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### LONG TERM MATERIALS TEST PROGRAM

Quarterly Report, April-June 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

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### LONG TERM MATERIALS TEST PROGRAM Quarterly Report

April Through June 1983

Program Manager Russell L. McCarron

Prepared for U.S.. DEPARTMENT OF ENERGY

Contract DE-AC21-79ET15457

GENERAL ELECTRIC COMPANY ADVANCED ENERGY PROGRAMS DEPARTMENT SCHENECTADY, NEW YORK

# GENERAL 🐲 ELECTRIC

#### ABSTRACT

Corrosion and erosion/corrosion testing of gas turbine materials in the effluent from a pressurized fluidized bed coal combustor continues under the Long Term Materials Test program. Two 1000-hour erosion/corrosion "screening tests" of twelve candidate gas turbine vane and blade base alloys and a variety of protective coating systems have been completed. Test conditions included 1350°F, 800-900 ft/s and particulate loadings of 30-90 ppm. Erosion/ corrosion degradation rates of 1-4 mils/1000 hours were observed with corrosion predominant in areas of particle impaction. FeCrAlY, CoCrAlY and rhodium aluminide coatings show significantly better resistance to degradation than unprotected base alloys, aluminide or platinum-aluminide diffusion coatings.

#### SUMMARY

The Malta pressurized fluidized bed coal combustion facility continues to operate at the following conditions: 1650°F bed temperature, 10 atmospheres pressure, 2.4 to 2.7 ft/s fluidizing velocity. Pittsburgh No. 8 coal (5% sulfur, 0.15% chlorine) is being burned with a low alkali (0.1 wt% sodium plus potassium) dolomite sulfur sorbent. A total of 2687 hours of testing have been completed.

Corrosion of gas turbine vane and blade base alloys and various protective coating systems is being evaluated at nominal metal temperatures of 1500°, 1300° and 1100°F in a 1500°F gas stream following three stages of cyclone cleanup. Erosion/corrosion degradation of these materials also is being evaluated in a high velocity test section at 800-1400 ft/s, 1350°F and dust loadings of 30-90 ppm.

Significant findings from the testing to date include:

- Unprotected nickel and cobalt-base blade and vane alloys are susceptible to hot corrosion at all metal temperatures.
- 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 several instances, corrosion rates increase with decreasing metal temperatures.
- o 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 (0.1 mil/1000 hours), but are susceptible to varying degrees of pitting attack at 1100° to 1300°F.
- o Erosion/corrosion degradation rates range from 1-4 mils/1000 hours for coatings and claddings 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.
- o Metal recession rates of 8 mils/1000 hours on a platinum-aluminide coated IN-738 pin and 2 mils/1000 hours on a Co-23Cr-10Al-0.4Y coated pin have been observed at 1200-1400 ft/s.

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### ACKNOWLEDGEMENTS

The individuals at the General Electric Company with major responsibility for this program are as follows:

| Program Manager                    | - R McCarron  |
|------------------------------------|---------------|
| 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.

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#### SECTION 2

### TEST OPERATIONS (TASK 2)

The PFBC test facility at Malta operated as scheduled during the April-June quarter accumulating 1180 test hours. A total of 2687 hours have been completed under the Long Term Materials Test program. The only change to the nominal operating conditions is that dolomite is now being supplied by National Lime and Stone Company, Findlay, Ohio instead of Pfizer, as Pfizer has phased out their dolomite operations.

Two significant operational problems were encountered during this quarter: The in-bed heat exchanger coil continues to exhibit degradation by erosion and/or corrosion with local repairs required at intervals of 300 to 650 hours, and cracking developed on the 304 stainless steel adapter tubes on the 1100°F cooled pin specimens in the low-velocity test section. Detailed discussions of both these problems are presented 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  $1650^{\circ}F$ , 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.

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 with dust loadings varying between 30 and 90 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 have averaged 4.99 wt% sulfur and 0.15 wt% chlorine.

### LTMT Operating Conditions

Combustor Coal Pittsburgh No. 8 Dolomite Pfizer, National Lime and Stone Co.-No. 11 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       | 1500°-1520°F                                       |
| Outlet Temp      | 1440°-1480°F                                       |
| Cooled Pin Temps | 1300°F <sup>+</sup> 50°F, 1100°F <sup>+</sup> 50°F |

High-Velocity Test Section

| Temp   | 1325°–1375°F                 |
|--|------------------------------|
| Dust Loading                                 | 30-90 ppm                    |
| Mean Particle Size (Microns) <sup>(1)</sup>  | 1.9-3.1                      |
| Percent Particles >10 Microns <sup>(1)</sup> | 2–10                         |
| Gas Velocity                                 | 800-900 ft/s (Airfoils)      |
|  | 1300-1400 ft/s (Erosion Pin) |

