0.60
Al ₂ O ₃	0.80
Fe ₂ O ₃	0.05
MnO	0.023
CuO	0.0005
Total S	0.033
Total Alkali	0.1

APPENDIX B

Daily Test Conditions

This appendix is reproduced from Appendix C of the private report from the FluidDyne Engineering Corporation detailing conditions of the exposure.

The following unit conversions apply to this appendix:

$$^{\circ}\text{C} = (5/9)(^{\circ}\text{F} - 32)$$

$$1 \text{ lb/hr} = 0.4536 \text{ kg/h}$$

$$= 0.1260 \text{ g/s}$$

FLUIDYNE ENGINEERING CORPORATION

Date	Time Period	Coal Feed lb/hr	Sorbent Feed lb/hr	Ca/S Ratio	O ₂ %	SO ₂ ppm	Bed Temp °F
5-1	19:30-22:00	52.0	22.4	3.8	3.0	360	—
5-2	04:30-07:30	54.7	22.3	3.6	3.8	250	—
5-2	10:15-16:30	55.5	21.0	3.3	3.4	358	—
5-2	21:45-02:00	53.7	21.2	3.5	3.1	378	1637
5-3	03:30-07:30	53.8	21.0	3.4	3.9	313	1635
5-3	17:00-06:00	51.3	15.1	3.6	3.3	346	1636
5-4	09:00-15:00	53.5	16.4	3.7	3.3	338	1635
5-4	17:00-24:00	53.3	20.6	3.4	3.4	342	1637
5-5	00:00-04:30	51.4	18.0	3.1	3.3	314	1635
5-5	13:00-21:20	52.0	19.1	3.2	3.3	344	1637
5-6	00:00-12:56	54.4	21.5	3.5	3.3	350	1636
5-6	16:15-22:40	55.6	19.8	3.1	3.4	373	1636
5-7	02:45-24:00	56.3	21.4	3.4	3.3	335	1636
5-8	00:00-24:00	54.5	21.2	3.4	3.3	325	1636
5-9	09:20-24:00	53.6	18.7	3.1	3.3	338	1636
5-10	00:00-07:00	56.3	20.0	3.1	3.4	306	1636
5-10	17:00-24:00	53.3	19.0	3.1	3.1	333	1636
5-11	00:00-05:00	56.1	21.2	3.3	2.8	407	1637
5-11	12:07-24:00	54.0	16.8	3.7	3.0	328	1636
5-12	08:40-24:00	52.8	17.8	3.0	3.2	350	1636
5-13	00:00-24:00	54.5	18.4	3.0	3.3	338	1636
5-14	12:00-24:00	55.4	19.2	3.1	3.3	344	1637
5-15	00:00-08:20	55.1	29.1	3.2	3.0	317	1636
5-15	08:20-16:40	54.2	15.3	2.5	3.1	375	1637
5-16	13:50-24:00	54.1	17.1	2.8	3.2	305	1637
5-17	13:00-23:00	54.2	19.1	3.1	3.3	346	1637
5-18	13:30-22:00	55.3	21.8	3.5	3.2	353	1636
5-19	03:15-11:00	54.1	18.9	3.0	3.1	423	1634
5-19	14:00-22:00	53.6	14.4	2.4	3.4	280	1634
5-20	04:00-16:50	55.6	18.8	3.0	3.4	329	1633
5-20	16:50-23:45	52.4	14.7	2.5	3.4	364	1636
5-21	12:12-19:00	59.0	22.2	3.3	3.4	406	1637

FLUIDYNE ENGINEERING CORPORATION

Date	Time Period	Coal Feed lb/hr	Sorbent Feed lb/hr	Ca/S Ratio	O ₂ %	SO ₂ ppm	Bed Temp °F
5-22	24:00-05:30	53.1	19.7	3.3	3.3	309	1636
5-22	10:15-15:00	55.1	21.2	3.4	3.3	410	1635
5-22	20:14-24:00	54.4	19.2	3.2	3.35	363	1636
5-23	24:00-10:00	56.0	23.0	3.6	3.3	342	1635
6-7	20:00-24:00	49.8	25.8	4.6	3.3	365	1633
6-8	01:00-09:00	49.1	21.0	3.8	3.3	374	1634
6-10	01:00-05:30	49.3	22.4	4.0	3.4	379	1635
6-11	08:00-24:00	59.6	27.5	4.8	3.0	336	1635
6-12	24:00-07:00	51.0	28.1	4.9	3.0	314	1633
6-13	10:00-03:00	59.5	21.1	3.7	3.2	329	1635
6-13	06:30-24:00	59.3	21.8	3.8	3.0	345	1634
6-14	13:30-24:00	51.1	23.3	4.0	3.2	366	1634
6-15	07:00-16:30	59.3	29.4	3.4	3.1	334	1634
6-16	Lost power; clean water tubes, clean flue and atomizer						
6-17	15:00-19:30	53.1	20.7	3.4	3.3	703	1636
6-18	16:00-24:00	54.4	24.6	4.0	3.3	524	1635
6-19	17:00-06:00	50.4	20.7	3.6	3.4	368	1613
6-20	14:30-24:00	52.7	29.1	4.9	3.2	362	1624
6-21	13:30-18:30	54.0	23.9	3.9	3.2	317	1628
6-22	17:30-08:15	49.6	23.8	4.2	3.5	343	1635
6-23	11:22-24:00	50.0	24.2	4.3	3.4	345	1635
6-24	19:30-03:00	49.5	23.6	4.2	3.5	262	1637
6-25	10:45-16:10	50.5	21.8	3.8	3.3	362	1636
6-26	13:30-02:00	50.7	23.7	4.1	3.3	355	1635
6-27	10:00-18:00	50.6	23.3	4.1	3.4	333	1636
6-28	10:30-24:00	51.3	22.7	3.9	3.5	346	1636
6-29	16:30-01:15	50.9	23.2	4.0	3.3	379	1637
6-30	08:00-16:00	50.6	21.7	3.8	3.7	315	1636
6-30	16:00-24:00	51.2	25.4	4.4	3.5	360	1635
7-1	11:00-24:00	51.4	23.7	4.1	3.4	390	1636
7-2	01:15-24:00	52.8	24.6	4.1	3.6	348	1636
7-3	24:00-09:00	54.1	27.1	4.4	3.6	399	1636

