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ATMOSPHERIC FLUIDIZED-BED COGENERATION AIR HEATER
EXPERIMENT: 1000-h LABORATORY TEST A

by

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ABSTRACT

A laboratory test program is described to evaluate the corrosion behavior of several metallic alloys, coatings, claddings, and weldments in support of the atmospheric fluidized-bed air heater experiment. Results are presented from the first 1000-h test (Test A) conducted at metal and gas temperatures of 871 and 899°C, respectively. Detailed information is also presented on the corrosion scale morphologies, scale compositions and thicknesses, intergranular penetration of the substrate material, and metal recession.

I. INTRODUCTION

The Atmospheric Fluidized Bed Cogeneration Air Heater Experiment (ACAHE) is a follow-on to the Generic Studies of Advanced Fluid-Bed Air Heater Technology and is intended to provide the data base required to proceed to the commercialization of the technology. The ACAHE project comprises several tasks coordinated between two major contractors, namely, Argonne National Laboratory (ANL) and Westinghouse Electric Corporation. Four different air heater cogeneration concepts were studied¹ in the project: (1) a fan- or turbine-exhaust-blown, bubbling, atmospheric fluidized-bed combustor (AFBC), (2) a fan- or turbine-exhaust-blown, circulating AFBC with a separate fluidized-bed heat exchanger, (3) a fan- or turbine-exhaust-blown coal pyrolyzer operated in series with an AFBC (the pyrolysis gas is combusted to raise turbine inlet temperature), and (4) a bubbling AFBC or coal pyrolyzer/AFBC that is pressurized to three to five atmospheres by part of the exhaust from a high-pressure turbine, while the remaining exhaust is expanded through a low-pressure turbine. During the course of the study, concerns were expressed over attempting to design, build, and warrant commercial facilities based on the above concepts without previous testing in FBC units. The ACAHE is being conducted to assess the performance of various heat exchanger materials at full commercial scale in order to increase confidence in the resultant designs of FBC air heater systems.

The purpose of this project then is to provide potential air heater manufacturers with needed information on the performance of an air-cooled heat exchanger operating in an FBC. The effort includes preparing test specifications, conducting laboratory materials tests, designing and fabricating of tube bundles, testing the tube bundles in an AFBC, and evaluating the test results. The purpose of the laboratory materials tests is to provide corrosion information on a variety of ASME-coded and noncoded structural materials, coatings, claddings, and weldments under a well-characterized laboratory test environment that simulates an FBC atmosphere. Table I lists test parameters, run identification, gas chemistry, and exposure time/cycling

TABLE I. Laboratory Materials Tests in Support of Cogeneration Program

Run Identification	Test Conditions					
	A	A2	E	G	F	C
Boiler Subcontractor	B&W, FW	B&W, FW	CE	CE	CE	FW
Test Priority	1	1	1	2	2	2
Gas Temperature, ^a °F	1650	1650	1650	1650	1650	1650
Metal Temperature, ^a °F	1600	1600	1600	1600	1175	1600
Oxygen Partial Pressure, atm	3×10^{-12}	3×10^{-12}	0.21	0.21	0.21	Cycling
Sulfur Partial Pressure, atm	$\sim 10^{-7}$	$\sim 10^{-7}$	-	-	-	Cycling
Deposit	CaSO ₄	CaSO ₄	CFB ash ^b	CFB ash ^b	CFB ash ^b	CaSO ₄
Cycle Period, h	500	500	500	500	500	500
Exposure Time, h	1000	3000	3000	1000	2000	2000
Gas Cycling	-	-	-	-	-	100-h low pO ₂ / 100-h high pO ₂

^aTo convert Fahrenheit values to Celsius, subtract 32 and multiply by 5/9.

^bAsh obtained from a circulating fluidized bed.

conditions. The listing includes all the tests that will be conducted in the laboratory test facilities in support of the data needs of Babcock and Wilcox (B&W), Foster-Wheeler (FW), and Combustion Engineering (CE) for selecting and designing tube bundle material. Priority 1 tests will be conducted first, followed by priority 2 tests. A listing of alloys that will be tested is given in Table II. Results from the first 1000-h test (test A) conducted in support of B&W and FW are presented in this report.

II. DESCRIPTION OF LABORATORY TEST PROCEDURE

A. Test Apparatus

In the corrosion experiment described below, ring specimens were used to simulate the heat exchanger tubes exposed to an environment arising from the combustion of coal. In addition, a number of experimental alloys were exposed in the form of flat coupons to evaluate their corrosion resistance. A schematic of the experimental setup is shown in Fig. 1. The experimental apparatus consisted of a corrosion probe to which the ring specimens of different alloys were attached. The probe was approximately 0.4 m in length, with a specially designed head that incorporated the cooling fluid path and thermocouples. A photograph of the fully assembled corrosion probe is shown in Fig. 2. The details of the ring specimens in the unassembled and assembled configurations with the thermocouples attached are shown in Fig. 3. The entire specimen probe was inserted in a reaction chamber constructed of aluminized Type 310 stainless steel. The specimens and gases were heated by resistance-wound heating elements surrounding the reaction chamber. The temperature readings of a control thermocouple at the outer surface of the reaction chamber were used to adjust the controller set points, thereby achieving the desired temperature.