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(1) Range of 15 analyses

### Coal Analysis Pittsburgh No. 8 (Wt%)

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|   | 400-268                       |                   |
|---|-------------------------------|-------------------|
| <u>Proximate Analysis (As Received)</u> | Range of<br><u>6 Analyses</u> | Avg. of           |
| Moisture                                |                               | <u>6 Analyses</u> |
| · · · · · · · · · · · · · · · · · · ·   | 2.18-4.70                     | 3.43              |
| Ash                                     | 10.30-16.69                   | 14.27             |
| Volatile                                | 33.40-37.60                   | 35.16             |
| Fixed Carbon                            | 45.21-49.92                   | 47.13             |
| Btu/lb                                  | 11521-12497                   | 12188             |
| <u>Ultimate Analysis (As-Received)</u>  |                               |                   |
| Moisture                                | 2.18-4.70                     | 3.43              |
| . Carbon                                | 62.44-71.15                   | 66.25             |
| Hydrogen                                | 4.48-4.86                     | 4.68              |
| Nitrogen                                | 1.14-1.36                     | 1.25              |
| Chlorine                                | 0.11-0.21                     | 0.15              |
| Sulfur                                  | 4.25-5.54                     | 4.99              |
| Ash                                     | 10.30-16.69                   | 14.27             |
| Oxygen                                  | 4.14-5.73                     | 4.97              |
| <u>Ash Analysis</u>                     |                               |                   |
| sio <sub>2</sub>                        | 32.52-37.47                   | 34.82             |
| A1203                                   | 18.38-19.77                   | 19.17             |
| Fe <sub>2</sub> 0 <sub>3</sub>          | 37.43-41.87                   | 39.65             |
| Tio                                     | 0.81-0.90                     | 0.85              |
| CaO                                     | 0.66-1.67                     | 1.09              |
| MgO                                     | 0.54-0.76                     | 0.63              |
| K <sub>2</sub> 0                        | 1.19-1.72                     | 1.47              |
| Na <sub>2</sub> 0                       | 0.19-0.41                     | 0.34              |
| s0 <sub>3</sub>                         | 0.91-1.72                     | 1.20              |

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|                        | <u>Pfizer</u> | National Lime &<br> | National Lime & |
|------------------------|---------------|---------------------|-----------------|
| CO <sub>2</sub> (loss) | 43.68         | 46.76               | 48.34           |
| CaŪ                    | 31.81         | 28.06               | 29.47           |
| MgO                    | 22.83         | 19.82               | 21.03           |
| SiO <sub>2</sub>       | 1.52          | 2.36                | 0.56            |
| A1203                  | 0.57          | 0.44                | 0.17            |
| Fe U 3                 | 0.22          | 0.14                | 0.21            |
| K <sub>2</sub> 0       | Ô.0           | 0.04                | 0.0             |
| Na20                   | 0.15          | 0.17                | 0.10            |
| SO3                    | 0.67          | 0.14                | 0.10            |
| ເາັ                    | 0.24          | 0.09                | 0.09            |

### Chemical Analyses of Dolomites (Wt%)

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 98 percent based on a nominal 5 percent sulfur coal.

Pfizer dolomite was used for the first 1923 hours of the long term test. Pfizer discontinued mining and supplying dolomite in January 1983. Several alternate suppliers were contacted and samples of their dolomites analyzed for chemical composition and size distribution. National Lime and Stone Company's dolomite has been selected for use throughout the remainder of the Long Term Materials Test. This dolomite is chemically similar to the Pfizer material, Table 2-3, and is available in four standard size distributions. Two of these size distributions are listed in Table 2-4 along with the Pfizer size distribution.

Initially, the No. 11 size dolomite was selected to replace the Pfizer material. Although the No. 11 material contained a larger fraction of particles in the 20 mesh range than Pfizer, it contained a relatively small fraction in the 100 and -100 mesh size range. Based on earlier experience during the corrosion validation tests, dolomite with large fractions of these fine particles caused operational problems with plugging of the coal feed system.

| Sieve<br>Mesh<br><u>No.</u> | <u>Pfizer</u> | National Lime | National Lime |
|-----------------------------|---------------|---------------|---------------|
| 10                          | 6.7           | 1.7           |               |
| 20                          | 25.3          | 49.7          | 3.2           |
| 30                          | 25.3          | 14.1          | 16.0          |
| 40                          | 32.7          | 12.0          | 37.5          |
| 50                          | 26.4          | .7.5          | 18.5          |
| 100                         | 7.5           | 10.0          | 18.4          |
| -100                        | 1.2           | 5.1           | 6.4           |

#### Dolomite Size Distributions

After three weeks (312 hours) of test operations using the No. 11 dolomite, the in-bed heat exchanger coil was observed to be experiencing severe erosion of the tube walls. (See discussion later in this section.) The occurrence of the severe erosion was directly attributed to the use of the relatively coarse dolomite. Subsequent test operations utilized National Lime Company's No. 20 material. A supply of the No. 20 dolomite had been ordered as a backup source after the supplier improved their processing to limit the amount of fines (<100 mesh) to 7 percent maximum. Inspection of the heat exchanger coil after two weeks of operation using the No. 20 dolomite, revealed no evidence of severe erosion, and plugging problems in the coal feed system have proven to be minimal. In addition, the sulfur capture has improved significantly with the change in dolomite.

#### Chemical Analysis of Bed and Cyclone Particulates

Samples of material from the bed off-take and each of the three cyclones were bulked for the four weeks of test operations in April and again for the three weeks of testing in May. The April test period, 1507-1923 hrs, was the last portion of the long-term test carried out utilizing Pfizer dolomite, while the May test period, 1923-2235 hrs, represents the testing conducted using National Lime and Stone Company's No. 11 dolomite. Tables 2-5 and 2-6 list the particulate analyses for each of these test periods. The various fractions are basically similar in composition, with one notable difference

|                   | Bed<br><u>Material</u> | First<br><u>Cyclone</u> | Second<br><u>Cyclone</u> | Third<br><u>Cyclone</u> |
|-------------------|------------------------|-------------------------|--------------------------|-------------------------|
| С                 | 1.58                   | 3.31                    | 0.72                     | 0.08                    |
| CO <sub>2</sub>   | 5.82                   | 9.83                    | 0.94                     | 0.52                    |
| CaŪ               | 29.42                  | 27.96                   | 18.80                    | 16.64                   |
| Mg0               | 20.48                  | 18.77                   | 8.54                     | 7.53                    |
| SiO <sub>2</sub>  | 8.14                   | 7.87                    | 19.86                    | 20.27                   |
| A1,03             | 8.78                   | 4.96                    | 13.91                    | 11.87                   |
| Fe203             | 4.52                   | 13.53                   | 20.66                    | 20.19                   |
| к,0               | 0.24                   | 0.17                    | 0.76                     | 0.74                    |
| Na <sub>2</sub> 0 | 0.30                   | 0.18                    | 0.35                     | 0.61                    |
| so3               | 26.22                  | 14.88                   | 11.38                    | 9.62                    |
| C1                | 0.06                   | 0.11                    | 0.17                     | 0.15                    |