FLUIDYNE ENGINEERING CORPORATION

Date	Time Period	Coal Feed lb/hr	Sorbent Feed lb/hr	Ca/S Ratio	O ₂ %	SO ₂ ppm	Bed Temp °F
7-4	24:00-14:15	55.1	26.5	4.2	3.6	339	1636
7-5	15:00-23:00	53.0	24.9	4.2	3.6	350	1637
7-6	11:00-19:00	55.1	22.5	3.6	3.4	349	1637
7-7	07:30-15:00	54.6	26.3	4.2	3.6	382	1636
7-8	06:15-18:00	53.1	27.0	4.5	3.4	375	1636
7-9	18:40-24:00	51.1	18.1	3.1	4.1	553	1631
7-10	11:00-18:00	53.0	29.5	4.9	3.7	354	1635
7-11	14:30-24:00	51.7	23.3	4.0	3.7	370	1637
7-12	09:00-19:00	53.1	29.5	4.9	3.8	360	1636
7-13	18:00-24:00	53.1	26.8	4.4	3.7	363	1634
7-14	19:30-24:00	49.7	20.9	3.7	3.7	343	1637
7-15	15:00-23:30	51.2	25.7	4.4	3.3	330	1636
7-16	05:00-12:30	49.5	21.5	3.8	3.4	330	1634
7-17	18:00-24:00	52.0	21.2	3.6	3.5	356	1636
7-18	18:20-01:00	56.3	22.6	3.5	3.6	364	1636
7-19	12:00-15:30	56.3	20.6	3.2	3.3	387	1636
7-20	05:30-14:00	56.3	26.7	4.2	3.8	345	1635
7-21	02:15-09:00	55.2	19.2	3.1	4.1	300	1626
7-22	11:00-21:15	53.9	25.7	4.2	3.4	337	1637
7-23	24:00-07:00	53.9	24.5	4.0	3.5	293	1636
8-1	08:00-12:15	43.8	20.1	4.0	3.0	357	1634
8-1	19:30-24:00	43.4	17.9	3.6	3.1	366	1636
8-2	06:30-12:00	43.5	18.3	3.7	3.2	340	1637
8-3	13:30-23:30	44.4	18.7	3.7	3.2	322	1637
8-4	08:30-16:30	44.1	17.7	3.5	3.1	368	1636
8-5	18:45-24:30	44.0	17.9	3.6	3.3	330	1637
8-6	05:30-20:15	43.6	18.1	3.7	3.3	346	1637
8-7	15:15-22:20	44.7	18.0	3.6	3.4	338	1638
8-8	10:15-20:15	43.3	20.2	4.1	3.4	355	1637
8-9	13:30-01:00	43.4	16.8	3.4	3.4	346	1637
8-10	14:00-20:00	41.6	18.7	4.0	3.5	290	1636
8-11	17:30-03:00	42.8	19.7	4.1	3.6	332	1637

FLUIDYNE ENGINEERING CORPORATION

Date	Time Period	Coal Feed lb/hr	Sorbent Feed lb/hr	Ca/S Ratio	O ₂ %	SO ₂ ppm	Bed Temp °F
8-12	16:45-02:30	43.0	19.0	3.9	3.3	352	1638
8-13	07:30-20:30	43.1	15.9	3.2	3.4	282	1637
8-14	24:00-14:00	43.3	18.3	3.7	3.5	343	1636
8-15	05:30-16:30	42.7	17.8	3.7	3.4	335	1636
8-16	09:00-17:00	43.0	16.8	3.4	3.3	340	1636
8-17	20:30-02:30	43.7	19.2	3.9	3.3	345	1636
8-18	16:30-00:30	44.2	23.6	4.7	3.5	356	1637
8-19	06:00-14:00	44.2	19.2	3.8	3.3	325	1636
8-20	09:00-23:00	44.3	19.0	3.8	3.4	340	1636
8-21	24:00-14:45	43.7	18.4	3.7	3.5	312	1637
8-22	14:30-24:00	43.6	19.8	4.0	3.4	339	1637
8-23	12:00-20:30	43.7	22.0	4.4	3.3	346	1637
8-24	05:15-09:30	45.2	19.2	3.8	3.4	307	1636
8-25	10:30-24:00	44.2	21.3	4.2	3.4	339	1637
8-26	10:30-19:30	44.2	20.9	4.2	3.3	357	1637
8-27	09:00-18:00	42.7	20.7	4.3	3.6	364	1637
8-28	13:30-21:30	44.9	20.4	4.0	3.4	374	1636
8-29	14:15-23:15	43.0	19.9	4.1	3.5	316	1637
8-30	24:00-07:00	44.2	22.2	4.4	3.5	305	1636
8-31	05:30-16:00	46.5	19.4	3.7	3.4	346	1635
9-1	13:00-23:00	45.2	20.2	3.9	3.5	338	1636
9-2	13:30-22:00	43.7	19.6	4.0	3.4	348	1637
9-3	04:30-14:30	44.9	19.0	3.7	3.5	329	1637
9-4	08:30-17:00	47.4	22.0	4.1	3.5	324	1636
9-5	14:35-02:30	43.7	17.7	3.6	3.4	357	1637
9-6	10:30-16:15	43.0	18.4	3.8	3.6	330	1638
9-7	06:30-18:00	43.8	17.3	3.5	3.4	358	1636
9-8	05:30-09:15	43.6	20.3	4.1	3.7	365	1637
9-9	07:00-14:45	43.2	21.4	4.4	3.4	391	1635
9-10	01:00-11:45	43.6	22.5	4.6	3.5	320	1636
9-11	08:00-21:45	42.5	19.4	4.0	3.5	300	1637
9-12	15:45-24:00	44.4	17.2	3.4	3.3	375	1636
9-13	11:45-23:15	44.1	19.6	3.9	3.8	350	1636

