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LONG TERM MATERIALS TEST PROGRAM

Quarterly Report for the Period January—March 1983

Work Performed Under Contract No. AC21-79ET15457

For
Morgantown Energy Technology Center
Morgantown, West Virginia

By
General Electric Company
Advanced Energy Programs Department
Schenectady, New York

Technical Information Center
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**Prepared for
U.S. Department of Energy
Assistant Secretary for Nuclear Energy**

**Prepared by
General Electric Company
Advanced Energy Programs Department
Schenectady, New York 12345**

ABSTRACT

Exposure of gas turbine materials to a PFBC effluent under the Long Term Materials Test Program has reached 1507 hours. Unprotected nickel and cobalt base blade and vane alloys show susceptibility to hot corrosion at all three metal temperatures investigated: 1500°F (gas temperature), 1300°F and 1100°F (air-cooled pins). Precious metal aluminide and M (Co,Fe) CrAlY overlay coatings continue to show good resistance to corrosion above 1450°F, but are susceptible to varying degrees of pitting attack between 1050°F and 1300°F.

Significant erosion/corrosion degradation of both base alloys and protective coatings/claddings has been observed on airfoil specimens exposed at 1350°F, 800-900 ft/s and dust loadings less than 100 ppm for 1085-hours. Corrosion predominately occurred in areas of direct particle impaction; i.e., leading edge and pressure surface, indicating an erosion/corrosion synergism. At gas velocities of 1200-1400 ft/s, a platinum-aluminide coated IN-738 pin experienced a metal recession rate of 8 mils/1000-hours.

The PFBC facility continues to show excellent operational reliability, accumulating over 1100 test hours this quarter. The only concern from an operations standpoint is the gradual thinning of the in-bed heat exchanger tubing at a rate of about 5 mils/100 hours off the diameter.

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SUMMARY

During the January-March 1983 quarter, 1107 hours of long term materials testing were completed. The PFBC test facility operated every week during this quarter except for two, one of which was a scheduled shutdown to change test specimens in the low-velocity and high-velocity test sections (1085 hours). No major operational problems were encountered, with the only persistent concern being the gradual wastage of the in-bed heat exchanger tubing at a rate of about 5 mils/100 hours off the tubing diameter. This metal loss was carefully monitored and the coil replaced after 1200 hours (actual heat exchanger operation was 1300 hours including startups and shutdowns).

Pittsburgh No. 8 coal and Pfizer dolomite continued to be used for the long-term test. Initial coal shipments (0-400 hour test period) contained sulfur and chlorine levels averaging 2.89 and 0.06 wt%, respectively. Subsequent shipments for the 400-1507 hour interval have averaged 4.74 wt% sulfur and 0.17 wt% chlorine.

A total of 1507 specimen exposure hours have been completed. After 1085 hours total test time, three sets of specimens with 800-hours exposure were removed from the LVTS for analysis of corrosion damage. These sets were identical to the specimens removed after 285 hours and included a complete group of materials at each of the three metal temperatures: 1500°F (un-cooled), 1300°F and 1100°F (air-cooled specimens). In addition, a second set of uncooled pins exposed for the full 1085-hours also was removed for metallurgical analysis. Corrosion penetration measurements from the LVTS pin specimens revealed the following:

- Reducing the metal temperature 200°-400°F below the gas temperature (1500°F) produces changes in the corrosion morphology but does not provide an overall reduction in corrosion rates. In many instances, corrosion rates actually increase at the lower temperatures.

- Ash deposits collected on the cooled pins contain up to an order of magnitude more alkali than deposits on the uncooled pins, strongly suggesting a vapor phase alkali deposition mechanism.
- MCrAlY and precious metal aluminide coatings show generally excellent corrosion resistance above 1450°F (0.5 mils/1000-hours), but are susceptible to varying degrees of pitting attack at 1050° to 1300°F.

The high-velocity test section was opened, and inspected for the first time after 1085 hours. All 24 airfoil specimens (4 each of 6 materials) and one erosion pin were removed for metallurgical evaluation of erosion/corrosion degradation. The following observations were made:

- Significant erosion/corrosion degradation of both base alloys, coatings and claddings occurred at 1350°F, 800-900 ft/s and dust loadings less than 100 ppm in the high-velocity test section. Corrosion predominately occurred in areas of particle impaction indicating an erosion/corrosion synergism.
- A metal recession rate of 8 mils/1000-hours was observed on a platinum-aluminide coated IN-738 pin exposed at 1200-1400 ft/s.

The next HVTS specimen inspection and replacement is scheduled to occur after approximately 2200 hours of testing. At that time, the airfoils will be metallurgically evaluated for erosion/corrosion degradation. New airfoils of four of the twelve materials screened during the first 2200 hours will then be installed for longer term exposures.

The next scheduled replacement of pins in the LVTS will take place after approximately 2600 hours of testing. Cooled and uncooled pins installed at the 1085-hour inspection will have been exposed for about 1500 hours.

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Technical Operations	- T. Bonds
Materials Support	- R. Brobst
Facility Operation and Maintenance	- A. deMaille
	- G. Goodrich
	- P. Jennes
	- E. Lambrecht
	- G. Logan
	- P. Palmer

This program is being ably directed from the Department of Energy by J. McClung at the Morgantown Energy Technology Center, Morgantown, WV.

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SECTION 1

TEST DESIGN AND QUALIFICATION (TASK 1)

This phase has been completed. No further effort is planned.

SECTION 2

TEST OPERATIONS (TASK 2)

During the January-March 1983 period, 1107 hours of testing were completed bringing the total number of hours on the long-term test to 1507. The combustor and test facility operated every week during this quarter except for two, one of which was a scheduled shutdown to change test specimens in the low-velocity and high-velocity test sections (1085 hours). No major operational problems were encountered. The only persistent concern was the gradual wastage of the inbed heat exchanger tubing at a rate of about 5 mils/100 hours off the tubing diameter. This metal loss was carefully monitored and the coil replaced after 1200 hours (actual heat exchanger operation was 1300 hours including startups and shutdowns). A discussion on tube wall thinning of the in-bed heat exchanger is presented later in this section.

Test Operations

The operating conditions for the long-term materials test are summarized in Table 2-1. The bed is operated at a nominal temperature of 1650°F, 10 atmospheres pressure and a fluidizing velocity of 2.4 to 2.7 ft/s. Excess air through the system is kept at a minimum of 20 percent and is generally in the 30 to 40 percent range. Combustion efficiency is consistently greater than 98 percent.

The temperature in the low-velocity test section varies between 1440° and 1520°F at a gas velocity of 23 ft/s. The high-velocity test section operates at nominally 1350°F and velocities of 800 to 1400 ft/s with dust loadings varying between 31 and 77 ppm.

Chemical analyses of the coal and dolomite are listed in Tables 2-2 and 2-3 respectively. Pittsburgh No. 8 coal with 3-5 wt% sulfur and about 0.10 wt% chlorine was selected for use in the long-term test. The shipments of coal for the first 400 hours of testing contained sulfur and chlorine levels averaging 2.89 and 0.06 wt%, respectively. Subsequent shipments used for the 400-1507 hour test interval have averaged 4.74 wt% sulfur and 0.17 wt% chlorine.

TABLE 2-1
LTMT Operating Conditions

Combustor

Coal	Pittsburgh No. 8
Dolomite	Pfizer
Bed Temperature	1640°-1660°F
Bed Pressure	150 psia
Fluidizing Velocity	2.4-2.7 ft/s
Bed Height	6 ft Nominal
Excess Air	30-40%
Ca/S	1.7-2.3

Low-Velocity Test Section (LVTS)

Gas Pressure	145 psia
Gas Velocity	23 ft/s
Inlet Temp	1490°-1520°F
Outlet Temp	1440°-1480°F
Cooled Pin Temps	1300° [±] 50°F, 1100° [±] 50°F

High-Velocity Test Section (HVTS)

Temp	1325°-1375°F
Dust Loading ⁽¹⁾	31-77 ppm
Mean Particle Size (microns) ⁽¹⁾	2.0-3.1
Percent Particles >10 microns ⁽¹⁾	2-7
Gas Velocity	800-900 ft/s (Airfoils) 1300-1400 ft/s (Erosion Pin)
Gas Pressure	35 psia

(1) Range of 8 analyses

TABLE 2-2

Coal Analysis
Pittsburgh No. 8
(Wt%)