### Chemical Analyses of Bed and Cyclone Particulate (LTMT - 4/83, Pfizer Dolomite) (Wt%)

### TABLE 2-6

### Chemical Analyses of Bed and Cyclone Particulate (LTMT - 5/83, National Lime #11 Dolomitc) (Wt%)

|                  | Bed<br><u>Material</u> | First<br><u>Cyclone</u> | Second<br>Cyclone | Third<br><u>Cyclone</u> |
|------------------|------------------------|-------------------------|-------------------|-------------------------|
| C                | 1.13                   | 1.58                    | 1.28              | 0.55                    |
| <sup>C0</sup> 2  | 3.85                   | 4.04                    | 0.58              | 0.33                    |
| CaŪ              | 31.27                  | 28.45                   | 18.14             | 15.53                   |
| MgO              | 21.73                  | 18.70                   | 11.10             | 9.18                    |
| SiO <sub>2</sub> | 8.74                   | 9.50                    | 18.85             | 19.10                   |
| A1203            | 4.49                   | 5.63                    | 12.08             | 13.13                   |
| Fe203            | 5.73                   | 11.87                   | 13.41             | 12.87                   |
| к <sub>2</sub> 0 | 0.38                   | 0.24                    | 0.92              | 1.00                    |
| Na20             | 0.18                   | 0.28                    | 0.54              | 0.59                    |
| s0 <sub>3</sub>  | 20.83                  | 19.11                   | 18.70             | 12.48                   |
| C1               | 0.20                   | 0.17                    | 0.14              | 0.18                    |

being the reduced level of  $CO_2$  carryover to the first cyclone when the National Lime dolomite was used. This reduction in  $CO_2$  carryover indicates the new dolomite is more reactive than the Pfizer, consistent with the fact that SO<sub>2</sub> levels in the flue gas have also been reduced.

#### Alkali Probe Measurements

Water soluble alkali levels measured during the Long Term Materials Test are 0.08-0.41 ppm Na, 0.17-3.21 ppm K, and 9-10 ppm chlorides in the combustion products. 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

Metal wastage of the stainless steel, water-cooled in-bed heat exchanger coil was discussed in detail in the January-March 1983 quarterly report. Essentially, the single-helix coil design currently used in the PFBC facility at Malta experiences diametrical thinning of the 310 SS tubing at a rate of 3-6 mils/100 hours of service. Some degree of correlation appears to exist between tube thinning rate and one or more operational parameters: bed height, fluidizing velocity, bed particle size, and bed pressure fluctuations.

During the April-June quarter, the in-bed heat exchanger coil was replaced twice. The first of these coils was installed in March following 1300 hours of successful service using the previous coil. The coil installed in March was again experiencing tube diameter thinning rates of 3-6 mils/100 hours with an isolated area near the bottom of the coil showing 7-8 mils/100 hours of metal loss. Even at the higher diametrical thinning rate, the 0.065-inch thick wall tubing was expected to last a minimum of 1000 hours.

After approximately 620 hours of service, failure of this coil occurred by complete fracture of the inlet tube just below the Swagelok fitting that connects the 310 SS coil to the Inconel 625 riser tube, Figure 2-1. Figures 2-2 (a & b) are scanning electron micrographs of the fractured tube wall. Approximately one-half of the wall thickness toward the OD, Figure 2-2a, is covered with a thick corrosion or oxide layer with the remaining area extending to the ID covered with fatigue striations, Figure 2-2b. The fatigue striations indicate propagation from the OD to the ID of the tube wall. It is unclear, however, whether the failure initiated on the outer surface by pitting or