FLUIDDYNE ENGINEERING CORPORATION

Date	Time Period	Coal Feed lb/hr	Sorbent Feed lb/hr	Ca/S Ratio	O ₂ %	SO ₂ ppm	Bed Temp °F
9-14	10:30-21:00	44.6	20.6	4.1	3.3	325	1636
9-15	03:50-12:30	43.4	20.4	4.2	3.5	355	1635
9-16	15:00-21:00	46.3	20.9	4.0	3.3	366	1636
9-17	12:30-24:00	48.0	21.7	4.0	3.6	384	1638
9-18	08:10-21:00	47.3	20.3	3.8	3.5	353	1637
9-19	16:30-24:30	47.2	21.3	4.0	3.7	301	1636
9-20	15:00-24:00	46.3	18.9	3.6	3.7	385	1635
9-21	16:30-24:00	46.6	21.7	4.1	3.6	406	1636
9-22	11:00-21:30	46.7	23.6	4.5	3.5	308	1636
9-23	07:00-16:00	47.1	21.6	4.1	3.5	355	1636
9-24	07:00-23:30	47.2	20.4	3.8	3.4	355	1636
9-25	07:30-11:45	47.9	18.0	3.3	3.3	328	1635
9-26	07:30-16:00	45.5	19.6	3.8	3.3	330	1637
9-27	18:15-01:00	46.9	20.8	3.9	3.5	326	1637
9-28	16:15-02:00	45.2	14.2	2.8	3.3	290	1637
9-29	14:00-21:30	44.7	12.6	2.5	3.3	331	1637
9-30	24:00-08:30	44.9	17.2	3.4	3.4	334	1637
10-1	14:30-23:30	45.4	17.1	3.3	3.6	320	1637
10-2	14:30-23:30	45.0	20.7	4.1	3.7	340	1637
10-3	01:30-10:45	45.3	15.3	3.0	3.6	289	1637

APPENDIX C

Adjustment of Feed Rate Data

This appendix is reproduced from Appendix A of the private report from the FluidDyne Engineering Corporation detailing conditions of the exposure.

The following unit conversion applies to this appendix:

$$\begin{aligned} 1 \text{ pph} &= 1 \text{ lb/hr} = 0.4536 \text{ kg/h} \\ &= 0.1260 \text{ g/s} \end{aligned}$$

FLUIDDYNE ENGINEERING CORPORATION

Coal and sorbent feed rates measured during the 500 and 1000-hour tests were corrected for the amount of "fines" entrained by the air flow which leaks across the rotary valve. During this test program two rotary valves were used; air leakage rates were measured for both valves prior to test and periodically while in use. Using these measured leakage rates, calculated terminal velocities as a function of particle size, and size distributions for both the coal and limestone, the amount of coal and sorbent entrained was estimated. Results of this analysis appear in Table A-1 and show that during the 500-hour test the amount of coal and sorbent entrained increased from 7.1 to 10.2 lb/hr and 1.7 to 3.6 lb/hr, respectively. Prior to the start of the 1000-hour test, the rotary valve was replaced with one having a tighter clearance. Estimated entrainment rates for coal and sorbent during this test were lower as expected and increased from 3.1 to 6.9 lb/hr and .4 to 2.1 lb/hr, respectively.

Supporting data shown in Table A-2 represent measured entrainment rates of coal and sorbent for a 192-hour period during the 1000-hour test. This material was collected in a separate baghouse and was weighed before disposal. The collection rate for this period averaged 8.4 ± 2.9 lb/hr. Further, a size analysis of the entrained material collected during this period showed that 99.3% of the material collected was smaller than 200 μm (approximately 70 mesh). If particles smaller than 200 μm for the coal and sorbent feed were completely elutriated, then 16% of the coal fed and 13% of the sorbent fed would have been lost. Coal and sorbent feed rates measured during this period averaged $50.5 \pm .6$ lb/hr and 23.3 ± 1.0 lb/hr, respectively. Using these feed rates and assuming particles smaller than 200 μm are completely elutriated, the average collection rate of fines would have been 11.1 lb/hr.

FLUIDDYNE ENGINEERING CORPORATION

In summary, a comparison of the three methods used to evaluate the loss of coal and sorbent elutriated by air leaking across the rotary valve shows general agreement. On the basis of this comparison, the coal and sorbent feed rates were corrected using the calculated entrainment rates reported in Table A-1. Average values were used in each case.

Typical particle size distributions for coal, sorbent and the elutriated fines are shown in Figure A-1. Curves shown in Figure A-1 are derived from the measured particle size distribution curve fit to a Rosin Rammler distribution.

	Air Leak Flow pph	(1) Coal Feed Entrained %	(1) Sorbent Feed Entrained %	(2) Coal Feed Entrained pph	(2) Sorbent Feed Entrained pph
500 Hour Test	120	13.2	8.7	7.1	1.7
	180	18.9	18.4	10.2	3.6

1000 Hour Test	50	5.9	1.6	3.1	.4
	120	13.2	8.7	6.9	2.1

(1) Based on particle size distributions shown in Figure A-1.

(2) Based on averaged coal and sorbent feed rates measured during test.

FLUIDDYNE ENGINEERING CORPORATION**ENTRAINMENT RATE, ELUTRIATED FINES
FROM COAL/SORBENT FEEDER**

Date	Time Hours	Collection Rate lb/hr
6-23	0652	7.1
6-23	1810	10.7
6-25	2100	-
6-26	1730	6.4
6-27	1700	5.3
6-28	1730	8.2
6-29	1313	9.6
6-30	0700	5.9
6-30	1440	14.0
Average	$\bar{X}$	8.4
Standard Deviation	S	2.9

FIGURE 1 TYPICAL PARTICLE SIZE DISTRIBUTIONS FOR COAL AND SORBENT USED DURING 3000-HOUR CORROSION TEST AND ELUTRIATED FINES FROM COAL FEED SYSTEM

**THIS PAGE
WAS INTENTIONALLY
LEFT BLANK**

APPENDIX D

Summary of Upset Conditions

This appendix is reproduced from Appendix E of the private report from the Fluidyne Engineering Corporation detailing conditions of the exposure.

FLUIDYNE ENGINEERING CORPORATION

DATE	TIME	HOURS LOST	DESCRIPTION
May 1	1900		Test conditions established, start of 500-hour test
May 5		4	Coal feed interruption caused by loss of draft control.
May 18		4	Coal feed interruption caused by plugged feed tube.
May 21		10.5	Coal feed interruption caused by plugged feed tube
May 23	1000		Test completed.
June 7	2100		Test conditions established, start of 1000 hour test.
June 9		24	Test interrupted due to restriction in flue duct.
June 16		20	Power outage. Maintenance performed on cooling water system.
June 22		6	Problems with combustor heat balance, test interrupted to remove one of the two water tubes and replace with air-cooled trimmer tube.
July 8		23	Failure of air-cooled trimmer tube. Test interrupted to remove and replace tube.
July 12		3.5	Coal feed interruption caused by plugged feed tube.
July 13		6.0	Coal feed interruption caused by plugged feed tube.
July 18		5.5	Power panel failure.
July 23	0700		Test completed.