<u>Proximate Analysis (As Received)</u>	<u>0-400 Hrs (Avg of 2)</u>	<u>401-1507 Hrs (Avg of 4)</u>
Moisture	2.05	2.89
Ash	14.40	13.24
Volatile	34.44	35.79
Fixed Carbon	49.12	48.08
Btu/lb	12398	12455
<u>Ultimate Analysis (As-Received)</u>		
Moisture	2.05	2.89
Carbon	68.30	67.72
Hydrogen	4.83	4.77
Nitrogen	1.32	1.29
Chlorine	0.06	0.17
Sulfur	2.89	4.74
Ash	14.40	13.24
Oxygen	6.16	5.18
<u>Ash Analysis</u>		
SiO ₂	50.10	33.63
Al ₂ O ₃	22.13	18.99
Fe ₂ O ₃	19.55	40.70
TiO ₂	1.02	0.84
CaO	2.26	1.27
MgO	0.79	0.65
K ₂ O	2.02	1.34
Na ₂ O	0.19	0.32
SO ₃	1.48	1.32

TABLE 2-3

Pfizer Dolomite Analysis
(Range of Two Analyses)

	<u>Wt%</u>
CO ₂ (Loss)	43.7-45.9
CaO	54.90-55.01
MgO	39.48-39.90
SiO ₂	2.63-4.05
Al ₂ O ₃	0.74-0.99
Fe ₂ O ₃	0.17-0.38
K ₂ O	0.0
Na ₂ O	0.07-0.26
SO ₃	0.09-1.16
Cl ⁻	0.004-0.24

Pfizer dolomite is mixed with coal at a constant ratio of 100 lbs coal/55 lbs dolomite to yield a Ca/S ratio of 1.7 to 2.3. Sulfur capture typically ranges from 91 to 95 percent based on 5 percent sulfur coal. Pfizer was no longer mining and supplying dolomite as of 1/31/83, consequently, a search was undertaken to locate a new supplier of dolomite with similar chemical and physical properties to the Pfizer material. There was enough Pfizer dolomite in-house to reach the 2500-hour mark of the long-term test.

Figure 2-1 shows the temperature profile in the bed region, freeboard, cyclones and test sections for the current test conditions compared to previous runs during the corrosion validation testing phase of this program. The profiles show a much more uniform temperature distribution in the bed region with lower heat losses through the system (compare the 2/25/82 profile with the 1/11/83 profile both at nominal 1650°F bed conditions).

The improved temperature profile throughout the system is directly related to the reinsulation of the in-bed heat exchanger downcomers, the lower combustor section, cyclone pressure vessel and tee-section ahead of the low velocity test section, performed during the April-June 1982 period. Figure 2-2 shows the typical variation in the system temperatures over an 8-hour period.

Another system improvement, implemented in October 1982, was the activation of an automatic controller for monitoring bed temperature and regulating coal-feed to maintain a constant bed temperature. Coal feed had been manually regulated, during the corrosion validation tests, resulting in relatively large temperature fluctuations, $\pm 30^{\circ}\text{F}$, compared to the current $\pm 10^{\circ}\text{F}$ fluctuations.

Heat and Mass Balances

Overall heat and mass balances are presented in Tables 2-4 and 2-5. Excellent closure (typically less than $\pm 5\%$) between the energy and mass inputs and outputs are shown, as anticipated for these calculations. Each balance represents a single data point selected during a period of steady-state test operations.

Figure 2-1 - Temperature Profile Throughout Malta PFBC Test Facility

Figure 2-2—Operating Temperatures for a Typical 8-Hour Test Period

TABLE 2-4
Heat Balances from Test Data

<u>Test Period</u>	
<u>Heat inputs (10⁶ Btu/hr)</u>	<u>Jan. 11, 1983</u>
In coal feed	1.41
In air	0.00
As heat of sulphation of CaCO ₃	0.03
<hr/>	
Total Input	1.44
<hr/>	
<u>Heat outputs 10⁶ Btu/hr</u>	
In bed offtake - sensible	0.01
- potential	0.00
In elutriated solids - sensible	0.02
- potential	0.01
In exhaust gases - sensible	0.62
- potential	0.00
- latent	0.05
To cooling water - tube bank	0.64
- nozzles etc.	
Heat of calcination of dolomite	0.04
Bed temperature (°F) of dolomite	1650
<hr/>	
Total Output	1.39
<hr/>	
Closure for Heat Balance	0.97
<hr/>	

TABLE 2-5

Mass Balance From Test Data

	<u>Lb/hr</u>
Total air in flow	1331
Coal feed rate	114
Dolomite feed	63
Nitrogen in flow	<u>67</u>
Total Flow In	1575
Effluent gas out	1425
Bed offtake	25
Cyclone offtake	39
Nitrogen added to effluent	<u>67</u>
Total Flow Out	1556
Closure for Mass Balance	.99

Chemical Analysis of Bed and Cyclone Particulates

Samples of materials from the bed off-take and each of the three cyclones were collected on a weekly basis for the first 285 hours and for the 285-1085 test period. At the end of each period, the samples collected weekly were composited together and a representative sample of each was analyzed for chemical composition. Tables 2-6 and 2-7 list these analyses for each test period.

The concentrations of Na_2O and K_2O are shown to increase from the first to the third cyclone catches. An explanation for this may be that Na_2O and K_2O are generally tied-up with the mineral constituents in the coal ash. The first cyclone captures the larger particles elutriated from the bed which tend to be the spent dolomite particles (CaO , MgO). The second and third cyclones capture larger relative fractions of the coal ash (Al_2O_3 , SiO_2 and Fe_2O_3). Since Na_2O and K_2O are usually associated with the ash component, their concentration would tend to be higher in the second and third cyclone catches.

Alkali Probe Measurements

The alkali sampling system shown schematically in Figure 2-3, is used to determine the alkali content of the PFB effluent (parts per million by weight). The effluent gas is sampled just upstream of the entrance to the low-velocity test section. A 2-inch diameter mini cyclone and an Astro-quartz filter, contained in the oven, remove particulate from the gas sample. The oven maintains a temperature of 1400°F ; however, heat losses in the tubing (tubing is insulated but not heat traced) allow the gas to cool to between 1000° and 1200°F . The particulate-free gas sample then passes from the oven into two stainless steel U-tubes filled with stainless steel balls. These U-tubes sit in a cold water bath, maintained at 50°F , where the alkali vapors condense.

Both the dust collected in the mini cyclone/filter and the condensates from the U-tube are analyzed for water soluble alkali (Na and K), sulfate and chlorides to determine the total alkali available for corrosion of the gas turbine materials.

TABLE 2-6

Chemical Analysis⁽¹⁾ of Bed and Cyclone Particulate Catch
 LTMT - 285 Hours
 (Wt%)

	<u>Bed Material</u>	<u>First Cyclone</u>	<u>Second Cyclone</u>	<u>Third Cyclone</u>
C	2.24	4.26	0.98	0.76
CO ₂	4.22	13.14	1.17	0.57
CaO	23.39	35.60	23.49	22.34
MgO	16.53	25.13	12.82	14.11
SiO ₂	23.78	14.97	28.81	28.82
Al ₂ O ₃	9.91	6.46	15.89	16.13
Fe ₂ O ₃	4.30	5.86	8.54	7.72
K ₂ O	1.14	0.41	1.08	1.11
Na ₂ O	0.25	0.13	0.32	0.39
SO ₃ ⁻⁻⁻	19.01	10.98	8.04	8.23
Cl ⁻	----	.005	.004	.004

⁽¹⁾The data represents a single analysis of a composite sample collected during this period.

TABLE 2-7

Chemical Analysis⁽¹⁾ of Bed and Cyclone Particulate
 LTMT 285 - 1085 Hours
 (Wt%)

	<u>Bed Material</u>	<u>First Cyclone</u>	<u>Second Cyclone</u>	<u>Third Cyclone</u>
C	1.39	3.51	0.95	0.62
CO ₂	4.24	9.46	0.83	0.50
CaO	28.11	30.08	22.58	22.04
MgO	20.35	21.07	10.60	10.39
SiO ₂	12.37	9.89	20.51	22.64
Al ₂ O ₃	5.34	5.00	13.28	14.81
Fe ₂ O ₃	5.37	9.56	16.30	14.64
K ₂ O	0.53	0.31	0.79	0.91
Na ₂ O	0.15	0.14	0.32	0.63
SO ₃ ⁻⁻	24.19	16.49	13.34	12.23
Cl ⁻	0.24	0.19	0.15	0.20

(1) The data represents a single analysis of a composite sample collected during this period.