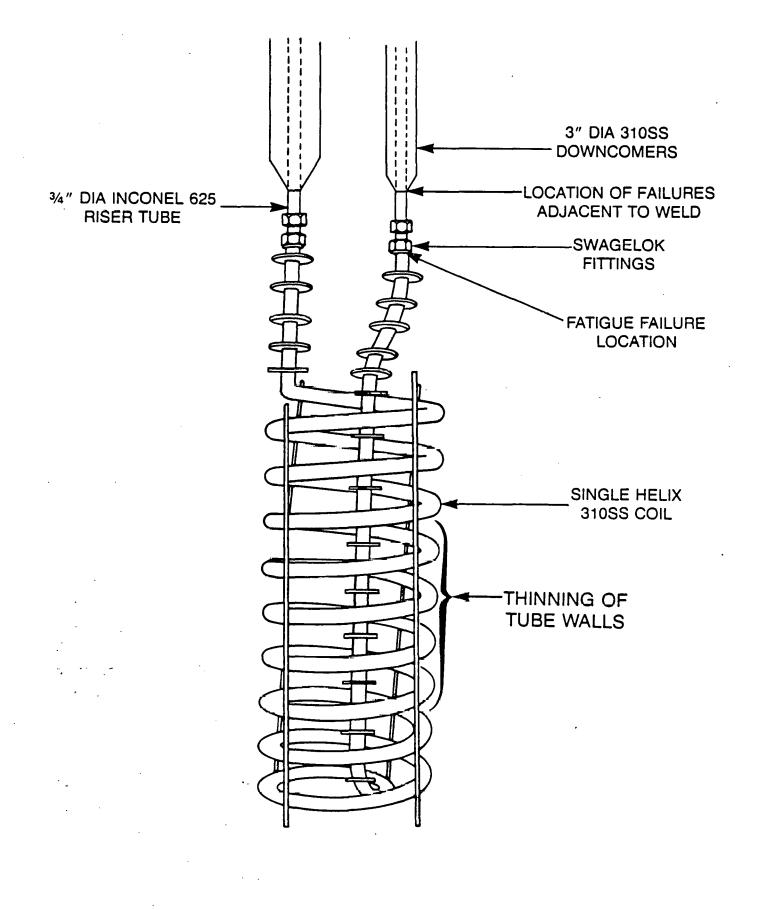


Figure 2-1. IN-BED Heat Exchanger Coil Assembly

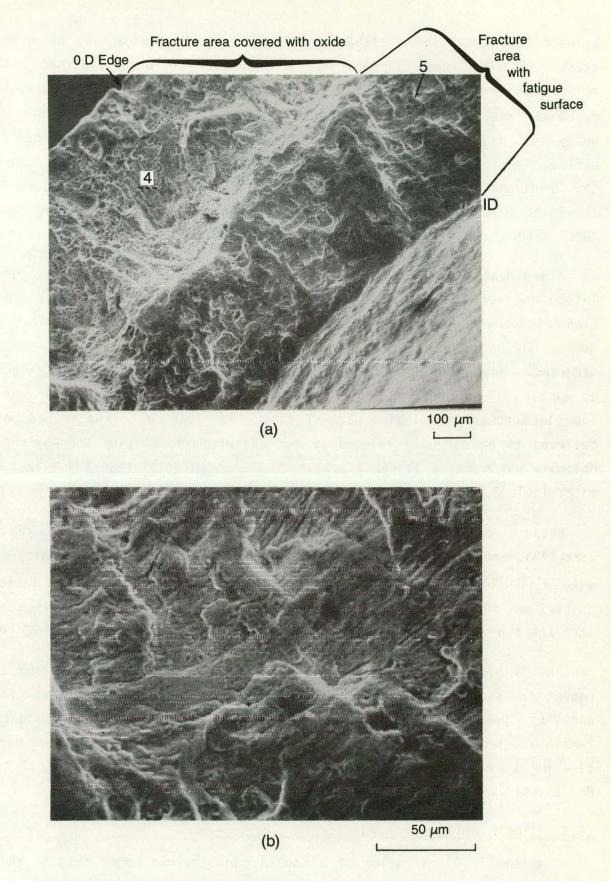


Figure 2-2. Fracture Surface of 310SS Tubing a) Corrosion Towards OD and Fatigue ID b) High Magnification of Fatigue Striations

crevice type corrosion, followed by fatigue crack propagation, or a fatigue crack initiated whose surface was subsequently corroded/oxidized. Pitting attack has previously been observed at various locations on the in-bed heat exchanger assembly. (Fractures similar to that described above have previously occurred on the heat exchanger heat exchanger assembly, but these earlier fractures occurred in the downcomer assembly at the weld connecting the 3/4-inch diameter riser tube to the 3-inch downcomer, see Figure 2-1.) The inlet tube was replaced and the coil successfully returned to service at a later date.

A new heat exchanger coil was installed at the beginning of May. Installation and service of this coil coincided with the use of National Lime and Stone Company's No. 11 dolomite. Inspection of the coil following three weeks, 312 hours of testing, revealed that severe erosion of the coil had occurred. Measurements indicated that the tube diameter had been reduced by as much as 40-45 mils, 12-15 mils/100-hours, with most of the metal loss taking place toward the inside part of the coil. Erosion of the in-bed coil is believed to be directly related to the use of National Lime Company's No. 11 dolomite which has a larger fraction of coarse material than the Pfizer dolomite, Table 2-4.

Because of the severe erosion, the in-bed coil was again replaced, June 1983, and test operations commenced using the No. 20 dolomite which has a size distribution much closer to the Pfizer material as shown in Table 2-4. Inspection of the latest coil after approximately 225 hours service showed that the tube thinning rate had again decreased to less than 6 mils/100 hours.

The in-bed heat exchanger coil continues to be the most problematic component in the PFBC facility at Malta. Multiple failure modes make life predictions speculative and monitoring of coil appearance and dimensions will remain as standard operating practice. It appears clear that the bed particle size is a critical parameter affecting erosion behavior of the coil in the Malta test facility.

#### LVTS - 1100°F Pin Specimen Adapter Tube Failure

A schematic illustration of a cooled pin specimen under test in the lowvelocity test section is shown in Figure 2-3. Cooling air enters each pin through 3-ft long, 1/4-in. diameter stainless steel tubes connected to a mani-