FLUIDYNE ENGINEERING CORPORATION

DATE	TIME	HOURS LOST	DESCRIPTION
August 1	1500		Test conditions established, start of 1500 hour test.
August 10		5	Coal feed interruption caused by plugged feed tube.
August 11		1.5	Coal feed interruption caused by plugged feed tube.
August 29		2	Coal feed interruption caused by plugged feed tube.
September 8		20.5	Power outage.
October 3	2100		Test completed.

**THIS PAGE
WAS INTENTIONALLY
LEFT BLANK**

APPENDIX E

Analysis of Trimmer Tube Failure

This appendix is reproduced from Appendix J of the private report from the FluidDyne Engineering Corporation detailing conditions of the exposure.

The following unit conversions apply to this appendix:

$$^{\circ}\text{C} = (5/9) (^{\circ}\text{F} - 32); \text{ for } \Delta T, ^{\circ}\text{C} = (5/9)^{\circ}\text{F}$$

$$1 \text{ psi} = 6.895 \text{ kPa}$$

$$1 \text{ in.} = 25.4 \text{ mm}$$

$$1 \text{ lb} = 4.448 \text{ N}$$

INTERNAL CORRESPONDENCE

NUCLEAR DIVISION

POST OFFICE BOX X, OAK RIDGE, TENNESSEE 37830

To (Name) T. G. Godfrey ✓
 Division
 Location

Date October 24, 1978

Originating Dept.

Answering letter date

Copy to H. A. Nelms
 T. W. Pickel
 W. C. T. Stoddart
 WO-G-2207E-AA
 File - NoRC

Subject Investigation of Trimmer
 Tube Failure

The attachment contains analyses of a tube in an intermediate level heat exchanger or "trimmer tube" that failed in a fluidized bed coal combustor. Stresses in excess of yield were produced in the tube by thermal expansion which was caused by inoperative sliding end joints as evidenced by the deformed shape of the tube after removal from the combustor. A mode of failure progression is postulated and the magnitude of stresses in the tube are estimated by analysis. Since an effective sliding joint between the tube and the combustion bed would eliminate the structural problems found, this analysis was purposefully limited in scope to several hand calculations and a small program for a calculator.

The history of loading on the tube in the fluidized bed consists of a steady increase in temperature from normal room temperature to 1640°F at most and then a repeated oscillation of temperature between 1440°F and 1640°F until the failure after about 400 hours of continuous operation. Combustion products flow past the tube during operation and cooling air passes through the tube but loads produced by these flow conditions are inconsequential relative to thermal loads and are ignored. The fact that the tube served previous duty as a water carrying heat exchanger tube was also considered negligible except for the existence of inclusions distributed over the external surface of the tube.

As the tube was heated, compressive stress increased until the tube buckled as a fixed-fixed column. The ends of the broken tube, when assembled are co-linear and straight for several inches verifying the fixity of the end constraints. Inelastic buckling could have occurred at an average temperature over the tube as low as 280°F at which point the entire cross-section has yielded. Just before or after the column type buckling, a local diamond shaped buckle occurred at midspan. The reduction in stiffness at the local deformation caused the thermal strain to be dissipated primarily in bending at the midplane. After overall buckling commences, axial force does not increase significantly and may decrease after local buckling or with increase in temperature.

The nominal stress at the extreme fiber on the tension side approaches 20,000 psi or nearly twice the yield strength at 1600°F. However, stress at an inclusion at that point is at least twice as great and a crack will start there immediately. Note that although nominal stress values at the extreme compressive fiber and in the region of the local buckle exceed 20,000 psi cracks do not start in compressive stress fields.

The crack tip maintains a high tensile stress and at 1500°F even 6000 psi tension can cause 304 stainless steel to fail in 100 hours. The crack penetrates the tension side of the tube, leaving the "shear lips" visible in photographs of the fracture surface, and travels on two fronts on each side of the remaining circumference. Crack progression here is not dependent on fatigue or repeated loads. The crack, however, is driven by an overall compressive force and before it can sever the tube, it must leave the tension side of the neutral axis after the center of the local buckle goes into tension. It cannot and stops, leaving a "W" shaped cross-section intact with the buckled region now partially in tension and with the axial load much reduced.

At this point, the tube is still functional with some remaining axial force keeping the crack faces close enough to not disturb air flow inside. Extreme fiber stresses can still exceed yield in which case the tension at the center of the local buckle would cause the ductile material to be drawn out. Creep has not been mentioned before, but is very significant in explaining the final severing of the pipe. These effects must be ignored, however, in this limited study.

A program to find the neutral axis of the tube cross-section at the local buckle was developed for use on the Hewlett Packard HP-97 calculator. This program, which uses the Ramberg-Osgood plastic stress-strain relationship for 304 stainless steel, was instrumental in determining stress across the cross-section. It would be useful in any continuation of analysis and consequently the program will be retained and available for use.

CR Hammond
C. R. Hammond

CRH/phs

Attachment

TRIMMER TUBE ANALYSIS

Photographs of the broken, deformed tube showed very little rotations of the ends - indicating a fixed-fixed end condition. The free span of the tube is about 18 in. with 5 in. on each end elastically supported. The effective length of the tube for purposes of a buckling analysis is between 18 in. and $\frac{28}{2}$ in. or 14 in.

The radius of gyration of the tube is

$$\rho = \sqrt{\frac{\pi[(0.25)^4 - (0.25 - 0.049)^4]}{\pi[(0.25)^2 - (0.25 - 0.049)^2]}} = 0.321$$

The slenderness ratio then is

$$L/\rho = \frac{18}{0.321} \text{ to } \frac{14}{0.321} = 56 \text{ to } 44$$

The Euler buckling formula does not apply to beams with slenderness ratios below 100. Plastic buckling is assumed.

The increase in temperature necessary to cause a strain of 0.002 in./in. which is near the yield strain of this material is

$$\epsilon_y = 0.002 = \alpha \Delta T = 9.6 \times 10^{-6} \Delta T \quad 1*$$

$$\Delta T = 208^\circ\text{F}$$

If the initial temperature is 72°F, the critical temperature is

$$T_{cr} = 280^\circ\text{F}$$

Yield stress at this temperature is 31 ksi/in.² and the axial force is

$$P = 31 \text{ ksi/in.}^2 (0.06942 \text{ in.}^2) = 2.2 \text{ ksi}$$

Note that the critical temperature must be average temperature over the 28 inch span.

After the initiation of buckling, the tube displaces laterally. Bending is then produced in the tube as shown below.