Figure 2-3 Dust/Alkali Sampling System

To date, water soluble alkali levels measured during the long-term materials test are 0.09-0.41 ppm Na, 0.51-3.21 ppm K and 9-10 ppm chlorides in the combustion products. (Those values represent the range of 3 alkali samples of 4 hours each in duration made during the first 1085 hours of testing.) These alkali levels are one to two orders of magnitude greater than the acceptable levels for industrial gas turbines; about 0.02 ppm alkali in the combustion products.

In-Bed Heat Exchanger - Erosion

Since operation of the pressurized fluidized bed combustor at Malta was initiated in April 1981, erosion/metal wastage has been observed on the water-cooled in-bed heat exchanger coil. Efforts have been made to understand this problem and minimize its effects on disrupting test operations.

Two triple helix design coils were utilized during the qualification testing and approximately 100 hours of the first corrosion validation test (11/81). This relatively dense pack of tubing was designed to break up bubbles and provide a uniform fluidizing action in the bed. Erosion of the two triple helix design coils was locally severe and highly unexpected and resulted in coil lives of only 150 and 400 hours. In both cases, failure occurred unexpectedly during periods of test operations and required emergency shutdown of the facility. (Details of these early heat exchanger erosion problems are summarized in the LMT Program Annual Report No. DOE/ET/15457-1110, Oct 1980-Sept 1981, and the Quarterly Progress Report No. DOE/ET/15457-1112, Oct-Dec 1981).

Following failure of the second heat exchanger coil, a single helix heat exchanger coil was designed, fabricated and installed in November 1981. This new heat exchanger, Figure 2-4, with a few modifications to its initial design, afforded a significant improvement in coil life and eliminated the severe localized erosion which caused failure of the earlier heat exchanger. Although wastage of the tube walls was not completely stopped, thinning occurred at a rate of about 3 to 6 mils per 100 hours of test operations off the tube diameter, Figure 2-4. Table 2-8 summarizes the erosion data for each of the test periods in which the first single helix coil was utilized.

Figure 2-4--Single Helix Heat Exchanger Design

TABLE 2-8

In-Bed Heat Exchanger Tube Thinning Data

	<u>DATES</u>	<u>HOURS</u>	<u>MILS/100 HRS</u>
	<u>First Single Helix</u>		
Corrosion Validation Test 1,2,3	{ 11/30/81 to 12/29/81	135	.0015/.0030 ⁽¹⁾
	{ 12/29/81 to 3/5/82	105	0/0
	{ 3/5/82 to 4/9/82	280	.0032/.0025
Shakedown Test	4/9/82 to 7/6/82	20	---
EPRI 2x200 Hour Tests	{ 7/6/82 to 8/11/82	221	.0062/.0048
	{ 8/11/82 to 9/13/82	200	No meas.
LTMT	3/7/83 to 3/14/83	110	.018*/0
	<u>Second Single Helix</u>		
	{ 11/1/82 to 12/20/82	400	.0033/.0015
	{ 1/1/82 to 1/31/82	378	.0055/.0032
LTMT	{ 1/31/82 to 2/7/83	104	.004/.004
	{ 2/7/83 to 2/21/83	203	.0045/.002
	{ 2/28/83 to 3/7/83	114	.008/.002
	<u>Third Single Helix</u>		
	3/14/83 to 3/21/83	104	.005/.000
	3/21/83 to 3/28/83	99	.003/.005

(1) Change in minimum dimension/change is maximum dimension.

The first single helix coil was removed from service in September 1982 after approximately 960 hours of operation. This coil was designated a "spare", and a new (second) single-helix coil was installed for the beginning of the long-term test in October 1982. The tube wall thinning rates measured on this coil are also listed in Table 2-8, with the erosion rate remaining in the same range of 3-6 mils per 100 hours as measured on the first single helix coil.

After 1200 hours service (actual coil life was over 1300 hours including startups and shutdown periods), distortion of the coil necessitated its replacement. A new coil was not immediately available, so the "spare" (first single helix coil) was reinstalled for a one-week period (3/7-3/14/82). Inspection of the spare coil after this one week service revealed several areas on the top coil had experienced severe erosion similar to that experienced on the triple helix coils. The reoccurrence of this severe erosion has been attributed to the distorted nature of the coil, and also to operation of the combustor with a deep bed (8 ft) compared to normal operations (6 ft). The deep bed resulted from problems with the ΔP gage and the actual height was not determined until shutdown (determined by the amount of bed material removed).

A third single helix coil is now in service and measurements of tube diameters indicate that thinning is continuing at the same rate as on the two previous coils: i.e., 3-6 mils/100 hours. Tube thinning data on this coil also is listed in Table 2-8.

The thinning of the heat exchanger tubing walls (0.065 inch thick) at a rate of 3 to 6 mils per 100 hours (diameter loss) should allow for a coil life of 1000-1500 hours. Detailed measurements and a complete coil inspection are being made after every 200-300 hours of operation.

Careful review of operating conditions during the various test periods indicate some degree of correlation between the tube thinning rate and several operational parameters including:

Bed height

Fluidizing velocity

Particle size

Pressure fluctuations in the bed

No detailed study of this problem is planned as it is not within the scope of the current program. Operating conditions and tube diametrical changes will continue to be monitored to minimize the potential for unexpected heat exchanger failures. Although heat exchanger tube wall thinning has not been eliminated, the problem has changed from a severe, unpredictable nature to a manageable condition having minimal effect on test operations.

SECTION 3

MATERIALS EVALUATION (TASKS 3, 5 AND 6)

A total of 1507 specimen exposure hours have now been completed under the long-term materials test with 1107 hours accumulated during the period from January-March 1983. As noted in the previous quarterly progress report (October-December 1982), material test specimens were removed from the LVTS for metallurgical analysis after 285 hours. These specimens included a complete set of materials at each of three metal temperatures: 1500°F (uncooled), 1300°F and 1100°F (cooled). After 1085 hours total test time, an identical group of specimens was removed for analysis of corrosion damage. This group had accumulated 800 hours exposure. In addition, a second set of uncooled pins exposed for the full 1085 hours also was removed for metallurgical analysis. Upon removal of each set of LVTS pins, an identical replacement set was installed which is used for longer exposure intervals. (The complete LVTS specimen exposure test plan was presented in the October-December quarterly report and is included as an Appendix to this report.)

Upon removal from testing, each specimen was lightly brushed to remove the loose ash deposits. These deposits were bulked together from all the specimens at each temperature and exposure interval, and then submitted for analyses of water soluble Na, K, Ca, Mg, SO_4^{--} and Cl^- .

After cleaning, three cross sections were cut from each LVTS pin specimen and metallographically prepared for microscopic examination. Corrosion penetration data reported herein represents the maximum penetration measured on any of these three cross sections.

The high-velocity test section also was opened and inspected for the first time after 1085 hours. All 24 airfoil specimens (4 each of 6 materials) and one erosion pin were removed for metallurgical evaluation of erosion/corrosion degradation. The test section was then reloaded with airfoils of 6 different materials for the next (approximately) 1000 hours of testing.

The HVTs airfoil specimens were contour traced to determine metal recession due to erosion followed by sectioning at the midplane for determination of corrosion penetration. The erosion/corrosion data represents the maximum values measured on any of the three impulse or one reaction airfoil tested.

The following sections summarize the details of the metallurgical analyses performed on the 285 and 800-hour LVTS cooled pin specimens, 285, 800 and 1085-hour uncooled LVTS pin specimens and the airfoils exposed 1085 hours

LVTS Corrosion Pins - 285, 800 and 1085-Hour Exposures

The inlet gas temperature to the low-velocity test section varies between 1490° and 1520°F with an exit temperature approximately 50°F lower. Fifteen different base alloys, coatings and claddings are being tested for corrosion resistance at this gas temperature. Thirteen different materials are being tested at nominal metal temperatures of 1300° and 1100°F to provide a simulation of cooled turbine components operating in a hot PFBC effluent.

Uncooled Pins - 1500°F

Table 3-1 summarizes the maximum corrosion penetration measurements on each material at 1500°F for the three exposure intervals, 285, 800 and 1085 hours. Corrosion of all materials in the uncooled condition was characterized by generally uniform attack around the circumference of the pins and also between the three cross sections. The nickel-base alloys, U 500, IN-730 and IN-939 experienced surface and subsurface oxidation with some areas of spherical sulfides beneath the oxidation attack. Figure 3-1 shows a photomicrograph of the typical corrosion on the nickel-base alloys. U-500 experienced the deepest penetration of the nickel-base materials, 3.4 mils in 1085 hours.