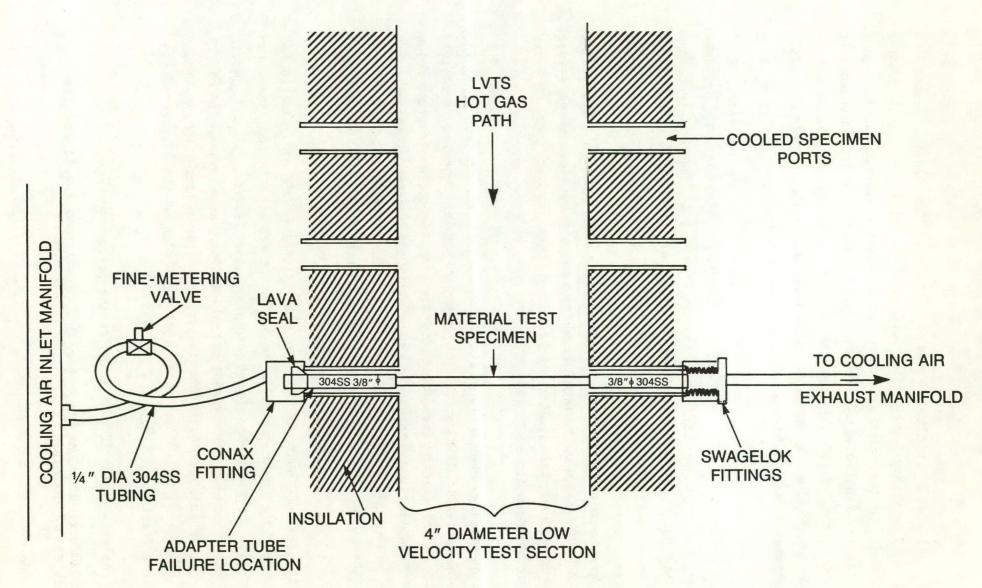


Figure 2-3. Schematic of LVTS Cooled Pin Specimen Configuration

fold which distributes air to all 60 cooled pins. The 1/4-in. diameter x 6-in. long test specimens are fitted with 3/8-in. diameter 304 SS adapter tubes as shown. On the inlet side, the 304 SS adapter tube passes through a Conax-Lava seal fitting to which the cooling air inlet tube is connected. On the exit side, the 3/8-in. diameter stainless adapter tube is fitted with a Swagelok fitting welded to its end. The Swagelok fitting threads directly into the specimen ports in the low-velocity test section.

At the 1500-hour mark of the long-term test, a failure of the 304 SS adapter tube occurred on the inlet side of an 1100°F cooled pin. The specimen which failed had been in test since the outset of the long-term test, and therefore, had seen the full 1500 hours of exposure. Failure of the specimen was noted by a sharp rise in the temperature of the stainless steel tubing (normally near room temperature) connecting the pin to the cooling air manifold, and an accumulation of liquid condensate in the bottom of the cooling air manifold. The pin was isolated from the manifold by closing the needle valve which regulates the cooling airflow, and then removed from the test section after shutdown at week's end.

Inspection of the adapter tube after removal of the specimens from the test section, revealed that the 3/8-in. diameter adapter tube was completely plugged with a hard cement-like deposit on the pin side of the fracture while the piece of adapter tube leading to the cooling air manifold remained relatively free of deposit. The fracture was located approximately 3 1/4-in. from the end of the adapter tube, putting its location 1-in. inside the Lava seal connection.

During the next 600 hours of testing, 8 additional 1100°F pins exposed for periods of 1800 to 2100 hours failed in a similar manner. In addition, four 1100°F pins with only 600-1000 hours exposure also failed. In all cases, failure exhibited similar characteristics: 1) 1100°F cooled pin specimens, 2) sharp rise in the inlet or exit tube temperature, 3) accumulation of liquid condensate in the cooling air manifold, and 4) plugging of the adapter tube on one side of the fracture.

Failure analyses of the 12 pins revealed the following:

1. All failures occurred 3-1/4 to 3-1/2 inches from the tip of the inlet end of the pin,

- 2. Multiple cracks were evident adjacent to the primary fracture location,
- The liquid condensate was dark green in color and had a pH of 2-2.5.
- Solid deposits collected in the fracture area contained 10<sup>2</sup> 10<sup>4</sup> ppm chloride, and 36-39% sulfate.
- 5. Based on the appearance of the adapter tubes of unfailed specimens, and surface temperature measurements (250°-350°F), on the Lava seal fitting welded to the test section, the service temperature of the adapter tubes in the fracture area is estimated at less than 300°F.

The 1100°F cooled pins operate 350°-450°F below the gas temperature, 1440°-1520°F. This temperature difference imposes stress/strain on the cooled pins during high-temperature service. Figure 2-4 shows a scanning election photomicrograph of a failed 3/8-inch diameter 304 SS adapter tube. The tube

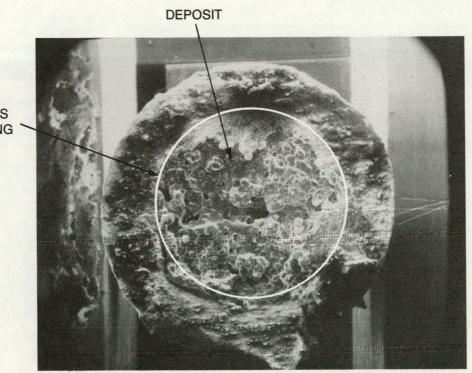


Figure 2-4. Cross-Section of Failed 304SS Adapter Tube. Tubing Is Plugged with Hard Deposit.

3/8" 304SS TUBING ID is plugged with a hard deposit which was determined to be water soluble. X-ray analysis of this deposit revealed it was composed of PFB combustion products, Ca, K, S, Mg, Al, Si along with Fe, Ni and Cr, Figure 2-5. Chemical analysis of deposits taken from the OD of the tube in the fracture area revealed high levels of chlorides and sulfates as noted in 4 above. A photomicrograph of a longitudinal section from the adapter tube is shown in Figure 2-6, illustrating the multiple transgranular branching nature of the cracks, characteristic of stress corrosion or corrosion fatigue cracks in stainless steel exposed to chloride environments. The thermal stresses imposed on the pins during service, combined with the presence of high levels of chlorides and sulfates caused stress corrosion or corrosion fatigue cracking of the stainless steel adapter tubes.