TABLE II. List of Alloys That Will be Tested in Various ANL Material Tests

Form of Alloys/Coatings/Weldments	Test Identification		
	A, A2, C	E, G	F
Ring Specimens	304 (304/309/304)	304	T22
	800	800	T91
	RA 330	310	304
	310	347	347
	316	304/IN82/310	T91/304
	Haynes 188	304/IN82/800	T91/310
	CoCrAlY on 800H	304/308L/347	T91/800H
	800/310/304H	310/IN82/800	T91/347
	21-33 Nb-Mn/800	800 Chromized	T22 Chromized
	800H Chromized	304 Chromized	T91 Chromized
	304H Chromized	CoCrAlY on 800H ^a	304 Chromized
Flat Coupons	253MA	253MA	CoCrAlY on 800H
	556	430	253MA
	HK40	Sandvik Mn-Nb 800	HK40
	FW-4C	CoCrAlY on 800H	HR-3C
	HR-3C	54E	FW-4C
	Mn-Nb on 800H	53C	Sandvik Mn-Nb 800
	54E	RV 8413	
	53C	INCO alloy	
	RV 8413	Ni Aluminide	
	INCO alloy	(IC-50)	
	Ni Aluminide	Fe Aluminide	
	(IC-50)	(FA-41)	
	Fe Aluminide	Ni-Cr Aluminide ^b	
	(FA-41)	(IC-266)	
	Ni-Cr Aluminide ^b		
	(IC-266)		

^aTest G only.^bPreoxidized.

Fig. 1. Schematic of the Experimental Setup Used in the Present Studies.

Fig. 2. Photograph of Fully Assembled Corrosion Probe.

Fig. 3. Details of the Ring Specimens in the Unassembled and Assembled Configurations with the Thermocouples in the Attached Position.

B. Temperature Control and Measurement

The temperature of the simulated combustion gas was controlled and maintained at 900°C using the chromel-alumel thermocouple located at the outer surface of the reaction chamber. The gas temperature was measured using a thermocouple inserted in the thermowell from the top flange of the reaction chamber. Two Pt/Pt-Rh thermocouples were spot welded (onto outer surfaces) such as to be on the top and bottom samples of the specimen assembly and were used to measure the metal temperature. In addition, three chromel/alumel thermocouples were spot welded on the inside surfaces of the specimens at the top, center, and bottom levels of the specimen assembly. The inside thermocouples were also used to monitor the metal temperature since those on the outside (fireside) were generally degraded following long exposure in the presence of the deposit material and sulfur-containing gas. The output of all the thermocouples was either recorded on a strip chart recorder or monitored in a Data-Pro digital display unit for the entire course of the run.

The corrosion test probes were thermally cycled by shutting off the furnace after every 500 h of exposure at the test temperature. The gas and metal temperatures were monitored during both the heatup and cooldown cycles of the run. Typical time-temperature profiles for gas and metal during heatup and cooldown are shown in Fig. 4. After they were cooled to room temperature, the exposed specimens were examined visually, photographed, and recoated with the deposit material; they were then inserted into the reaction chamber, and the test was restarted.

Fig. 4. Typical Time-Temperature Profile for Gas and Metal during Heatup and Cooldown Cycle.

C. Gas Chemistry and Flow Control

The primary purpose of conducting the corrosion tests in the laboratory setup was to simulate the environment that is relevant to subsequent testing of tube bundles at the Rocketdyne test facility in Canoga Park, CA. Consequently, the desired gas chemistries for various tests were established jointly by ANL, B&W, FW, and CE; these chemistries were based on the mechanistic understanding of the corrosion processes that occur in the presence of deposits and in simulated combustion atmospheres.^{2,3} The desired oxygen and sulfur partial pressures in the exposure environment for test A were $\sim 1 \times 10^{-12}$ and 1×10^{-7} atm, respectively, at a gas temperature of 1650°F (899°C). Thermodynamic calculations were made to determine the room-temperature inlet gas composition that will establish the desired oxygen and sulfur partial pressures at the elevated temperature of the test. Based on the calculations, a gas mixture containing 1 mL of CO, 200 mL of CO₂, 0.5 mL of SO₂, and 49.5 mL of N₂ was selected as the inlet gas mixture. This mixture establishes partial pressures (in atm) of $p_{O_2} = 3.1 \times 10^{-12}$, $p_{S_2} = 9.7 \times 10^{-8}$, $p_{SO_2} = 2 \times 10^{-3}$, and $p_{SO_3} = 1.1 \times 10^{-9}$ at a temperature of 1650°F (899°C). The environment established by this gas mixture is shown in Fig. 5, which depicts the CaO-CaS-CaSO₄ stability phase fields as a function of oxygen and sulfur partial pressures. Gas mixtures of 1 vol % CO in CO₂ and 1 vol % SO₂ in N₂ and pure CO₂ were procured for use in the tests for B&W and FW.