The cobalt base vane alloy, FSX-414, exhibited primarily internal oxidation attack of the carbide structure and the grain boundaries, Figure 3-2. Local areas of a relatively thick: i.e., 2 mils, duplex oxide surface scale also were observed.

TABLE 3-1
LTMT 1500°F Corrosion Data (Mils)

Material	Maximum Penetration (Mils)			Coating/Cladding Thickness (mils)
	285 Hrs	800 Hrs	1085 Hrs	
U-500	1.3	1.3	3.4	--
IN-738	1.0	1.9	2.3	--
IN-939	0.8	2.1	2.0	--
FSX-414	3.2	1.8	2.8	--
RT-19	1.8	1.8	1.3	2-3
RT-22	0.2	0.1	0.3	3-4
RT-44	N.D.*	N.D.	0.1	3-4
FeCrAlY (Clad)	N.D.	0.2	0.3	9-10
FeCrAlY (PVD)	N.D.	0.2	0.2	8-12
CoNiCrAlY (Clad)	0.5	1.0	1.0	9-10
CoCrAlY (23Cr-9Al)	N.D.	N.D.	N.D.	6-8
CoCrAlY (30Cr-5Al)	N.D.	0.6	0.2	6-8
CoCrAlY (29Cr-6Al)	0.8	1.2	1.4	8-9
Ceramic-TBC	Spalled	Spalled	Spalled	8-9
IN-671	N.T. ⁺	N.T.	6.9	9-10

*N.D. - None Detected
⁺N.D. - Not Tested

Figure 3-1 U-500 After 1085 Hours at 1500°F

Figure 3-2 FSX-414 After 1085 Hours at 1500°F

The "straight" aluminide (RT-19) coating had areas of internal attack to the interdiffusion zone of the IN-738 substrate in both the 285 and 800 hours specimens. The 1085-hour specimen showed the same type of attack, but no areas of complete coating penetration were observed, Figure 3-3. The platinum-aluminide (RT-22) and rhodium-aluminide (RT-44) coatings showed generally good resistance to hot corrosion on all the uncooled specimens, Figure 3-4a and b. Only isolated areas of surface oxidation up to 0.3 mil thick were observed on RT-22 after 1085 hours, and 0.1 mil of surface oxide on RT-44.

The physical vapor deposition FeCrAlY and two CoCrAlY (23Cr-9Al, 30Cr-5Al) coatings, and the FeCrAlY cladding, Figure 3-5, all exhibited no attack in 285 hours and significantly less than 1.0 mil of oxidation in the 800 and 1085 hour specimens. CoNiCrAlY cladding and plasma sprayed CoCrAlY (29Cr-6Al) coating had areas of corrosion to maximum depths of 1.0 and 1.4 mils respectively in 1085 hours. Corrosion of the Co-29Cr-6Al-1Y coating, Figure 3-6, and CoNiCrAlY cladding was characterized by internal oxidation with a narrow alloy depletion zone ahead of the oxidation attack. No sulfides were observed in any of the MCrAlY type materials.

The thermal barrier coating (MgO stabilized ZrO_2) experienced severe spallation of the ceramic outer-layer at each exposure interval. This coating was applied by a plasma spray process and the susceptibility to spallation may be due to the processing used to apply the coating and not necessarily due to interaction with the PFBC effluent. Currently, an alternate ceramic coating is being considered for testing.

IN-671 (Ni-48Cr) cladding is being evaluated in the long-term materials test as a "flag" material. IN-671 showed very poor performance in both the Exxon and CURL/Stal-Laval 1000-hour PFB tests, and is being exposed in the GE/Malta PFBC facility to compare its performance with the earlier tests. As indicated by the data in Table 3-1, IN-671 has experienced 6.9 mils of sulfidation attack in 1085 hours, Figure 3-7, easily the worst material tested at 1500°F.

Figure 3-3 RT-19 Aluminide Coating After 1085 Hours at 1500°F

Figure 3-4 (a) RT-22 Platinum - Aluminide Coating After 1085 Hours at 1500°F

Figure 3-4 (b) RT-44 Rhodium-Aluminide Coating After 1085 Hours at 1500°F

Figure 3-5 FeCrAlY Cladding After 1085 Hours at 1500°F

Figure 3-6 Plasma - Sprayed CoCrAlY After 1085 Hours at 1500°F

Figure 3-7 IN-671 Cladding After 1085 Hours at 1500°F

LVTIS Cooled Pins

Corrosion on the cooled pin specimens was generally non-uniformly distributed around the circumference and also along the length of the pins. Circumferentially, corrosion attack was more severe on the sides and downstream face than beneath the ash deposit on the upstream surface. Longitudinally, corrosion was predominant toward the cooling air inlet end of the 1300°F pins and toward the cooling air exit end of the 1100°F pins. Figure 3-8 shows a schematic of a cooled pin specimen instrumented with two thermocouples to measure temperature variations along the length of the pins. As the tabulated data beneath the drawing shows, the ΔT across the two-inch center portion of the pins was 110°-130°F. Thus, the maximum corrosion penetration on the 1300°F pins (Table 3-2) generally was observed on the end of the specimen having a metal temperature in the range of 1150°-1210°F, and on the 1100°F pins the worst attack (Table 3-3) occurred between about 1040° and 1110°F. (These observations regarding the areas of maximum corrosion should not be interpreted to mean corrosion was absent in other locations on the cooled pins, but only of a lesser magnitude.)

1300°F

Maximum corrosion penetration measurements for the 1300°F pin specimens are summarized in Table 3-2. The maximum penetration increased with the longer exposure time for all materials except Co-30Cr-5Al-0.4Y coating which was not attacked.

Corrosion of the bare nickel-base alloys, U-500, IN-738 and IN-939, was generally characterized by the presence of relatively thick surface oxide/scale layers with less internal oxide penetration than observed on the uncooled specimens of the same material. Many areas of the nickel-base alloys exhibited a region of densely populated spherical sulfides immediately below the surface oxide layer. Figure 3-9 shows typical photomicrographs of these corrosion morphologies after 800 hours.

The cobalt-base alloy, FSX-414 experienced internal oxidation at 1300°F similar to that observed at 1500°F, Figure 3-10. No sulfides were evident on either the 285 or 800-hour specimen.

Figure 3-8 Schematic of Cooled LVTS Corrosion Pins With Two Thermocouples (Measured Temperature Ranges Are Shown)

Figure 3-9 (a) U-500 After 800 Hours at 1300°F

Figure 3-9 (b) IN-738 After 800 Hours at 1300°F

Figure 3-9 (c) IN-939 After 800 Hours at 1300°F

Figure 3-10 FSX-414 After 800 Hours at 1300°F

TABLE 3-2
LTMT 1300°F Corrosion Data
(Mils)

<u>Materials</u>	<u>Maximum Penetration (Mils)</u>		<u>Coating/Cladding Thickness (mils)</u>
	<u>285 Hrs</u>	<u>800 Hrs</u>	
U-500	0.8	3.1	---
IN-738	0.8	1.0	---
IN-939	1.0	1.4	---
FSX-414	1.0	2.0	---
RT-19	N.T. ⁺	2.0	2-3
RT-22	1.2	N.T.	3-4
RT-44	N.D.*	0.2	3-4
FeCrAlY (Clad)	1.8	2.5	9-10
FeCrAlY (PVD)	N.D.	1.5	8-12
CoNiCrAlY (Clad)	1.8	3.5	9-10
CoCrAlY (23Cr-9Al)	N.D.	5.0	6-8
CoCrAlY (30Cr-5Al)	N.D.	N.D.	6-8
IN-671	---	2.8 (1085 Hrs)	9-10

⁺N.T. - Not Tested
*N.D. - None Detected

All of the protective coating/cladding systems evaluated experienced more corrosion attack on the 1300°F pin specimens than on the uncooled specimens except for the Co-30Cr-5Al-0.4Y coating and IN-671 cladding. The "straight" aluminide coating (RT-19) was completely penetrated (2 mils) to the inter-diffusion zone in only 285 hours, while the 800-hour specimen showed similar depth of attack with spallation of the oxide scale, Figure 3-11. Surprisingly, despite the complete coating penetration in 285 hours, there was no evidence of substrate attack on the 800-hour specimen. Apparently, the inter-diffusion layer acted as a protective barrier on the IN-738.