The 1300°F cooled pins operate in the same environment as the 1100°F but there is only a 150°-250°F temperature difference between the pins and the test section gas temperature. The higher metal temperatures of the 1300°F

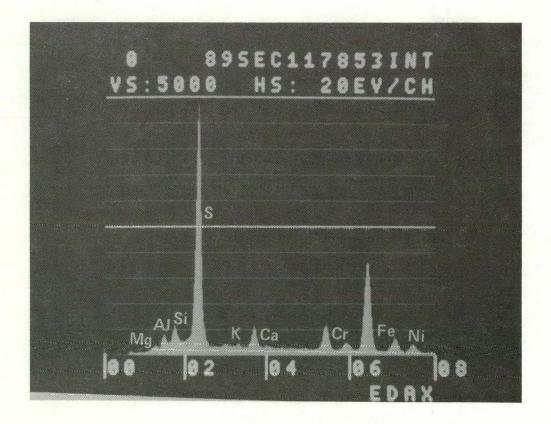


Figure 2-5. X-Ray Spectrum of Deposit Inside Adapter Tube

100 µm

Figure 2-6. Cracking of 304SS Adapter Tube

pins is apparently sufficient to reduce the thermal stresses below a level which causes cracking (within the 2687 hours of testing completed to date). The adapter tube metal temperature at the inlet end of the 1300°F pins should be about the same as the 1100°F pins, but the adapter tubes on the exit end operate about 200°F hotter.

The corrective action taken to alleviate this problem was to replace all the 304 SS inlet adapter tubes on the 1100°F pins with Inconel 625 tubing. This change has been performed on all the long-term pins which have been exposed 1800-2100 hours and also on all new pins to be tested at 1100°F. Inconel 625 is a high strength nickel-base alloy with better fatigue resistance than 304 SS and better resistance to corrosion by chlorides. Further, the 1/4-in. diameter stainless tubes, connecting the 1100°F pins to the cooling air inlet and exhaust manifolds have been replaced with new stainless tubing to minimize potential pitting failures caused by exposure to the low pH condensate resulting from an adapter tube failure.

### SECTION 3 MATERIALS EVALUATION (TASKS 3, 5 AND 6)

A total of 2687 specimen exposure hours have now been completed under the Long Term Materials Test with 1180 hours accumulated during the period from April-June 1983. After 2235-hours of testing, the high-velocity test section was opened and the 24 airfoils and one erosion pin specimen were removed for metallurgical evaluation of erosion/corrosion degradation.

No corrosion pins were removed from the low-velocity test section during this test period. The next pin replacement is scheduled for the first week in July. At that time, two sets of uncooled (1500°F) pins with approximately 1600 and 2700 hours of exposure will be replaced along with one set of cooled pins at each temperature, 1100° and 1300°F, and 1600 hours exposure.

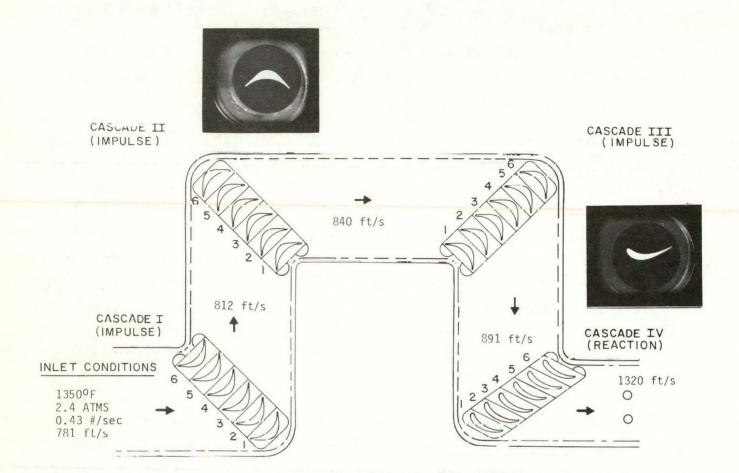
#### High-Velocity Test Section Airfoils - 1150 hours

Six materials were exposed in the high-velocity test section during the 1085 to 2235-hour test interval to evaluate their resistance to erosion/ corrosion degradation at temperatures of 1325°-1375°F. These materials included the following:

IN-738
IN-939
RT-19 (aluminide, pack coating)
RT-44 (rhodium-aluminide, pack coating)
Co-23Cr-10A1-0.4Y (PVD coating)
Fe-25Cr-4A1-0.5Y (PVD coating)

This matrix of airfoils represented the second nominal 1000-hour erosion/ corrosion "screening" test. Actual exposure time for this test was 1150 hours.

Figure 3-1 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 increased from about 900 ft/s at the leading edge of the reaction airfoils to 1200-1400 ft/s at the trailing edge. A single



### Figure 3-1. Schematic of High Velocity Test Section

1/4-inch diameter pin specimen was located downstream of the reaction airfoils in the high-velocity, 1200-1400 ft/s, gas region. During this test period, a Co-23Cr-10Al-0.4Y coated pin was tested.

Dust loadings through the high-velocity test section varied from 34 to 94 ppm (range of 9 measurements) as measured by the Balston Filter dust sampling system. The mean particle size ranged from 1.9 to 2.4 microns with between 2 and 10 percent of the population greater than 10 microns.

Based on the results of the two screening tests (results of the first screening test were reported in the January-March 1983 LTMT Quarterly Progress Report), a materials test matrix was selected for long-term exposure. The test matrix and materials selection criteria are discussed later in this section.

#### Deposition

The concave (pressure) surface of each airfoil was covered with a thin 1-2 mil thick layer of shiny, reddish-brown deposit. These deposits covered most of the concave surface on both the impulse and reaction airfoils. The airfoils exposed during the first 1000-hour test period were generally deposit-free on the pressure surface. The presence of pressure surface deposits indicated that the particulate was probably less erosive during the most recent test period. No difference in dust loadings was noted between the two 1000-hour test periods, but the size distributions did change slightly as follows: 0-1085 hours, mean 2.0-3.1 m with 2-7 percent >10 m; 1085-2235 hours, mean 1.9-2.4 m and 2-10 percent >10 m.