Flow rates of mixed gases and pure CO₂ were adjusted using on-line flowmeters to achieve the desired composition. The flowmeters were calibrated over a wide range with various gases, and calibration curves (see Fig. 6) were developed for several flowmeter systems. Each of the mixed and pure gases from the cylinders flowed through low-pressure regulators and flowmeters before they mixed and entered the reaction chamber. The final gas mixture flowed through a 4-mm-OD quartz tube preheater that was located inside the reaction chamber before the gas was passed over the specimens. The flow rates of all the inlet gases were monitored periodically to ensure that the desired inlet gas mixture was present at all times during the test.

D. Specimen Description

Candidate metallic alloys for the tests were chosen based on an extensive review of the corrosion data that have been developed over the years in several FBC units.⁴ The alloys were in either ring or flat-coupon form, and their chemical compositions are listed in Table III. The ring specimens were fabricated from a 0.5-in. (1.27-mm) pipe size, schedule 40 pipe material of several alloys. Tables IV and V list the alloys and coatings/claddings/weldments, respectively, and indicate the supplier/fabricator for various specimens used in the laboratory test program. A fabrication drawing of the ring specimens indicating detailed dimensions is shown in Fig. 7. Eleven ring specimens were assembled in the corrosion probe. In addition, a number of alloys in the form of flat coupons were exposed to the same atmosphere as the ring specimens; however, the ring specimens were internally cooled with air to achieve a metal temperature of 1600°F (871°C), while the flat coupons (without cooling) were tested at the gas temperature of 1650°F (899°C). The coupon specimens were ~ 5 to 10 mm in length, 5 mm in width, and ~ 1 -2 mm in thickness and had holes ~ 1 to 2 mm in diameter to suspend them from a quartz tree within the reaction chamber. The surfaces of all the bare specimens were fine polished. The clad and coated alloys were tested in the as-clad and as-coated

Fig. 5. CaO-CaS-CaSO₄ Phase Stability Fields Calculated for Several Temperatures. The dashed line represents thermodynamic gas phase equilibria in the combustion gas in the absence of sulfur sorbents. The gas environment of tests A and A₂ is indicated by a solid circle.

conditions; the weldment specimens were tested in the as-welded condition (see Table III).

E. Deposit Composition and Application Procedure

In the present test, the outer surfaces of the ring specimens and one surface of the flat coupons were coated with reagent-grade CaSO₄, whose analysis is given in Table VI. The deposit material was made into a water-based paste and applied onto the alloy specimens. The thickness of the deposit layer was in the range of 0.25 to 0.50 mm. The specimens with the deposit were dried overnight at room temperature and visually examined to ensure adherence of deposit to the substrate. The corrosion probe specimens and the flat coupons coated with deposit material were loaded into the reaction chamber and heated to the desired temperature in the test environment. The corrosion test probes were cooled to room temperature after every 500 h of exposure at the test temperature. During the cooldown period, the specimens were recoated with the deposit material to ensure that there was no starvation of reactant for the continuation of the corrosion process. In test A, which had a total exposure time of 1000 h, specimens were recoated once after a 500-h exposure. In tests such as A₂, with a total exposure time of 3000 h, recoating was done after every 500 h of exposure for a total of five times.

Fig. 6. Calibration Curves for Several Flowmeters for Various Gas Mixtures Used in the Present Tests.

F. Specimen Examination

Upon completion of the exposures, both macroscopic and microscopic examinations of various specimens were conducted. Cross sections of the ring specimens were examined using a scanning electron microscope (SEM) equipped with an energy dispersive x-ray analyzer (EDAX) and an electron microprobe to identify the morphological features of corrosion product phases in the scale layers and to establish the thickness of scales and depths of intergranular penetration, if any, of the substrate material. The measurements were made both on the deposit-coated and air-exposed sides of ring specimens and deposit-coated and gas-exposed sides of flat-coupon specimens. The measurements involved determination of the maximum values and average values (average of three) for scale thickness and penetration depth. In addition, optical metallography was used to determine the unaffected wall thickness of various exposed specimens. The results were used with initial wall thicknesses of unexposed material to calculate surface recessions in the specimens. In the case of coated specimens, concentration gradients for various elements in the coating and substrate alloy were determined in both as-coated conditions and after exposure to the test environment. The weldment specimens were examined in detail to establish (1) the hardness variation across the interface from the base metal to the filler metal and (2) microstructures of the filler metal, base metal, and the heat-affected zone.

TABLE III. Chemical Composition of Alloys, Claddings, and Coatings Used in the Present Program