Figure 3-11 RT-19 Aluminide Coating After 800 Hours at 1300°F

The platinum-aluminide (RT-22) coating showed isolated areas of pitting attack in 285 hours at 1300°F, Figure 3-12. (Due to a mixup in specimen identification there was no RT-22 pin tested for the 800-hour exposure at 1300°F). The rhodium-aluminide (RT-44) coating was one of the most corrosion resistant materials at 1300°F. After 800 hours, there was only one isolated pit 0.2 mils deep. Figure 3-13 shows the typical appearance of RT-44 after 800 hours at 1300°F.

Figure 3-12 RT-22 Platinum-Aluminide Coating After 285 Hours at 1300°F

Figure 3-13 RT-44 Rhodium-Aluminide Coating After 800 Hours at 1300°F

Corrosion of the MCrAlY coatings and claddings was characterized by pitting attack at 1300°F. After 285 hours CoNiCrAlY and FeCrAlY claddings exhibited isolated areas of pitting attack as shown in Figure 3-14a and b. Eight hundred hours exposure at 1300°F caused localized pitting attack on all of the MCrAlY coatings and claddings except the Co-30Cr-5Al-0.04Y coating. Figures 3-15a and b show this pitting attack on FeCrAlY and Co-23Cr-9Al-0.4Y physical vapor deposition coatings.

A single IN-671 clad specimen was exposed for the full 1085 hours at 1300°F. Although the type of attack (sulfidation) was the same as on the uncooled pin, the maximum depth of penetration was only 2.8 mils, less than half of the attack on the uncooled pin.

1100°F

Corrosion penetration for most materials at 1100°F, Table 3-3, was generally less than at 1300°F after 800 hours. Exceptions to this were IN-738, CoNiCrAlY cladding and Co-30Cr-5Al-0.4Y coating. At 1100°F, the cobalt and

Figure 3-14 (a) CoNiCrAlY Cladding After 285 Hours at 1300°F

Figure 3-14 (b) FeCrAlY Cladding After 285 Hours at 1300°F

Figure 3-15 (a) FeCrAlY Coating After 800 Hours at 1300°F

Figure 3-15 (b) CoCrAlY(Co-23Cr-9Al-0.4Y) Coating After 800 Hours at 1300°F

TABLE 3-3
LTMT 1100°F Corrosion Data
(Mils)

<u>Material</u>	<u>Maximum Penetration (Mils)</u>		<u>Coating/Cladding Thickness (Mils)</u>
	<u>285 Hrs</u>	<u>800 Hrs</u>	
U-500	1.5	1.4	---
IN-738	0.5	2.8	---
IN-939	0.6	1.0	---
FSX-414	1.2	1.1	---
RT-19	2.0**	2.0**	2-3
RT-22	0.8	0.7	3-4
RT-44	0.8	N.D.	3-4
FeCrAlY (Clad)	N.D.*	0.2	9-10
FeCrAlY (PVD)	N.D.	1.2	8-12
CoNiCrAlY (Clad)	3.2	5.6	9-10
CoCrAlY (23Cr-9Al)	N.D.	0.2	6-8
		(1.0 @ 1085 Hrs)	
CoCrAlY (30Cr-5Al)	N.D.	0.5	6-8
IN-671	N.T. ⁺	1.9 (1085 Hrs)	9-10

**Coating Failure

*N.D. - None Detected

⁺N.T. - Not Tested

nickel-base alloys experienced less subsurface attack, and thicker surface oxide layers than at 1300°F. Figures 3-16 and 3-17 show photomicrographs of corrosion on FSX-414 and IN-738, respectively, after 800 hours.

The three aluminide coatings experienced similar corrosion behavior at 1100°F to that observed at 1300°F. The straight aluminide (RT-19) coating on IN-738 was completely penetrated after 285 and 800 hours, Figure 3-18. No substrate attack was observed. Platinum-aluminide (RT-22) coated IN-738 showed isolated areas of pitting attack similar to that shown in Figure 3-12 at 1300°F, while the rhodium aluminide coating (RT-44) continued to show good corrosion resistance. Only two small pits 0.8 mils deep were observed on the 285 hour RT-44 specimen and no corrosion on the 800-hour pin.

After 800 hours at 1100°F, CoNiCrAlY cladding continued to experience extensive pitting attack, Figure 3-19, as had occurred at 285 hours. The other MCrAlY overlay coatings showed generally good corrosion resistance in the 800-hour exposures, with only the FeCrAlY physical vapor deposition coating showing localized pitting attack, Figure 3-20.

A Co-23Cr-9Al-0.4Y coated pin which showed no visible attack after 285 hours at 1100°F, was reinstalled and exposed for the full 1085 hours completed to date. Corrosion attack up to 1.0 mil deep was observed after 1085 hours, Figure 3-21, indicating that although the MCrAlY coating showed good corrosion resistance in 800 hours, longer exposures may cause severe attack.

The IN-671 clad pin exposed for the full 1085 hours at 1100°F experienced 1.9 mils of internal oxidation attack with subsurface sulfide formation. This is the least amount of attack on IN-671 at any of the three metal temperatures being evaluated, but still shows the materials high susceptibility to degradation in the PFB environment.

Corrosion Rates

Table 3-4 lists corrosion rate estimates in mils/1000 hours for each material at each exposure temperature. This data was obtained by normalizing

Figure 3-16 FSX-414 After 800 Hours at 1100°F

Figure 3-17 IN-738 After 800 Hours at 1100°F

Figure 3-18 RT-19 Aluminide Coating After 800 Hours at 1100°F

Figure 3-19 CoNiCrAlY Cladding After 800 Hours at 1100°F

Figure 3-20 FeCrAlY Coating After 800 Hours at 1100°F

Figure 3-21 CoCrAlY (Co-23Cr-9Al-0.4Y) After 1085 Hours at 1100°F

TABLE 3-4
Normalized Corrosion Data Summary
(Mils/1000 Hours)

<u>Materials</u>	<u>LVTS</u> <u>1100°F**</u>	<u>LVTS</u> <u>1300°F**</u>	<u>LVTS</u> <u>1500°F***</u>
U-500	1.8	3.9	3.1
IN-738	3.5	1.3	2.1
IN-939	1.3	1.8	1.8
FSX-414	1.4	2.5	2.6
RT-19	2.5*	2.5*	1.2
RT-22	0.9	1.5	0.3
RT-44	<1.0	0.3	0.1
FeCrAlY (Clad)	0.3	3.1	0.3
FeCrAlY (PVD)	1.5	1.9	0.2
CoNiCrAlY (Clad)	7.0	4.4	0.9
CoCrAlY (23Cr-9Al)	0.9	6.3	<0.1
CoCrAlY (30Cr-5Al)	0.6	0.1	0.2
CoCrAlY (29Cr-6Al)	---	---	1.3
Ceramic	---	---	Spalled
IN-671	1.8	3.5	6.4

*Coating Failure

**800-hour data normalized to 1000 hours for cooled pins.

***1085-hour data normalized to 1000 hours for uncooled pins.

the longest exposure data at each temperature to 1000 hours; i.e., 800-hour corrosion penetration measurements for 1100° and 1300°F, and 1085-hour data at 1500°F. All materials were assumed to exhibit linear corrosion rate behavior.

The normalized corrosion rate data shown in Table 3-4 gives an indication of the relative behavior of all the materials as a function of temperature. It can also be used as an early indication of which materials probably would not provide an acceptable parts life of 25,000 hours for a PFB fired gas turbine. A corrosion rate of about 1 mil/1000 hours or less is generally considered an acceptable rate of attack.

Based on the criterion of 1 mil/1000-hour as representing an acceptable corrosion rate, the following assessments can be made:

- Unprotected nickel and cobalt-base blade and vane alloys would not provide satisfactory parts lives at any temperature between about 1050° and 1500°F.
- MCrAlY overlay coatings and precious-metal aluminide coatings show good resistance to corrosion above 1450°, but are susceptible to varying degrees of pitting attack between 1050°-1300°F.

It is now planned to discontinue testing several of the coating/cladding materials which have consistently shown very poor corrosion resistance in PFB testing to date, and substitute alternate materials not currently included in the test matrix. Those materials to be dropped include:

RT-19 (aluminide) coated IN-738
CoNiCrAlY (PFB-6) clad IN-738
IN-671 clad IN-738
Ceramic thermal barrier coating (MgO stabilized Zr_2O_3)

In addition, the FeCrAlY clad IN-738 specimens would be dropped, as cladding is no longer considered a technically viable process compared to low pressure plasma spray and physical vapor deposition processes for protecting gas turbine vanes and blades. New coating compositions specifically designed for low temperature corrosion applications will be selected for exposure in place of the above list of materials to be dropped.