The convex (suction) surface of each airfoil was generally deposit-free toward the leading edge, but covered with a triangular buildup of lightly adherent, friable, reddish-colored ash deposit toward the trailing edge. The deposit pattern is similar to that during the first 1000-hour test period. These trailing edge deposits were bulked for all 24 airfoils and chemically analyzed for concentrations of Na, K, Ca, Mg, Cl and SO<sub>4</sub>. Table 3-1 lists the chemical analyses of these ash deposits.

### TABLE 3-1

### Chemical Analyses of Ash Deposits from HVTS Airfoils -1150 Hours (1085-2235-Hour Test Interval)

|     | WT% Water<br><u>Soluble</u> |
|-----|-----------------------------|
| Na  | 0.074                       |
| K   | 0.14                        |
| Ca  | 7.8                         |
| Mg  | 0.67                        |
| SO4 | 22.0                        |
| CI  | 0.014                       |

### Erosion

Each airfoil was contour traced prior to and after exposure in order to determine the metal loss due to erosion. Table 3-2 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.

### TABLE 3-2

### HVTS - Metal Recession Data 1150 Hours, 1350°F

|                   | METAL<br>Leading Edge | LOSS (MILS)*<br><u>Pressure Surface</u> | Coating/Cladding<br>Thickness (Mils) |
|-------------------|-----------------------|---|--------------------------------------|
| IN-738            | 0.7-3.5               | 0-2.0                                   |                                      |
| IN-939            | 0-2.0                 | 0-1.0                                   |                                      |
| RT-19             | 0-1.2                 | 0-0.3                                   | 2-3                                  |
| RT-44             | 0-0.7                 | 0-0.8                                   | 3-4                                  |
| Co-23Cr-10A1-0.4Y | 0-0.8                 | 0-1.2                                   | 6-8                                  |
| Fe-Cr-Aly         | 0-1.0                 | 0-1.0                                   | 8-12                                 |
| Co-23Cr-10A1-0.4Y | 2.3                   |   | 6-8                                  |
| (Erosion Pin)     |                       |   |                                      |

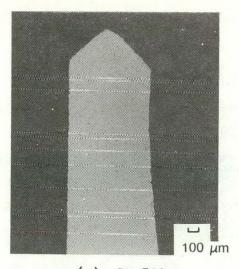
\*Range of measurements on 4 airfoils

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 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.

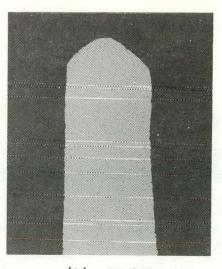
Figure 3-2 shows the leading edge profile of the six airfoils exposed in the third impulse cascade. The unprotected nickel-base alloys, IN-738 (Figure 3-2a) and IN-939 (Figure 3-2b) lost 2-3.5 mils of metal at the leading edge and showed varying degrees of sharpening to a knife-edge configuration. The pressure surfaces were roughened with up to 1 to 2 mils of metal loss.

The RT-19 aluminide coating exhibited metal loss to 1.2 mils on either side of the leading edge (Figure 3-2c) and some thinning of the coating along the pressure wall (<0.05 mils). RT-44 rhodium-aluminide coating showed good erosion resistance with less than 1 mil of metal recession at any location on the airfoils, (Figure 3-2d).

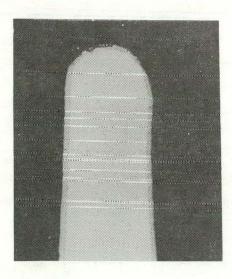
The MCrAlY overlay coatings also showed good erosion resistance on the airfoils with the Co-23Cr-10Al-0.4Y experiencing 0.8 mil or less of leading edge metal loss (Figure 3-2e), and the FeCrAlY coating less than 1.0 mil of metal loss (Figure 3-2f). The pressure surface of the MCrAlY coated airfoils typically were roughened with a maximum of 1.2 mils of metal removal.



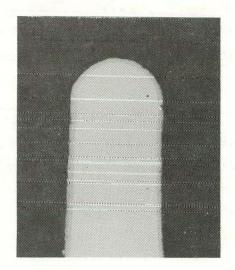
(a) IN-738



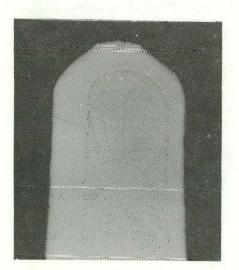
(b) IN-939



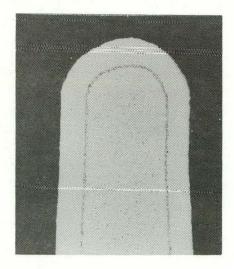
(c) RT-19 Aluminide Coating



(d) RT-44 Rhodium-Aluminide Coating



(e) Co-23Cr-10A1-0.4Y Coating



(f) FeCrAlY Coating

Figure 3-2. HVTS Airfoil Leading Edge Profiles After 1150 Hours at 1350<sup>0</sup>F

A cross-section of the Co-23Cr-10Al-0.4Y coated erosion pin is shown in Figure 3-3. Thinning of the coating occurred on either side of the leading edge resulting in a cam-like appearance. Maximum metal loss of the coating was 2.3 mils. The pin was exposed to gas/particle velocities of 1200--1400 ft/s and despite the relatively large leading edge radius, 0.125-inch, it still experienced severe erosion.

### 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 3-3 lists the maximum corrosion penetration for each material after 1150 hours of exposure.