Material	C	Cr	Ni	Mn	Si	S	Mo	Cu	Al	Fe	Other
304	0.08	18.34	8.10	1.50	0.27	0.018	0.27	0.16	-	Bal.	-
800	0.08	20.12	31.70	0.96	0.24	0.006	0.30	0.73	0.39	Bal.	Ti 0.31
RA 330	0.05	18.59	35.14	1.13	0.92	0.006	0.18	0.10	-	Bal.	Nb 0.12
310	0.07	25.00	18.73	1.21	0.64	0.006	0.002	0.14	-	Bal.	-
316	0.05	17.27	13.79	1.60	0.73	0.016	2.20	0.17	-	Bal.	-
188	0.08	23.4	23.3	0.7	0.4	-	0.6	-	-	1.4	Co 35.7, Al 0.22, W 14.6, La 0.05
CoCrAlY/800H	-	22.6	4.9	-	0.53	-	-	-	12.4	1.0	Co 59.5, Y 0.65
21-33 Nb-Mn FM ^a	0.19	22.2	33.1	1.58	0.35	-	-	-	-	Bal.	Nb/Ta 2.0, Ti 0.07
309 FM	0.11	23.0	14.7	0.54	0.74	-	-	-	-	Bal.	-
T91	0.084	8.57	0.09	0.46	0.40	0.003	1.02	-	0.014	Bal.	N 0.053, Nb 0.073, V 0.198
T22	0.12	2.79	0.20	0.35	0.27	0.022	0.96	-	-	Bal.	N 0.014
347	0.079	17.68	12.37	1.77	0.37	0.002	-	-	-	Bal.	Nb 0.69
IN82 FM	0.02	18.7	Bal.	3.9	0.30	-	0.68	-	-	2.74	Nb/Ta 2, Ti 0.07
308L FM	0.025	21.0	10.0	1.75	0.40	-	-	-	-	Bal.	-
253MA	0.10	20.7	10.9	0.3	1.8	-	-	-	-	Bal.	Ce 0.03
556	0.10	22.0	20.0	1.5	0.4	-	3.0	-	-	Bal.	Co 20, W 2.5, La 0.02, Nb+Ta 1
HK40	0.40	28.0	20.0	2.0	2.0	-	0.5	-	-	Bal.	-
FW-4C	0.054	19.5	20.0	4.9	2.60	-	-	-	1.4	Bal.	-
HR-3C	0.06	24.8	2.04	1.24	0.40	-	-	-	-	-	Nb 0.48, N 0.247
Mn-Nb Mod. 800H	0.069	22.09	30.32	4.48	0.13	0.003	-	-	-	Bal.	Nb 0.91, N 0.042, Ce 0.08
54E	0.016	21.79	24.98	4.99	0.10	-	1.92	-	-	Bal.	N 0.087
53C	0.016	21.5	17.14	4.90	0.12	-	-	-	-	Bal.	N 0.251
RV 8413	0.005	18.53	0.06	0.001	0.025	0.003	-	-	5.91	Bal.	Hf 0.50
IC-50	-	-	Bal.	-	-	-	-	-	12.2	-	Hf 1.7, B 0.05
FA-41	-	-	-	-	-	-	-	-	15.0	Bal.	TiB ₂ 1.0
IC-266	-	8.2	Bal.	-	-	-	-	-	9.3	-	Zr 1.8, B 0.02
INCO Alloy	-	20.15	32.5	-	-	-	-	-	3-4	Bal.	-

^aFM indicates filler metal only.

TABLE IV. Listing of Metallic Alloys Indicating the Supplier/Fabricator for the Specimens Used in the Program

Material	Supplier/Fabricator			
	ANL	FW	B&W	CE
<u>Ring Specimens:</u>				
304H	X			
800H	X			
RA330	X			
310	X			
316	X			
347	X			
T22	X			
T91	X			
Haynes 188	X		X	
<u>Flat-Coupon Specimens:</u>				
556			X	
FW-4C		X		
HK40		X		
HR-3C		X		
Sandvik Mn-Nb 800H			X	
253 MA		X		
430	X			
Fe-18Cr-6Al (RV 8413)		X		
FM 53C		X		
FM 54E		X		
INCO Alloy			X	
Ni Aluminide (IC-50)	X			
Fe Aluminide (FA-41)	X			
Ni-Cr Aluminide (IC-266)	X			

TABLE V. Listing of Coatings and Weldments Indicating the Supplier/Fabricator for the Specimens Used in the Program (Ring Specimens)

Material	Supplier/Fabricator			
	ANL	FW	B&W	CE
CoCrAlY on 800H	X		X	
T22 Chromized	X			X
T91 Chromized	X			X
304H Chromized	X			X
800H Chromized	X			X
304/310/800H	X		X	
304/309/304	X		X	
T91/IN82/304	X			X
T91/IN82/310	X			X
T91/IN82/347	X			X
T91/IN82/800H	X			X
304/IN82/310	X			X
304/IN82/800	X			X
304/308L/347	X			X
310/IN82/800H	X			X
21-33/20-32Nb/800H	X	X

Fig. 7. Engineering Drawing of Ring Specimens Used in Corrosion Tests.

TABLE VI. Analysis of Reagent-Grade CaSO_4 Used in the Present Test

Alloy $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$	99.2%
Insoluble in HCl	<0.03%
Chloride (Cl)	<0.002%
Nitrate (NO_3)	0.005%
Carbonate (CO_3)	Passes test
Heavy Metals (as Pb)	<0.001%
Iron (Fe)	<0.005%
Mg and Alkali Salts (as SO_4)	0.04%

G. Characterization of Coated Specimens

Chromized samples of Type 304 stainless steel and Incoloy 800H, used in test A, were prepared by CE of Chattanooga, TN. The CoCrAlY coating on Incoloy 800 was prepared by Plasmadyne Corp. Figure 8 shows the hardness profile for the as-chromized 304H stainless steel specimen, etched to accentuate the coating/substrate interface region. Figure 9 shows the concentration profiles for Fe, Cr, and Ni in the as-coated 304H stainless steel; the chromium concentration ranges from a peak value of ~62 wt % at the surface to ~32 wt % at ~200 μm from the surface. Figures 10 and 11 show the hardness and concentration profiles for as-chromized Incoloy 800H specimens. A comparison of Figs. 8 and 10 indicates that the hardness level of chromized 800H alloy is ~75% higher than that of chromized 304H, while the thickness of chromized

Fig. 8. Hardness Profile for As-Chromized Type 304H Stainless Steel Specimen.