Chemical Analyses of Ash Deposits from LVTS Pins

Table 3-5 lists the chemical analyses of the ash deposits collected from each group of pin specimens (group=all pins exposed at a given temperature) after 285 and 800 hours of exposure. The data shows that the deposits on the cooled pins are substantially richer in alkali, sodium plus potassium, than the uncooled pins.

High Velocity Test Section Airfoil - 1085 Hours

Six materials were exposed in the high-velocity test section during the first 1085 hours of testing to evaluate their resistance to erosion/corrosion degradation at temperatures of 1325°-1375°F. These materials included the following:

- U-500
- FSX-414
- RT-22 (platinum-aluminide)
- FeCrAlY - clad
- CoNiCrAlY - clad
- Co-30Cr-5Al-0.4Y - PVD

Figure 3-22 shows a schematic of the high-velocity test section. One airfoil of each material was installed in each of the four cascades (three impulse and one reaction). The gas velocity was 800-900 ft/s through the three impulse cascades and then increases from about 900 ft/s at the leading edge of the reaction airfoils to 1200-1400 ft/s at the trailing edge. A single 1/4 inch diameter pin specimen was located downstream of the reaction airfoils in the high-velocity, 1200-1400 ft/s, gas region. During the first 1085 hours, a platinum-aluminide (RT-22) coated pin was tested.

Dust loadings through the high-velocity test section varied from 31 to 77 ppm (range of 8 measurements) as measured by the Balston Filter dust sampling system. The mean particle size varied from 2.0 to 3.1 microns with between 2 and 7 percent of the population greater than 10 microns.

TABLE 3-5
Chemical Analysis of Ash Deposits from
LVTS Pins
(Wt% Water Soluble)

285 HOURS				
	Metal Temperature			
	1500°F	1300°F	1100°F	
Na	0.032	0.16	0.25	
K	0.15	1.8	2.9	
Ca	8.0	9.2	7.8	
Mg	0.23	0.48	1.6	
SO ₄	14	20	24	
Cl	0.004	0.002	0.002	
800 HOURS				
Na	0.058	0.17	0.13	
K	0.31	1.2	0.96	
Ca	9.1	8.1	8.6	
Mg	1.6	1.7	2.3	
SO ₄	32.5	35	29	
Cl	0.018	0.002	0.005

Figure: 3-22 Schematic of High Velocity Test Section

Characterization of Erosion/Corrosion/Deposition Effects

Prior to installation in the HVTS, the midplane of each airfoil specimen was profile contour traced at 20X magnification. Following exposure, each airfoil was soft bristle-brushed, acetone rinsed to remove all loose deposits, dried, and weighed. The parts were then profile traced, and measurements of metal recession and/or deposition were made by comparing the pre-test and post-test profiles. Figure 3-23 shows the general form of the erosion/deposition effects. Erosion is measured as the metal recession at the tip of the leading edge and along the pressure surface. The maximum metal recession generally occurred at the leading edge. The contour tracing technique was not used on clad specimens since the cladding is a uniform 0.010 in. thick and provides a good reference for post-test measurements.

Following post-test contour tracing, each specimen was sectioned at the midplane where the tracing had been made. One-half of each specimen was then mounted and metallographically prepared and evaluated. The microscopic evaluation was carried out to measure for corrosion attack (i.e., oxide or sulfide penetration), deposit thickness, and metal recession. Recession measurements on the base and coated specimens were compared with measurements from the contour tracings. Corrosion penetration was measured at the maximum depth of sulfide or oxide penetration below the existing metal surface; see Figure 3-24.

Deposition

Upon removal from the test section, each airfoil was covered with a loose powdery type reddish-brown deposit on the concave (pressure) surface. These deposits apparently occurred during disassembly of the pressure vessel, which caused ash buildup on the channel walls to become dislodged and fall onto the airfoils. These ash deposits were very powdery and could be removed by light brushing. The only adherent deposit on the concave surface was located immediately behind the leading edge, where a thin 1-2 mil layer of shiny, reddish-brown scale had formed. The remainder of the concave surface on both the impulse and reaction airfoils was deposit free and generally dark.

REACTION AIRFOIL

IMPULSE AIRFOIL

Figure 3-23 Schematic of Airfoils Showing Areas Where Erosion and Deposition Generally Occurred

Figure 3-24 Erosion and Corrosion Measurements

gray to black in color indicating an oxidized or corroded metal surface. The absence of deposits on the concave surface also suggested the particulate was erosive.

The convex (suction) surface of each airfoil was covered with a buildup of lightly adherent friable, reddish-brown ash deposit that required light scraping to remove it from the airfoil surface. Just behind the leading edge and along the platform regions on the trailing edge, a thin layer of the dense, adherent, reddish-brown deposit again was present.

Erosion

Each airfoil was contour traced prior to and after exposure in order to determine the metal loss due to erosion. Table 3-6 lists the measured amounts of leading edge and concave/pressure surface metal loss due to erosion/corrosion. The values listed represent the range of metal loss determined on the four airfoils of each material.

Leading edge metal loss due to erosion generally increased from the first to the third impulse airfoil cascade, reflecting a slight increase in velocity, from 800 ft/s at the first cascade to 850-900 ft/s at the third cascade. Metal loss at the leading edge of the reaction airfoil was slightly reduced compared to the third impulse cascade. Although the gas velocity increases slightly from the third to the fourth (reaction) cascade, the leading edge radius of the reaction airfoils is about 0.040 inch versus only 0.015 inch on the impulse airfoils. Airfoil leading edge radius significantly affects particle impaction efficiency; i.e., for a given dust loading and particle size distribution, as leading edge radius increases the number of particle impactions decreases.

Figure 3-25 shows the leading edge profile of the six specimens exposed in the third impulse cascade. U-500, FSX-414 and the RT-22 platinum-aluminide coating experienced maximums of 4-5 mils of metal loss at the leading edge and 2-3.3 mils along the pressure surface. The nominal 3 mil RT-22 coating was completely eroded away both at the leading edge (third cascade) and along the concave surface (third and fourth cascades).

(a) FSX-414

(b) U-500

(c) RT-22

(d) FeCrAlY

(e) CoNiCrAlY

(f) Co-30Cr5Al-0.4Y

Figure 3-25 HVT5 Airfoils After 1085 Hours at 1350°C

TABLE 3-6

HVTS - Metal Recession Data
1085 Hours, 1350°F

	Metal Loss (Mils)*		Coating/Cladding Thickness (Mils)
	Lead Edge	Pressure Surface	
U-500	0 - 4.0	1.0 - 2.0	---
FSX-414	1.0 - 5.0	1.0 - 3.0	---
RT-22	1.0 - 4.0	0.3 - 3.3	3-4
CoNiCrAlY (Clad)	1.4 - 3.4	1.3 - 1.7	9-10
Co-30Cr-5Al-0.4Y	0 - 2.0	0.9 - 2.0	6-8
FeCrAlY (Clad)	0.7 - 1.7	1.5 - 1.9	9-10
RT-22 (Erosion Pin)	3.8 (Coating)	+ 4.2 IN-738 = 8	3-4

*Range of measurements on 4 airfoils.

The MCrAlY-type coatings and cladding showed overall less metal loss than the base alloys or the aluminide coating. The Co-30Cr-5Al-0.4Y and Fe-25Cr-4Al-0.4Y coatings experienced 2.0 mils or less of erosion both at the leading edge and along the concave surface, while the CoNiCrAlY cladding showed maximum losses of 3.4 mils and 1.7 mils, respectively.

The most severe erosion occurred on the 0.25 inch diameter pin specimen located downstream of the reaction airfoils and exposed to gas velocities of 1200-1400 ft/s. The platinum-aluminide (RT-22) coating was completely penetrated at areas located on either side of the 12 o'clock position (facing the flow). The 3.8 mil coating was penetrated and 4.2 mils of IN-738 substrate lost to erosion. Figure 3-26 shows a cross-section of this erosion pin specimen.

Corrosion

Corrosion on the airfoil specimens in the high-velocity test section predominately occurred in areas where particle impaction/erosion also had occurred; i.e., at the tip of the leading edge and along the concave (pressure) surface. Little or no corrosion occurred on the convex (suction) surface of the airfoils. Table 3-7 lists the maximum corrosion penetration for each material after 1085 hours exposure.

U-500 exhibited sulfidation attack at the leading edge and along the pressure surface to a maximum depth of 1.6 mils, Figure 3-27. The sulfidation on the U-500 airfoils was similar in appearance to sulfidation attack on some areas of the U-500 LVTS pin specimens exposed at 1300°F. Corrosion on the convex (suction) surface consisted of only shallow oxidation.