*Superscripts indicate references listed below.

$$M_0 = P\delta - M + \int_a^{L/2} K y(x) x dx - V \frac{L}{2}$$

$$V = \int_a^{L/2} K y(x) dx$$

The relative magnitude of M_0 and M is a function of lateral displacement but is unknown since the modulus of the elastic support is unknown.

Inspection of the broken tube reveals a diamond shaped deformation at midspan that resembles a local buckle in a cylindrical shell. The fact that only one buckle is found indicates an a-symmetric stress distribution over the cross-section such as produced in bending. The midspan location of the deformation indicates either that M_0 exceeded M when the buckle formed or that the elastic support helped retain the cross-section.

The literature² indicates clearly that such a local instability would not occur for the size of tube considered at stresses below yield. Whatever the origin of the buckle, its presence significantly weakened the midspan section of tube containing it. To provide a closer look at the plane of failure, that cross-section was represented as shown:

The geometry of the cross-section was simplified somewhat to achieve mathematical tractability. The half-width of the buckle, r , was measured from a photograph of the tube. Forces in the tube after buckling were not known, but the plastic deformation of the tube was measured after the tube was removed after failure. The radius of curvature of the tube at midspan was found to be about 6 inches. It was assumed that this deformation occurred before a crack could progress far.

Even during plastic deformation, plane cross-sections of beams remain plane. Thus, the longitudinal strain across a cross-section is

$$\epsilon = \frac{y}{\rho},$$

where y is distance from the neutral axis and ρ is the local radius of curvature for plastic as well as elastic deformation. The stress strain curve for 304 stainless steel at a given temperature can be represented simply as³

$$\epsilon = \frac{\sigma}{E} + \left[\frac{\sigma - \sigma_p}{K} \right]^{1/m}$$

where temperature, T , is in degrees Kelvin and

$$E = 3.3675 \times 10^7 - 13.823 T$$

$$\sigma_p = \sigma_y - 12\,198 + 6.7093 T$$

$$\sigma_y = 1.25 (25\,850.5 - 12.8179 T)$$

$$K = 69\,964 - 25.4491 T$$

$$m = 0.25824 + 7.749 \times 10^{-5} T$$

If the elastic strain (the first term in the expression above) is neglected, then the equation can be inverted to give the total stress as

$$\sigma = \sigma_p + K \left[\frac{y}{\rho} \right]^m$$

It is assumed that if absolute values of y and σ are taken, this expression holds for tension and compression.

The neutral axis is found by equating the integral of the stress over the cross-section to the axial force on the tube. The axial force at a particular temperature is taken to be the yield stress at that temperature times the area of the tube. At 1640°F, the axial force, then, was assumed to be 948 lb. An iterative scheme was followed to find the neutral axis with all integrations approximated by summation. The entire procedure was programmed for the HP-97 portable calculator. Results are as follows:

$$T = 1640^\circ\text{F}$$

$$P = 948 \text{ lb.}$$

$$y_o = 0.172 \text{ inches (above horizontal diameter)}$$

$$\sigma_{\max} \text{ (tension)} = 18,000 \text{ psi}$$

$$\sigma_{\min} \text{ (compression)} = -25,000 \text{ psi}$$

The compressive axial force was increased and as a result the maximum stress decreased while the compressive stress also decreased demonstrating the importance of the assumed force.

Stresses were also calculated in the same way at the temperature 1440°F. For this case and with the axial force again equal to the yield stress times the area, the results were:

$$T = 1440^\circ\text{F}$$

$$P = 1070 \text{ lb.}$$

$$y_o = 0.1835 \text{ in.}$$

$$\sigma_{\max} \text{ (tension)} = 19,000 \text{ psi}$$

$$\sigma_{\min} \text{ (compression)} = -27,000 \text{ psi}$$

The radius of curvature used in this case was 6.33 inches. This value was determined from a linear elastic, finite element model⁴ of the tube generated at the FluidDyne Engineering Corporation. This Model imposed a thermal load on an original configuration in the shape of the deformed tube as it was recovered from the combustor. Changes in temperature of -100°F and -300°F were applied to represent the tube at the low end of its temperature oscillations. Stresses calculated from this model following linear elasticity are not realistic but the distribution of strains along the deformed tube is useful. The change in radius of curvature at the midspan was found for a -200°F temperature by interpolating between the two computed results. The same change was applied to the 6 inch radius and the neutral axis position was determined.

The maximum stress decreases as the temperature increases although the distance of the extreme fiber in tension from the neutral axis increases. Stress is more strongly influenced by the temperature than the strain.

These stresses calculated exceed average yield strengths at both temperatures.⁵ The maximum stress at 1640°F is approximately equal to the tensile strength of the material at that temperature. Stress is high enough that consideration of fatigue is not necessary, the material will fail in a matter of time. Reference 5 indicates that at 1500°F, a specimen of 304 stainless steel will fail on average in 100 hours under a constant stress of only 6000 psi.

Although it seems that the local buckle alone would cause eventual failure of the pipe by localizing high strains, the presence of surface inclusions near the same cross-section is also detrimental to the tube. The intensification in the plastic stress produced by the inclusion is unknown, but any increase in stress would obviously shorten the time to failure.

REFERENCES

1. "Properties of Some Metals and Alloys," The International Nickel Company, Inc., 1968, p. 44.
2. Timoshenko and Gere, Theory of Elastic Stability, Second Edition, McGraw Hill Book Company, 1961, p. 470.
3. Nuclear Systems Design Handbook, Volume 1, Hanford Engineering Development Laboratory, Revised 1978, Section 2, p. 1.0-1.1.
4. "Tube Thermal Analysis," SAGS input and output sheets, Fluidyne Engineering Corporation, 1978.
5. "Mechanical and Physical Properties of the Austenitic Chromium-Nickel Stainless Steels at Elevated Temperatures," The International Nickel Company, Inc., 1963.