Fig. 9. Fe, Cr, and Ni Concentration Profile for As-Chromized Type 304H Stainless Steel Specimen.

Fig. 10. Hardness Profile for As-Chromized Incoloy 800H Specimen.

Fig. 11. Fe, Cr, and Ni Concentration Profile for As-Chromized Incoloy 800H Specimen.

layers is approximately the same in both specimens. Figures 12 and 13 show the hardness and concentration profiles for the CoCrAlY-coated Incoloy 800H specimen. Even though the hardness profile shows significant variability across the coating, the coating is at least a factor of two higher in hardness than the substrate alloy. The coating had a Co content of ~60 wt % and Cr and Al levels of ~23 and 14 wt %, respectively. The coating exhibited Fe and Ni in a concentration range of 2 to 5 wt %.

Fig. 12. Hardness Profile for CoCrAlY-coated Incoloy 800H Specimen.

H. Characterization of Weldment Specimens

Three different weldment specimens were exposed in test A of the program. Hardness profiles were determined on two of the weldments, namely, Type 304 SS welded with Type 309 SS filler metal and Incoloy 800 welded to Type 304 SS with Type 310 filler metal. A hardness profile was not determined for the Incoloy 800 specimen welded with 21-33 Nb filler metal because the number of specimens was inadequate. Figure 14 shows the hardness profiles for the two weldments identified above. Figure 15 shows the microstructure of the 304/309/304 weldment specimen observed after two different etching treatments, namely, 10% oxalic acid and 20% Picral-80% HCl. The dendritic structures of the filler metal zones are evident in both specimens. In addition, the magnified photographs of the different regions of the weldment specimen show a sharp contrast in structure between the 309 SS filler metal and the 304 SS base metal. Figure 16 shows the Cr, Fe, and Ni concentration profiles from the base metal across the heat-affected zone to the filler metal region. The profiles essentially reflect a slightly higher level in Cr and Ni concentrations in the filler metal compared with those in base metal.

Figure 17 shows the microstructure of the Incoloy 800/Type 310/Type 304 weldment specimen observed after the two etching treatments. Detailed microstructures of different regions of the specimen show a somewhat smaller grain size for the 800H base metal when compared with that for Type 304 SS

Fig. 13.
Elemental Concentration Profiles for
CoCrAlY-coated Incoloy 800H Specimen.

base metal. Figure 18 shows the Cr, Fe, and Ni concentration profiles for the weldment specimen in the 800/310 and 310/304 regions. The profiles for different elements for the 310/304 region reflect the initial concentration for the elements in the two materials. On the other hand, the profiles for the 800/310 region indicate significant readjustment of Ni and maybe Cr between the two alloys. For example, even though the Ni content in 800 and 310 base metals are 32.5 and 20 wt %, respectively, the profile for Ni in the weldment is almost flat.

Figure 19 shows the microstructure of the 800/21-33 Nb/800 weldment specimen observed after the two etching treatments. The pictures show that the V notch made prior to welding did not cover the entire wall thickness of the ring specimen, and the filler metal is seen only 2/3 of the wall thickness from the OD surface.

Fig. 14. Hardness Profiles for 800/310/304 and 304/309/304 Weldment Specimens.

Fig. 15. Microstructures of the 304/309/304 Weldment Specimen in the As-Welded Condition.

Fig. 16. Elemental Concentration Profiles for the 304/309/304 Weldment Specimen.

Fig. 17. Microstructures of the 800/310/304 Weldment Specimen in the As-Welded Condition.

Fig. 18. Elemental Concentration Profiles for the 800/310/304 Weldment Specimen.

Fig. 19. Microstructures of the 800/21-33 Nb/800 Weldment Specimen in the As-Welded Condition. (FM = filler metal.)

III. RESULTS

Figure 20 is a macroscopic photograph of specimens before and after exposure in test A. The photographs show that, even after 500 h of exposure, the deposit was fairly intact and adherent to the specimens. Some cracking of the deposit can be noted, which probably resulted on cooling of the sample probe from the test temperature. The initially applied deposit was physically removed from the specimen surfaces, and the samples were recoated with the deposit material for the second 500 h of exposure. The flat-coupon specimens, coated on one side, were hung from a quartz tree as shown in the photographs. Visual examination of the ring specimens on the corrosion probe showed significant swelling of the Incoloy 800 after 1000-h exposure.