Corrosion of FSX-414 was characterized by internal oxidation along the suction surface to a maximum depth of 3.0 mils. Along the pressure surface, oxide penetration was confined to patchy areas at the surface, but an extensive distribution of subsurface spherical sulfides was also present. FSX-414 exhibited primarily internal oxidation attack in the LVTS with little or no sulfide formation.

Figure 3-26 RT-22 Coated Erosion Pin After 1085 Hours at 1350°F and 1200-1400 Ft/s in HVTs

Figure 3-27 Corrosion on U-500 After 1085 Hours at 1350°F in HVTs

TABLE 3-7

HVTS - Corrosion Penetration Data
1085 Hours, 1350°F

<u>Material</u>	<u>Maximum Corrosion Penetration (Mils)*</u>	<u>Coating/Cladding Thickness (Mils)</u>
U-500	1.0-1.6	---
FSX-414	1.8-3.0	---
RT-22	N.D.-0.1 (3.0 Erosion Pin)	3-4
CoNiCrAlY (Clad)	0.3-0.8	9-10
Co-30Cr-5Al-0.4Y	1.4-1.7	6-8
FeCrAlY (Clad)	1.2-2.1	9-10

*Range of maximum penetrations measured on 3 airfoils.

The platinum-aluminide (RT-22) coated airfoils were virtually free of any corrosion save for one area beneath a deposit buildup on the suction surface of a single airfoil, maximum penetration was 0.1 mil. The RT-22 erosion pin located in the high-velocity region downstream of the reaction airfoils experienced extensive corrosion attack, 3.0 mils max., particularly on the downstream surface facing away from the gas flow, Figure 3-28. Corrosion was a frontal type of attack penetrating almost to the interdiffusion zone.

The MCrAlY coating and claddings exhibited significantly different corrosion characteristics in the high-velocity test section compared to the 1300°F pins in the LVTS. Corrosion on the airfoils was internal oxidation attack with a narrow alloy depletion zone ahead of the oxidation penetration. Figure 3-29a and b show typical attack on the Co-30Cr-5Al-0.4Y coating and FeCrAlY cladding. The CoCrAlY coating also showed isolated areas of oxidation attack on the suction surface, but FeCrAlY was corrosion free in this area. There was no evidence of pitting attack on either of these materials.

CoNiCrAlY cladding experienced only 0.8 mils of surface oxidation in this case on the suction surface. The extensive pitting attack which this material experienced in the LVTS at 1300°F and 1100°F was absent on the airfoils.

Analysis of the ash deposits collected from the suction surface of the airfoils and from the uncooled (1500°F) LVTS pins exposed 1085 hours are listed in Table 3-8. Chemically the airfoil deposits are similar to the uncooled pin deposits and do not show alkali enrichment as observed on the cooled pin deposits from the LVTS (Table 3-5). Thus, although the HVTS operates in the same temperature range as the 1300°F cooled pins, the airfoils are not cooled below the gas temperature and consequently, alkali vapor deposition is not enhanced. The absence of cooled metal surfaces and the difference in gas/particle dynamics between the LVTS and HVTS are most likely the primary factors contributing to the observed differences in corrosion of the various materials.

Figure 3-28 Corrosion on Downstream Surface of RT-22 Coated Erosion Pin After 1085 Hours at 1350°F and 1200-1400 Ft/s

Figure 3-29 (a) CoCrAlY Coating After 1085 Hours at 1350°F in HVTS

Figure 3-29 (b) FeCrAlY Cladding After 1085 Hours at 1350°F in HVTS

TABLE 3-8

Chemical Analysis of Ash Deposits
From HVTS Airfoils - 1085 Hours
(Wt% Water Soluble)

	<u>HVTS</u> <u>1350°F</u>	<u>LVTS Pins</u> <u>1500°F-1085 Hrs</u>
Na	0.033	0.036
K	0.066	0.18
Ca	7.7	10.7
Mg	0.47	0.55
SO ₄	19.5	28.0
Cl	0.012	.005

Erosion/Corrosion

An assessment of the relative performance of the six materials evaluated in the high-velocity test section can be made by comparing the maximum total affected metal measured on the airfoils. Table 3-9 lists the range of these measurements on the three airfoils (two impulse and one reaction) of each material which were destructively evaluated. The six materials fall into two general groups: those which show significant corrosion attack in the areas where erosion was taking place, FSX-414, U-500, Co-30Cr-5Al-0.4Y and FeCrAlY cladding; and those which experienced little or no corrosion attack with metal loss apparently related entirely to erosion, RT-22 coating and CoNiCrAlY cladding. (Categorizing RT-22 into the second group, erosion only, is based on the results from the airfoils only. To date, RT-22 is the only material tested as an erosion pin at 1200-1400 ft/s for 1000 hours, so that a valid comparison with other materials under similar conditions cannot be made at this time.)

In the first group the unprotected base alloys clearly showed more degradation, up to 5.0 mils, than the two MCrAlY materials. As noted in the discussion on corrosion, the base alloys exhibited sulfide penetration into the metal both at the leading edge and along the pressure surface where erosion occurred.

The CoCrAlY coating and FeCrAlY cladding alloys were substantially more resistant to degradation than the base alloys with only slightly more than 3.0 mils of total affected metal. Both of these materials showed internal oxidation attack on the leading edge and pressure surface with an alloy depletion zone evident on the CoCrAlY coating. This type of corrosion on the MCrAlY materials was not observed on the LVTS specimens.

The second group of materials, RT-22 coating and CoNiCrAlY cladding, appear generally resistant to corrosion in the high-velocity test section, but show susceptibility to erosion damage. The behavior of these two overlay coatings may be related to their inherent mechanical properties and less related to their high-temperature corrosion resistance. Aluminide coatings

such as RT-22 have ductile to brittle transition temperatures near 1600°F. Consequently, at 1350°F, the coating is brittle and may be subject to cracking and spallation under high-velocity particle impaction.

The CoNiCrAlY material, however, should have a significant level of ductility, as the cladding process involves forming sheet material around the airfoil at room temperature followed by diffusion bonding to the substrate. Therefore, the erosion/corrosion behavior of this material compared to the other two MCrAlY materials is not clearly understood. There was no evidence of the severe pitting attack which the material experienced in the LVTS, and its overall erosion/corrosion resistance is similar to the CoCrAlY coating and FeCrAlY cladding.

All of these materials derive their corrosion resistance and possibly some erosion resistance from the formation of protective chromium or aluminum oxide layers on the surface. The high-velocity impaction of particulate may be eroding this layer or disrupting its integrity so that it is no longer protective to the metal.

TABLE 3-9
HVTS Erosion/Corrosion Data

<u>Material</u>	<u>Maximum Total Affected Metal*(Mils)</u>	<u>Coating/Cladding Thickness (Mils)</u>
U-500	2.0 - 5.0	---
FSX-414	3.0 - 5.0	---
RT-22	1.0 - 4.0	3-4
CoNiCrAlY (Clad)	1.7 - 3.4	9-10
Co-30Cr-5Al-0.4Y	2.3 - 3.1	6-8
FeCrAlY (Clad)	1.9 - 3.2	9-10

*Range of maximum measurements on 3 airfoils.

SECTION 4

PROGRAM MANAGEMENT

During the January-March 1983 quarter, 1107 hours of long-term materials testing were completed. The Malta PFBC operated every week during this quarter except for one, which was a scheduled shutdown. Presently, it is the most reliable and lowest cost per hour PFBC operation in the world.

On March 22, 1983, there was a meeting of the LTMT Materials Advisory Committee at Malta, NY. The main purpose of the meeting was to review results from the first 1000 hours of materials exposure. There was agreement among the committee members that the test should continue with the following action items and modifications to be completed within a reasonable time:

- (1) At about 2500 hours of operation, the supply of Pfizer dolomite will be depleted. Pfizer is no longer marketing the material so that another source of dolomite must be identified. It was agreed that samples of dolomite from two suppliers recommended by Pfizer would be obtained and analyzed with the objective of selecting a new dolomite similar to the Pfizer material.
- (2) Drop the following materials: RT-19 coated IN-738 (cooled and uncooled), GE 2541 clad IN-738, PFB-6 clad IN-738 (cooled and uncooled), and the thermal barrier coating (MgO stabilized ZrO_2) on IN-738 (uncooled).
- (3) Select five (5) new candidate materials including: PVD CoCrAlY (30 Cr-10 Al). PVD NiCrAlY (40 Cr-10 Al) to be tested as cooled and uncooled pins and airfoils; an alternate ceramic coating, i.e., Y_2O_3 stabilized ZrO_2 as uncooled pins and airfoils; and a modified RT-22 coating, which is thicker and more ductile, to be tested as cooled and uncooled pins and airfoils. Also, a material of construction such as A-286 would be evaluated as a cooled pin specimen only.