APPENDIX F

Detailed Fire-Side Corrosion Measurements

Table F. Fireside corrosion measurements on high temperature tubes exposed in the Fluidyne AFBC over the range 810–870°C^a

Tube Sample ^b	Exposure (h)	Removal Time ^c (h)	Oxide Scale Thickness, μm			Intergranular Corrosion Depth, μm			Depth, μm , of Sulfide Inclusions			Tube Wall Thickness	
			av.	s.d.	max	av	s.d.	max	av	s.d.	max	av	s.d.
Type 304 Stainless Steel													
9A-1	500	500	5.2	2.2	7.0	10.8	1.2	12.9	19.5	3.1	24.0	1712	30
-2	500	500	6.1	1.9	10.6	25.7	7.8	34.5	36.7	4.9	41.5	1716	29
2C-1	1000	3000	9.0	3.8	16.9	25.4	22.5	66.6	39.0	23.8	73.5	1687	46
-2	1000	3000	9.6	3.4	14.7	16.6	5.9	25.9	25.3	9.5	36.3	1649	19
-3	1000	3000	12.2	5.4	23.7	46.6	35.6	104.6	63.7	36.9	126.4	1656	28
-4	1000	3000	12.8	4.7	20.3	36.8	13.9	57.7	62.3	13.1	76.3	1654	22
-5	1000	3000	8.9	3.2	15.9	25.2	13.9	54.2	48.1	15.6	81.7	1646	11
-6	1000	3000	9.5	2.9	13.3	31.0	13.0	43.8	50.3	14.3	73.6	1649	19
2C-1	1500	4500	12.1	7.3	30.9	69.0	19.1	93.5	77.0	31.0	107.9	1634	21
-2	1500	4500	9.2	5.2	16.6	55.2	46.3	105.3	91.7	53.1	145.3	1666	30
-3	1500	4500	6.7	1.5	8.4	17.5	4.1	25.9	48.9	7.0	53.2	1658	23
9A-1	3000	4500	12.6	9.3	26.9	30.0	19.2	57.1	68.2	32.0	119.5	1642	29
-2	3000	4500	10.9	4.9	20.3	35.6	29.5	79.4	73.2	27.3	121.6	1606	106
-3	3000	4500	7.0	3.2	10.9	31.8	8.1	46.2	67.4	18.6	89.7	1601	76
-4	3000	4500	7.6	1.9	10.5	33.4	10.4	51.5	56.4	8.1	67.5	1652	33
9D-1	4000	4500	6.4	1.7	8.3	25.0	8.3	37.1	48.6	8.6	67.2	1648	66
-2	4000	4500	8.5	3.0	13.2	30.4	4.2	37.4	56.5	11.2	72.7	1657	32
-3	4000	4500	9.2	3.6	15.4	33.4	6.3	40.5	59.9	10.5	74.9	1622	109
Type 310 Stainless Steel													
9B-1	500	500	4.1	2.0	5.4	21.3	10.2	33.7	27.0	7.5	38.4	1670	18
-2	500	500	5.5	1.3	6.8	12.8	5.9	16.9	33.2	3.6	35.8	1673	18
-5	500	500	5.5	0.7	6.4	12.9	3.5	20.3	21.4	4.6	25.1	1677	25
-6	500	500	5.2	2.4	7.8	15.2	3.1	18.2	24.7	6.7	35.5	1671	18
9D-3	500	500	4.4	1.5	5.9	14.2	4.5	20.2	30.6	5.8	38.4	1675	22
-4	500	500	6.3	2.0	9.2	12.8	4.9	21.4	31.0	9.6	45.0	1663	20
9C-1	1000	1500	5.7	1.8	8.2	28.2	13.2	40.7	34.1	15.1	53.4	1676	15
-2	1000	1500	6.3	2.2	10.2	18.9	5.5	26.7	32.7	5.6	40.8	1672	11
-3	1000	1500	5.5	1.7	8.2	23.8	10.0	39.6	42.8	13.6	55.0	1676	23
6B-1	1500	1500	6.2	2.4	10.7	17.8	8.1	30.4	36.6	7.5	44.8	1677	19
-2	1500	1500	6.0	1.0	7.0	17.4	6.6	28.7	37.2	7.3	48.8	1664	18
-5	1500	1500	6.4	1.4	8.9	21.5	7.1	31.0	38.5	6.4	49.1	1671	20
-6	1500	1500	5.9	0.8	7.2	19.5	6.6	27.2	40.5	18.8	52.8	1673	21
6B-1	3000	4500	7.2	3.7	15.8	52.8	17.3	86.1	61.1	17.8	94.5	1685	6
-2	3000	4500	7.1	2.5	11.8	21.0	8.1	34.0	40.2	8.7	52.5	1674	10
-3	3000	4500	6.2	2.4	10.7	18.7	16.0	53.4	43.6	17.3	79.6	1688	21
-4	3000	4500	6.2	1.8	10.1	15.5	9.2	32.2	33.9	6.3	44.0	1677	9
9C-1	3000	4500	6.2	2.6	8.3	40.2	14.4	57.2	53.9	10.5	70.8	1704	40
-2	3000	4500	5.6	1.3	7.6	23.4	8.4	33.3	36.3	5.3	43.0	1680	26
-3	3000	4500	6.7	2.7	13.0	26.2	12.9	47.2	51.3	10.3	63.3	1684	26
3C-1	4500	4500	13.5	4.8	21.5	34.2	29.2	99.0	52.8	25.4	57.4	1659	26
-2	4500	4500	11.2	2.8	17.5	37.3	13.0	54.2	63.2	13.6	77.9	1654	21
-3	4500	4500	12.4	1.9	15.2	45.9	11.0	65.3	69.0	7.2	81.7	1659	20
8D-1	4500	4500	7.2	1.2	8.8	21.1	7.8	31.1	49.8	14.6	71.5	1659	23
-4	4500	4500	7.5	1.1	9.0	30.6	3.5	37.0	58.5	3.6	62.3	1665	17
-5	4500	4500	6.7	1.7	9.1	44.2	17.2	82.9	70.3	30.3	102.2	1698	52
-6	4500	4500	7.0	1.0	8.4	34.4	15.2	67.4	54.8	16.5	87.2	1694	30

Table F. (Continued)