A. Scale Morphologies in Base Alloys

Figures 21 and 22 show the corrosion-product morphologies observed in 310, 316, RA 330, 188, 304, and 800 alloys on both the deposit and air side of the ring specimens after a 1000-h exposure in Test A. The specimens have been etched in oxalic acid to accentuate the grain boundaries in the substrate alloy. The figures show that the stainless steel alloys such as 304 and 316, which contain Cr in the range 17 to 18 wt %, predominantly developed Cr,Fe oxide scales in both the deposit and air sides. The scale thicknesses were fairly small, and the substrate exhibited a significant amount of carbide precipitation at the grain boundaries. Type 310 stainless steel with a chromium content of 25 wt % developed a thin Cr-oxide scale in the presence of deposit and on the air-exposed surface. The substrate microstructure showed substantial precipitation of carbide and sigma phases.

Fig. 20. Macroscopic Photograph of Specimens before and after Exposure in Test A.

Fig. 21. Microstructures of Ring Specimens of Types 310 and 316 Stainless Steel and Alloy RA330 after Exposure in Test A.

Deposit side**Air side** **188** **304** **800**

Fig. 22. Microstructures of Ring Specimens of Haynes 188, Type 304 Stainless Steel, and Incoloy 800 after Exposure in Test A.

The corrosion behavior of alloys such as RA 330 and Incoloy 800, which contained ~31 to 35 wt % nickel, was quite different when compared with that of austenitic stainless steels. The RA 330 alloy developed a porous Cr-rich oxide scale on the deposit side of the specimen; the scale being nonprotective, substantial internal sulfidation of the substrate material was observed. Similar morphological features, but somewhat more severe attack, were observed in the case of Incoloy 800. On the air-exposed surfaces, the specimens developed thin Cr-oxide scales. The cobalt-base alloy 188 developed a massive oxide scale on the deposit side of the specimen, even though the nickel content is fairly low (23.3 wt %) and the chromium content is fairly high (23.4 wt %). The oxide scale was porous and had a large concentration of cobalt, indicating the growth rates of Co-containing oxides are significantly higher than that of Cr oxide. No sulfur was detected in the specimen. Even in the air-exposed surface, substantial internal oxidation of the specimen was observed, even though the surface oxide was a thin Cr-rich oxide.

B. Scale Morphologies in Coated Specimens

Ring specimens of chromized 304 and 800 alloys and CoCrAlY-coated 800 alloy were analyzed after 1000 h of exposure in Test A. Figures 23 to 25 show the elemental concentration profiles across the coating to the substrate regions for the three specimens. In all three alloys, the coatings had an interaction with deposit/gas environment, and thin layers of corrosion reaction product were observed. The thickness of these interaction zones was of the order of 30 to 50 μm in these specimens. Except for the interactions in the surface regions of the coatings, the elemental concentration profiles developed using EDAX analysis were found to be similar to those reported earlier (see Figs. 9, 11, and 13) for as-coated specimens. In the case of a CoCrAlY-coated 800 specimen, some external migration of iron and nickel was noted at the coating/substrate interface region.

C. Scale Morphologies in Weldment Specimens

Ring specimens of three weldments, namely, 304/309/304, 304/310/800, and 800/21-33 Nb/800 were exposed in Test A. Figure 26 is an SEM photograph of the 304/309/304 weldment specimen after exposure. The dendritic structure of filler metal (309 alloy) is evident. The interaction zone on the deposit side (OD surface in the figure) of the specimen is fairly small, indicating acceptable performance for the weldment based on the 1000-h test. Figure 27 shows the SEM photographs of 304/310 and 800/310 interfaces for a 304/310/800 weldment specimen after exposure in Test A. Very minimal corrosion was observed in the 310 filler metal and 304, 800 base metals in the vicinity of welded region. On the other hand, alloy 800 base metal further away from the welded region was significantly affected; in particular, sulfidation of the substrate material and intergranular penetration of the alloy were noted (see photograph at right in Fig. 27). The susceptibility of alloy 800 base metal in the weldment specimen to sulfidation corrosion is in agreement with that observed on the alloy 800 ring specimen reported earlier. The lack of attack in alloy 800 in the welded region cannot be explained at present; it is possible that some readjustment of Cr and Ni concentration would have occurred between the 310 filler metal and 800 base metal.

Fig. 23. Fe, Cr, and Ni Concentration Profiles for Chromized Type 304H Stainless Steel after Exposure in Test A.

Fig. 24. Fe, Cr, Ni, and Al Concentration Profiles for Chromized Incoloy 800H after Exposure in Test A.

Fig. 25. Co, Al, Cr, Fe, and Ni Concentration Profiles for CoCrAlY-coated Incoloy 800H Specimen after Exposure in Test A.

Fig. 26. SEM Photograph of the OD Side of a Ring Specimen of 304/309/304 Weldment Showing the 304 Base Metal, 309 Filler Metal, and the Weld Interface Region after Exposure in Test A.

Fig. 27. SEM Photographs of the OD Side of a Ring Specimen of 304/310/800 Weldment Showing the Base Metal Regions and the 304/310 and 310/800 Weld Interface Regions. The picture at the right shows the morphology of attack observed in Incoloy 800 base metal far from the welded region.