In addition to the action items, several key suggestions were made as follows:

- (1) Additional metallurgical evaluation of specimens exhibiting low temperature corrosion, in order to better understand mechanisms (1100°F and 1300°F), should be considered in future program planning. (A proposal is in preparation to cover this work on the mechanism of low-temperature corrosion.)

- (2) S.J. Dapkunas pointed out that since the facility after a considerable investment of time and money, was now operating reliably at a fraction of the cost of earlier PFBC materials tests, serious consideration should be given to funding other work packages which could now "piggyback" on the test. For example, an in-bed oxygen probe to monitor oxygen partial pressures, in-bed heat exchanger materials testing, and testing of baghouse filter materials are a few examples of add-ons which could "piggyback" on the present test at low cost.

APPENDIX

MATERIALS EXPOSURE TEST PLAN

DOE Report Number DOE/ET/15457-1111, dated December 1981, presented the Long Term Materials Test Program - Materials Exposure Test Plan, including a summary of materials selection, materials exposure strategy and materials evaluation criteria. Since that report was issued, several changes have evolved in the test plan, primarily in the specimen exposure temperatures and specimen replacement schedules. These changes have come about as a result of the short-term corrosion validation tests which were conducted during the past year. These changes to the test plan are discussed below.

LVTS - Pin Temperatures

Exposure temperatures for the corrosion pins in the low velocity test section were planned to be 1600°F, 1450°F and 1300°F, with the lower temperatures achieved by air cooling hollow pin specimens. The 1600°F uncooled pin temperature was based on a bed operating temperature of 1750°F. However, a nominally 200-hour corrosion test at 1750°F (Ref. DOE/ET/15457-1428, July-Sept. 1982 Quarterly Report) caused excessively high corrosion rates on many materials both cooled (1300°F) and uncooled (1600°F). Consequently, the bed temperature for the long term test was selected at 1650°F. The reduction in bed operating temperature reduced the inlet temperature to the low velocity test section by approximately 90°F, i.e., 1510 20°F. Further, there exists a 60°F temperature drop through the test section so the temperature range of the uncooled pins is 1450°-1530°F.

During the corrosion validation tests, cooled pin specimens were exposed at a temperature of 1300 50°F. Many of the test materials also exhibited significant corrosion attack at these temperatures, as reported in the LTMT quarterly progress reports for April-June and July-September, 1982.

Based on the change in bed operating temperature and results of the corrosion validation tests, the exposure temperatures for the test materials

in the low velocity test section have been selected as 1500°F (uncooled), 1300 50°F. Temperature control on the cooled pin specimens is achieved using thermocouples implanted in each cooled pin and airflow control valves as described in Section 2.

LVTs - Pin Replacement Schedule

Table A-1 presents the materials test matrix for the low velocity test section. Figure A-1 shows the replacement schedule for each of the 13 materials to be tested as cooled pins. There are a total of 60 cooled pin positions in the LVTs. Four positions are assigned to each material, two for 1300°F and two for 1100°F testing. The total number of cooled pins required to complete this matrix is 12 per material, if it is assumed that a material will last for the duration of the long term test.

Testing the 13 materials presented in the original test plan, using four positions each, will utilize 52 of the 60 available positions. The remaining eight locations are being used as follows: 1) for testing specimens with two thermocouples for measuring the T across the length of the specimen in the gas flow (two specimens, one at each temperature); 2) testing IN-671 clad specimens (two specimens, one at each temperature); 3) testing two new plasma sprayed coatings designed for low temperature hot corrosion applications. (Both materials are in test at 1300°F, only one at 1100°F.); and 4) one position has been rendered unserviceable due to damage during rig overhauling last year.

Seventeen materials (Table A-1) are being tested as uncooled materials. Six positions are utilized for each material as shown in Figure A-2, for a total of 102 of the possible 120 positions. The remaining 18 positions are being used for: 1) thermocouple installations to monitor the gas temperature throughout the test section; 2) exposing RA333 test pins (material used for fabricating the LVTs); and 3) limited testing of IN-671 clad specimens. IN-671 has shown a high susceptibility to degradation in previous PFBC testing

TABLE A-1
LVTS MATERIALS TEST MATRIX

BASE ALLOY	PROTECTIVE SYSTEM	IDENTIFICATION	PROCESS	LOW VELOCITY TEST SECTION		
				UNCOOLED 1500°F	COOLED 1300°F	1100°F
IN-738	FeCrAlY	GE2541	Clad	X	X	X
IN-738	FeCrAlY	ATD-17	Coating	X	X	X
IN-738	CoCrAlY	PFB-6	Clad	X	X	X
IN-783	CoCrAlY	ATD-14	Coating	X	X	X
IN-783	CoCrAlY	ATD-2	Coating	X	X	X
IN-738	CoCrAlY	PS-1	Plasma Spray	X	N	N
IN-738	Aluminide+Pt	RT-22	Coating	X	X	X
IN-738	Aluminide	RT-19	Coating	X	X	X
IN-738	Thermal Barrier	PS-TBC	Plasma Spray	X	N	N
IN-738	Ni-48Cr	IN-671	Clad	S	S	S
IN-738	None	-----	-----	X	X	X
FSX-414	CoCrAlY	RT-44	Coating	X	X	X
FSX-414	CoCrAlY	ATD-2	Coating	X	X	X
FSX-414	CoCrAlY	ATD-14	Coating	X	N	N
FSX-414	FeCrAlY	ATD-17	Coating	X	N	N
FSX-414	None	-	-	X	X	X
U-500	None	-	-	X	X	X
IN-939	None	-	-	X	X	X

Y - Material to be Tested
N - Not Tested
S - IN-671 will be Tested Selectively

at Exxon and CURL, and is being used for comparison purposes only. Six positions are currently vacant but will be used for testing additional materials as necessary.

The uncooled pins are arranged in the low velocity test section so that one pin of each material is at approximately the same elevation, and therefore, the same temperature.

High Velocity Test Section - Airfoil Specimens

Twelve different base alloys, coatings and claddings have been selected for evaluation of their erosion/corrosion resistance in the high velocity test section, Table A-2. These materials are being screened in two 1000-hour test segments, six materials at a time. (A third 1000-hour test may be run if several new or alternate materials are selected for evaluation.) Following these 1000-hour tests, the three or four materials showing the best erosion/corrosion resistance will be tested for the duration of the long term test. During the long term exposure, one airfoil of each material in that test will be removed and replaced with a like specimen at some intermediate time, approximately 2500 hours. Additional specimen substitutions will be performed based on results of this interim inspection.

TABLE A-2

HVTS MATERIALS TEST MATRIX

BASE ALLOY	PROTECTIVE SYSTEM	IDENTIFICATION	PROCESS	HIGH VELOCITY TEST SECTION	
				- 1000-HR TEST	LONG TERM TEST
IN-738	FeCrAlY	GE2541	Clad	1	TBD
IN-738	FeCrAlY	ATD-17	Coating	2	
IN-738	CoCrAlY	PFB-6	Clad	1	
IN-738	CoCrAlY	ATD-14	Coating	1	
IN-738	CoCrAlY	ATD-2	Coating	2	
IN-738	CoCrAlY	PS-1	Plasma Spray	N	N
IN-738	Aluminide+Pt	RT-22	Coating	1	TBD
IN-738	Aluminide	RT-19	Coating	2	TBD
IN-738	Thermal Barrier	PS-TBC	Plasma Spray	N	N
IN-738	Ni-48Cr	IN-671	Clad	N	N
IN-783	None	--	--	2	TBD
FSX-414	Aluminide + Rh	RT-44	Coating	2	TBD
FSX-414	CoCrAlY	ATD-2	Coating	N	N
FSX-414	CoCrAlY	ATD-14	Coating	N	N
FSX-414	FeCrAlY	ATD-17	Coating	N	N
FSX-414	None	--	--	1	TBD
U-500	None	--	--	1	
IN-939	None	--	--	2	

(1) First 1000-hr screening test
 (2) Second 1000-hr screening test
 (N) Not tested

TBD - To be determined based on results of 1000-hr screening tests.