Tube Sample ^b	Exposure (h)	Removal Time ^c (h)	Oxide Scale Thickness, μm			Intergranular Corrosion Depth, μm			Depth, μm , of Sulfide Inclusions			Tube Wall Thickness	
			av	s.d.	max	av	s.d.	max	av	s.d.	max	av	s.d.
Type 316 Stainless Steel													
8C-1	500	2000	5.8	2.2	8.2	11.9	7.8	22.2	17.8	8.8	31.0	1628	43
-2	500	2000	4.9	1.6	7.6	14.3	6.8	23.0	16.2	8.8	29.2	1584	11
-3	500	2000	4.7	1.6	6.6	7.8	2.8	12.4	14.2	3.6	19.9	1601	9
-4	500	2000	6.6	3.5	11.5	16.4	13.4	45.5	18.2	13.1	48.2	1602	8
-5	500	2000	6.4	1.7	9.8	21.9	9.8	29.7	31.5	10.3	43.0	1559	14
-6	500	2000	4.2	0.6	5.5	8.6	2.3	11.0	16.3	3.6	20.7	1584	13
8C-1	1000	1500	12.9	7.4	28.0	33.4	17.4	48.7	44.8	13.9	73.5	1631	27
-2	1000	1500	8.4	2.7	14.1	21.0	9.1	38.3	40.2	9.6	52.4	1631	24
-3	1000	1500	9.9	2.5	13.2	38.6	8.8	48.9	58.2	9.2	68.1	1632	25
8C-1	1000	3000	10.2	5.0	20.5	28.6	7.7	37.5	32.6	13.2	49.0	1630	23
-2	1000	3000	10.8	5.0	19.4	21.6	9.4	36.2	34.7	17.7	67.7	1539	21
-3	1000	3000	10.0	2.4	13.8	29.7	20.4	77.4	42.2	23.3	94.7	1539	21
-4	1000	3000	8.9	1.1	10.7	35.3	23.7	82.8	48.5	25.0	99.5	1540	12
-5	1000	3000	10.7	1.2	11.5	31.6	9.0	40.5	40.6	10.0	36.4	1535	12
-6	1000	3000	17.2	8.3	32.9	58.8	26.2	100.3	74.1	26.6	119.3	1528	29
3B-1	1500	1500	8.4	2.1	12.4	26.2	8.1	37.5	46.0	10.7	61.8	1643	11
-2	1500	1500	8.6	1.4	11.2	22.6	4.3	28.8	46.7	5.1	54.1	1630	9
-3	1500	1500	8.0	1.0	9.4	21.9	6.6	36.1	46.8	8.6	59.7	1636	8
-4	1500	1500	9.0	1.8	11.9	30.9	11.4	50.1	48.4	8.4	62.1	1646	14
8C-1	1500	4500	13.2	5.8	21.4	48.0	21.8	73.0	66.5	19.2	90.1	1559	26
-2	1500	4500	14.0	7.0	22.3	52.5	13.9	77.8	68.7	15.4	97.1	1530	21
3B-1	3000	4500	24.7	15.9	46.7	40.8	18.7	68.1	55.8	24.6	87.7	1576	11
-2	3000	4500	16.2	41.6	21.2	36.9	20.8	60.3	50.9	17.7	68.8	1577	14
-3	3000	4500	9.9	3.3	15.3	18.1	8.0	33.8	34.2	14.9	66.9	1592	21
4C-1	4500	4500	12.4	4.2	20.1	36.6	12.3	58.0	68.4	9.4	80.0	1622	24
-2	4500	4500	11.7	4.7	22.6	31.3	14.4	60.7	69.7	14.8	92.8	1628	41
-3	4500	4500	9.9	2.0	13.7	31.5	3.3	38.3	63.9	9.2	76.8	1632	12
6C-1	4500	4500	10.2	1.7	12.3	31.9	13.5	54.9	48.6	13.3	65.2	1632	13
-2	4500	4500	14.2	4.0	19.6	35.8	12.5	46.9	61.9	14.6	91.6	1619	19
-3	4500	4500	12.4	5.3	22.6	45.4	19.7	89.0	76.2	15.9	104.6	1626	9
Alloy 800													
2C-1	500	500	5.6	1.6	7.1	28.7	9.2	43.4	38.0	8.3	49.6	1893	30
-2	500	500	6.1	1.8	8.5	40.1	16.0	46.4	47.9	17.5	80.2	1888	29
-5	500	500	5.4	2.4	8.5	19.4	2.5	22.7	27.6	3.8	35.0	1895	27
-6	500	500	6.0	1.2	8.0	33.1	8.1	45.4	42.2	9.5	59.2	1909	31
9A-5	500	500	5.5	1.4	7.8	32.6	7.8	43.1	37.9	12.7	60.2	1898	3
-6	500	500	6.7	1.0	8.2	25.3	8.9	43.9	33.9	8.7	53.7	1902	4
9D-1	500	500	4.7	2.5	9.4	26.3	25.0	41.3	32.3	23.3	45.5	1903	7
-2	500	500	5.2	0.6	6.0	12.7	4.5	22.1	20.4	2.4	23.7	1900	9
9B-1	1000	1500	5.6	1.2	7.6	36.5	14.7	57.8	45.8	14.0	72.4	1835	13
-2	1000	1500	7.9	1.3	9.6	33.3	20.4	57.7	54.9	9.0	66.9	1837	22
-3	1000	1500	7.8	1.1	9.4	50.0	7.0	60.7	56.7	12.4	78.5	1837	24
5D-1	1500	1500	6.0	2.0	9.6	24.5	14.8	56.7	35.1	13.1	58.3	1899	33
-2	1500	1500	6.0	1.8	9.7	36.9	23.1	73.6	58.3	24.0	88.9	1895	32
-5	1500	1500	7.5	2.2	11.7	80.4	41.8	172.2	96.6	42.0	92.8	1889	31
-6	1500	1500	8.0	1.4	10.9	46.9	22.2	84.2	45.6	17.3	68.3	1895	47
2C-1	1500	2000	7.1	1.6	10.2	62.3	12.1	83.0	64.4	9.9	83.0	1838	15
-2	1500	2000	8.1	2.7	11.6	87.4	16.2	105.5	98.6	16.0	15.6	1834	16
-3	1500	2000	8.7	2.8	13.6	90.4	17.2	119.7	96.8	13.0	12.6	1835	20
8C-3	1500	4500	4.8	2.4	8.0	77.2	34.0	112.0	101.4	27.6	36.6	1851	19
-4	1500	4500	5.9	1.9	7.8	45.4	24.1	79.5	70.8	21.7	00.0	Ground	
5D-1	3000	4500	6.5	3.8	9.2	40.0	19.4	62.0	54.4	13.8	77.3	1850	14
-2	3000	4500	7.2	3.4	12.1	103.4	21.5	130.8	118.9	20.2	44.2	1860	27
-3	3000	4500	10.0	4.6	18.0	90.2	29.5	122.7	103.3	19.2	127.1	1861	28
4B-1	3000	4500	10.3	3.2	14.1	72.2	31.0	102.8	84.9	30.3	24.2	1801	32
-2	3000	4500	10.1	2.9	14.9	98.7	18.2	120.6	99.9	25.1	28.7	1854	27
-3	3000	4500	9.9	1.0	10.7	100.3	19.2	135.2	121.8	13.3	42.2	1803	31
2B-1	4500	4500	9.4	2.4	13.0	59.3	11.8	79.7	69.0	12.2	84.0	1901	18
-2	4500	4500	10.0	2.8	15.2	94.2	29.1	133.1	110.2	23.1	44.0	1891	10
-5	4500	4500	10.6	2.3	13.8	88.6	23.4	130.9	102.5	19.8	46.6	1892	23
-6	4500	4500	13.0	2.3	16.5	67.2	14.6	99.9	86.9	8.4	98.9	1904	24
4B-1	4500	4500	11.2	2.1	12.2	81.5	21.7	108.0	99.9	19.7	28.3	1891	36
-4	4500	4500	11.5	2.2	15.7	121.5	15.3	146.4	134.3	16.4	56.3	1890	29

^aFor each feature, values reported are averages of eight measurements taken around the circumference of the sample, the standard deviation, and the greatest value among the measurements.