Figure 28 shows the SEM photographs of the 800/21-33 Nb/800 weldment specimen after exposure in Test A. The corrosion attack on both the filler metal and alloy was substantial; the matching composition of 21-33 Nb filler metal to that of alloy 800 base metal was not beneficial since the alloy 800 specimens were attacked severely in the simulated FBC environments of Test A. The scale morphology in alloy 800 (far away from the welded region) base metal developed in the welded specimen is also shown in Fig. 28. The sulfidation attack of the alloy and intergranular penetration of sulfur in the substrate are evident from this figure.

D. Scale Morphologies in Coupon Specimens

A number of flat-coupon samples of several alloys (see listing in Table II) were also exposed in Test A. One of the surfaces of the specimens was coated with CaSO_4 deposit, while the other was exposed bare to the gas phase. Figures 29 through 32 are SEM photographs of the morphologies of corrosion product layers on the deposit and gas side for various specimens tested.

Fig. 28. SEM Photographs of the OD Side of a Ring Specimen of 800/21-800/21-33Nb/800 Weldment after Exposure in Test A. The picture at the right shows the morphology of attack observed in Incoloy 800 base metal far from the welded region.

Alloy 253 MA containing 1.8 wt % Si exhibited (Cr,Fe)sulfide scale on the deposit side, while (Cr,Si)oxide was observed on the gas side of the specimen. An increase in the silicon content to 1.8 wt % from 0.3 to 0.4 wt % in conventional austenitic steels did not improve the corrosion resistance of the alloy in the presence of CaSO_4 deposit. Alloy 556, which contained ~20 wt % Co and 2.5 wt % W, developed Cr-rich oxide in the deposit side, but internal sulfidation of the substrate alloy was noted. On the other hand, the alloy developed Fe and (Co,Fe)oxide on the gas side of the specimen. The presence of Cr-rich oxide scale under the deposit indicates that the oxygen partial pressure under the deposit is much lower than that in the gas phase. However, the presence of internal sulfides in the substrate material on both sides of the specimen indicates that neither of the oxide scales are resistant to sulfur transport. Alloy HK40 (a cast version of Type 310 stainless steel), which contained ~0.4 wt % carbon, exhibited severe corrosion, especially in the deposit side of the specimen. The oxide was predominantly (Fe,Mn)type, which is porous enough to internally sulfidize the alloy. Even on the gas side of the alloy, the oxide seems porous enough to cause significant internal attack.

The FW-4C alloy developed (Fe,Cr)oxide scale on both the deposit and gas side exposed surfaces. In the region below the scale, chromium depletion was observed. Further into the alloy substrate, significant precipitation (probably of carbides) was observed, which was accentuated in Fig. 30 due to chemical etching of the specimen. Alloy HR-3C, in general, developed a very thin oxide scale; in isolated locations in the deposit side of the specimen, a sublayer of Cr sulfide was observed. However, the total attack of this alloy was very minimal. The Mn-Nb modified 800H exhibited substantial oxidation/sulfidation attack on the deposit side of the specimen and massive oxidation on the gas side of the specimen. A comparison of the results for this alloy with that for Incoloy 800 shows very little improvement in corrosion resistance by modification of the base alloy composition.

Fig. 29. Microstructures of Coupon Specimens of Alloys 253MA, 556, and HK40 after Exposure in Test A.

Fig. 30. Microstructures of Coupon Specimens of Alloys FW-4C, HR-3C, and Mn-Nb Modified 800H after Exposure in Test A.

Deposit side**Gas side** **54E** **53C** **RV 8413**25 μ m
└───┘

Fig. 31. Microstructures of Coupon Specimens of Alloys 54E, 53C, and RV8413 after Exposure in Test A.

Fig. 32. Microstructures of Coupon Specimens of Aluminide Alloys IC-50, FA-41, and IC-266 after Exposure in Test A.

Alloy 54E exhibited significant sulfidation on both deposit and gas side exposed surfaces. The susceptibility of the alloy to sulfidation even in a gaseous environment precludes the use of this alloy in FBC atmospheres. Alloy 53C developed very thin (Cr,Mn) oxide scales, and the extent of internal penetration was also minimal. Alloy RV 8413 developed Al-oxide scale on both the deposit and gas side exposed surfaces. However, significant precipitation of (Fe,Cr,Hf) intermetallic particles was observed in the alloy substrate. Even though the corrosion resistance in an FBC atmosphere for this alloy is good, the effect of the precipitates on the mechanical properties of the material should be determined.

The nickel aluminide material IC-50 developed predominantly a nickel-sulfide phase which can lead to a Ni-Ni₃S₂ eutectic at temperatures above 645°C. In prolonged exposures, the alloy will undergo catastrophic corrosion, since the protective oxide scale (alumina scale even if it forms) will be destroyed by the presence of the liquid sulfide. The iron-base aluminides with TiB₂ (alloy FA-41) formed (Fe,Al)oxide scales on both the deposit and gas side exposed surfaces. No internal penetration of the substrate alloy was noted; however, a distribution of fine (Fe,Al) intermetallic particles were obtained due to the exposure at elevated temperature. The preoxidized alloy IC266 exhibited (Cr,Al,Ni)oxide in both the deposit- and gas-exposed surfaces; however, the preoxidation treatment probably resulted in the oxide scale observed in the specimen. No sulfur is noted in the subscale region, but intergranular precipitation of oxide was observed to a depth of ~50 μm.