^bThe tube is designated by the column and row within the test bed. For each tube the sample number increases from the inlet end (lower temperature).

^cTime during the 4500-h test at which sample was removed.

**THIS PAGE
WAS INTENTIONALLY
LEFT BLANK**

ORNL/TM-7734/P2
 Distribution
 Categories
 UC-90e, -h

INTERNAL DISTRIBUTION

1-2. Central Research Library	38. J. R. Keiser
3. Document Reference Section	39. C. C. Koch
4-5. Laboratory Records Department	40. R. P. Krishnan
6. Laboratory Records, ORNL RC	41. W. J. Lackey, Jr.
7. ORNL Patent Section	42. K. H. Lin
8. V. B. Baylor	43. C. T. Liu
9. R. A. Bradley	44. L. E. McNeese
10. A. J. Caputo	45. A. R. Olsen
11. P. T. Carlson	46. T. W. Pickel
12. J. V. Cathcart	47. P. L. Rittenhouse
13. R. H. Cooper, Jr.	48. A. C. Schaffhauser
14-18. J. H. DeVan	49. S. Y. Shiao
19. J. I. Federer	50. M. Siman-Tov
20. M. K. Ferber	51. G. M. Slaughter
21. A. P. Fraas	52. V. J. Tennery
22-26. T. G. Godfrey	53. P. F. Tortorelli
27. J. P. Hammond	54. G. C. Wei
28. W. A. Hartman	55. C. L. White
29. B. Heshmatpour	56. A. L. Bement, Jr. (Consultant)
30-32. M. R. Hill	57. A. P. Fraas (Consultant)
33. R. S. Holcomb	58. E. H. Kottcamp, Jr. (Consultant)
34. J. K. Huffstetler	59. Alan Lawley (Consultant)
35. H. Inouye	60. T. B. Massalski (Consultant)
36. J. E. Jones, Jr.	61. R. H. Redwine (Consultant)
37. R. R. Judkins	62. A. D. Rossin (Consultant)

EXTERNAL DISTRIBUTION

63. ALON PROCESSING, INC. Grantham St., Tarentum, PA 15084
 W. A. McGill
64. ARGONNE NATIONAL LABORATORY, 9700 South Cass Ave., Argonne, IL 60439
 Ken Natensan
65. BABCOCK AND WILCOX RESEARCH CENTER, P.O. Box 835, Alliance, OH 44601
 P. L. Daniel

66. BATTELLE-COLUMBUS LABORATORIES, 505 King Ave., Columbus, OH 43201
I. G. Wright
- 67-68. CURTIS-WRIGHT CORPORATION, One Passaic St., Wood-Ridge, NJ 07075
J. M. Mogul
L. Silvagi
69. ELECTRIC POWER RESEARCH INSTITUTE, 3412 Hillview Ave., Palo Alto, CA 94304
J. Stringer
- 70-71. FLUIDYNE ENGINEERING CORPORATION, 5900 Olson Memorial Highway, Minneapolis, MN 55422
D. G. DeCoursin
K. R. Drake
72. FOSTER WHEELER DEVELOPMENT CORPORATION, 12 Peach Tree Hill Road, Livingston, NJ 07039
J. F. Blough
73. LOCKHEED PALO ALTO RESEARCH LABORATORY, 3251 Hanover St., Palo Alto, CA 94304
R. A. Perkins
74. NATIONAL COAL BOARD, Coal Research Establishment, Stoke Orchard, Cheltenham, Glos. GL52 4RZ
A. J. Minchener
75. NATIONAL COAL BOARD, Coal Utilisation Research Laboratory, c/o BCURA Ltd., Randalls Road, Leatherhead, Surrey KT22 7RZ
Malcolm Wilkins
- 76-77. TENNESSEE VALLEY AUTHORITY, 1020 Chestnut St., Tower 2, Chattanooga, TN 37401
W. D. Goins
H. W. Withers
78. OHIO STATE UNIVERSITY, Dept. of Metallurgical Engineering, 116 West 19th Ave, Columbus, OH 43210
R. A. Rapp
- 79-80. UNIVERSITY OF CALIFORNIA, Lawrence Berkeley Laboratory, Berkeley, CA 94720
A. V. Levy
D. P. Whittle

81. UNIVERSITY OF KENTUCKY, Institute for Mining and Minerals
Research, Iron Works Pike, Box 13015, Lexington, KY 40583
A. A. Sagüés
82. UNIVERSITY OF PITTSBURGH, Dept. of Metallurgy, Pittsburgh, PA
15261
G. H. Meier
- 83-86. DOE, MORGANTOWN ENERGY TECHNOLOGY CENTER, P.O. Box 880,
Morgantown, WV 26505
J. W. Byam, Jr.
L. K. Carpenter
F. D. Gmeindl
L. D. Strickland
87. DOE, OFFICE OF BASIC ENERGY SCIENCES, 20 Massachusetts Avenue, N.W.,
Washington, DC 20545
Stanley M. Wolf
- 88-92. DOE, OFFICE OF FOSSIL ENERGY, 20 Massachusetts Avenue, N.W.,
Washington, DC 20545
S. J. Dapkunas
John Geffken
William Siskind
E. C. Trexler
G. Voelker
- 93-94. DOE, OAK RIDGE OPERATIONS OFFICE, P.O. Box E, Oak Ridge, TN 37830
Office of Assistant Manager for Energy Research and Development
E. E. Hoffman
L. L. Radcliffe
- 95-499. DOE, TECHNICAL INFORMATION CENTER, OFFICE OF INFORMATION SERVICES,
P.O. Box 62, Oak Ridge, TN 37830
For distribution as shown in TID-4500 Distribution Category,
UC-90e (Direct Combustion of Coal) and UC-90h (Materials and
Components)