E. Corrosion Product Thickness Data

Scanning electron microscopy and optical metallography were used to determine the thickness of scale layers and depth of intergranular penetration in the substrate material. In addition, surface recessions in various specimens were calculated from the measurements on the exposed specimens and the initial wall thickness data. Table VII lists the scale thickness, depth of penetration, and metal recession data for ring specimens of the alloys, coatings, and weldments examined in Test A. Similar information for coupon specimens is listed in Table VIII.

IV. CONCLUSIONS

A laboratory test program is being conducted to evaluate the corrosion behavior of several metallic alloys, coatings, claddings, and weldments in support of the atmospheric fluidized-bed air heater experiment. Results from the first 1000-h test (Test A) are presented in this report. These results include detailed information on the corrosion scale morphologies, scale compositions and thicknesses, intergranular penetration of the substrate material, and metal recession. Based on the information, a number of conclusions can be drawn:

1. Austenitic stainless steels such as Types 304, 316, and 310 stainless steel showed acceptable corrosion behavior even in the presence of simulated sulfur sorbent.

TABLE VII. Scale Thickness, Depth of Penetration, and Metal Recession Data for Ring Specimens of Several Alloys, Coatings, and Weldments after Exposure in Test A

Material	Gas Side				Air Side				Metal Recession, μm
	Scale Thickness, μm		Penetration Depth, μm		Scale Thickness, μm		Penetration Depth, μm		
	Max.	Average	Max.	Average	Max.	Average	Max.	Average	
304	54	40	102	64	63	27	132	70	272
800	3900	3728	924	772	18	11	81	43	2514
RA 330	318	292	300	254	24	13	66	52	529
310	39	24	33	18	18	13	27	12	132
316	18	12	36	30	78	46	42	33	142
188	1542	1316	528	422	12	9	36	30	2426
CoCrAlY on 800	48	32	18	9	33	24	30	18	131
304/309/304	21/39/54	13/30/40	60/108/102	26/66/64	21/30/63	10/24/27	78/258/132	70/175/70	a
800/310/304	42/33/24	23/20/9	66/48/21	58/24/12	36/24/24	19/9/11	114/66/24	92/29/16	a
800/21-33/800	90/96/78	12/61/34	78/54/96	37/34/52	27/24/42	15/9/21	57/39/90	35/28/48	a
800 Chromized	138	64	96	48	138	68	138	80	388
304 Chromized	102	58	126	80	96	58	36	16	316

^aNot measured due to weld geometry.

TABLE VIII. Scale Thickness, Depth of Penetration, and Metal Recession Data for Flat-Coupon Specimens of Several Alloys after Exposure in Test A

Alloy	Deposit Side				Gas Side				Metal Recession, μm
	Scale Thickness, μm		Penetration Depth, μm		Scale Thickness, μm		Penetration Depth, μm		
	Max.	Avg.	Max.	Avg.	Max.	Avg.	Max.	Avg.	
253MA	40	25	57	24	25	5	50	30	103
556	114	40	150	75	40	25	300	200	420
HK40	252	122	252	126	180	125	200	100	518
FW-4C	66	38	72	25	35	20	80	30	121
HR-3C	54	11	42	11	10	5	30	20	90
Mn-Nb on 800H	906	261	618	439	-	920	-	-	1216
54E	170	82	272	170	-	100	250	150	744
53C	36	7	60	9	5	5	-	-	72
RV8413	30	14	40	25	5	5	-	-	95
IC-50	150	91	-	-	95	76	-	-	190
FA-41	5	-	-	-	3	-	-	-	13
IC-266	36	18	95	58	25	16	85	65	103

2. Alloys such as Incoloy 800 and Haynes 188 and, to a lesser extent, RA 330 were found susceptible to catastrophic attack, especially in the presence of CaSO_4 deposit.
3. The CoCrAlY-coated 800 specimen exhibited a low rate of scaling, but the stability of the coating under thermal cycling needs to be established since the underlying base metal is susceptible to catastrophic corrosion.
4. Alloys 304 and 800 with chromized surface treatments exhibited low to moderate attack of the coating layers.
5. All the three weldment specimens (ring type) exhibited minimal attack on both the deposit- and air-exposed surfaces; however, the susceptibility of the alloy 800 base metal (far away from the weld zone) to corrosion was very evident.
6. Among the coupon specimens, alloy 556, HK40, Mn-Nb modified 800H, and 54E exhibited significant corrosive attack in the presence of deposit and in the simulated combustion atmosphere.
7. Alloys such as 253 MA, FW-4C, HR-3C, 53C, and RV 8413 exhibited minimal attack in these environments. In particular, the alumina-forming alloy RV 8413 looked superior in its corrosion resistance.
8. Among the aluminide materials, nickel aluminide (IC-50) was found to corrode via formation of nickel sulfide in the presence of deposit and in the gas-phase environment. The iron-aluminide (FA-41) had the least attack, while the preoxidized nickel-base alloy IC-266 exhibited intergranular penetration in the substrate material.

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