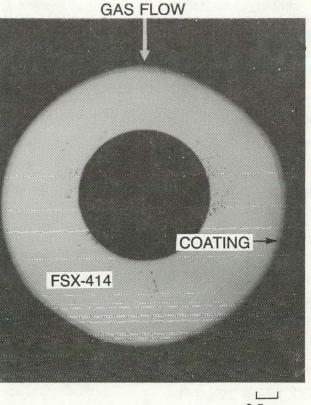




Figure 3-3. CoCrAlY Coated Erosion Pin After 1150 Hours at 1350°F, 1200-1400 ft/s in HVTS

#### TABLE 3-3

### HVTS - Corrosion Penetration Data 1150 Hours, 1350°F

| Material          | <u>Corrosion</u> | Maximum<br>Penetration | (Mils)* | Coating/Cladding<br>Thickness (Mils) |
|-------------------|------------------|------------------------|---------|--------------------------------------|
| IN-738            |                  | 1.2-4.2                |         |                                      |
| IN-939            |                  | 1.6-2.1                |         |                                      |
| RT-19             |                  | 1.2-2.2                |         | 2-3                                  |
| RT-44             |                  | 0                      |         | 3-4                                  |
| Co-23Cr-10A1-0.4Y |                  | 0.5-0.6                |         | 6-8                                  |
| FeCrAly           |                  | 0.8-1.0                |         | 8-12                                 |

\*Range of maximum measurements on 3 airfoils

IN-738 and IN-939 experienced extensive sulfidation attack to maximum depths of 4.2 and 3.0 mils, respectively. Sulfides were generally confined to the leading edge and along the pressure surface, Figure 3-4, with only minor surface oxidation evident on the suction surface and around the trailing edge of the airfoils.

The aluminide coating, RT-19 showed hot corrosion attack as shown in Figure 3-5 with penetration of the coating to the interdiffusion zone. No attack of the IN-738 was observed. In addition to corrosion at the leading edge and pressure surface, the aluminide coating also showed corrosion attack around the crown region of the airfoil on the suction surface.

The rhodium-aluminide RT-44, coating exhibited excellent corrosion resistance with no evidence of attack on any specimen. Figure 3 6 shows the typical appearance of the RT-44 coating after 1150 hours exposure.

The MCrAlY coatings also showed generally good corrosion resistance with both the Co-23Cr-10A1-0.4Y and Fe-Cr-AlY coatings exhibiting areas of internal oxidation less than 1.0 mil deep, Figures 3-7 and 3-8. These areas were again confined to areas of particle impaction with no evidence of corrosion around the suction (convex) surface. Along with internal oxidation of the coating, the CoCrAlY material exhibited a narrow depletion zone ahead of the oxidation attack, Figure 3-7.

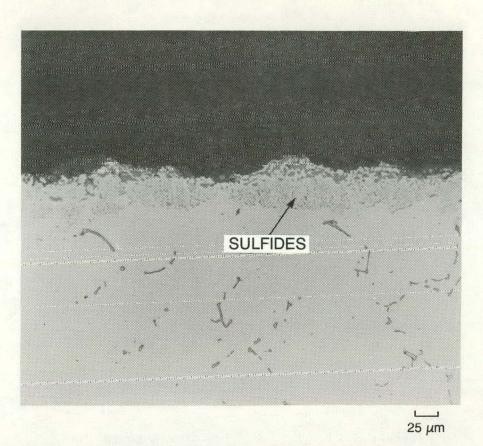


Figure 3-4. Sulfidation Corrosion on Pressure Surface of IN-378 After 1150 Hours at  $1350\,{}^{\rm O}\,{\rm F}$ 

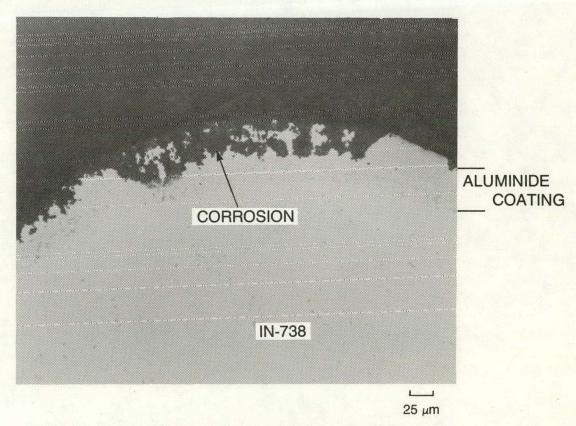


Figure 3-5. Corrosion at Leading Edge of RT-19 Aluminide Coating After 1150 Hours at 1350°F

**RT-44 COATING** and a second · · · · FSX-414 1 1 25 µm Figure 3-6. RT-44 Rhodium-Aluminide Coating on FSX-414 After 1150 Hours at  $1350^{\circ}$  F INTERNAL OXIDATION CoCrAIY Coating 3-995 W IN-738 25 µm Figure 3-7. CoCrAlY Coating Showing Internal Oxidation on Pressure Surface After 1150 Hours at 1350°F

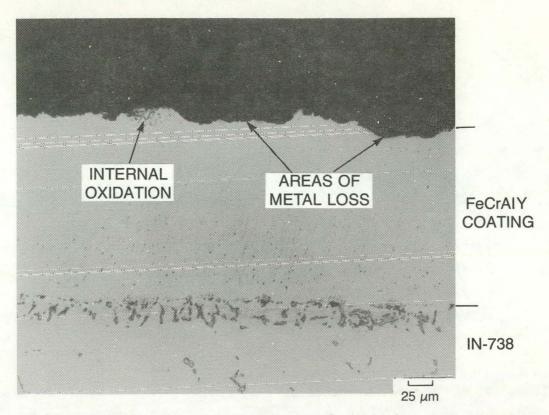


Figure 3-8. FeCrAlY Coating Showing Internal Oxidation on Pressure Surface After 1150 Hours at 1350°F

The absence of corrosion on the suction surface of airfoils can be attributed to the fact that the airfoils are uncooled and operate at the gas temperature of (1350°F). Therefore, there is no alkali enrichment of the deposits as has been observed on the 1300°F cooled pins in the LVTS where the metal surfaces are 200° below the gas temperature (1500°F). This temperature difference between the gas and the metal surface results in condensation of the vapor-phase alkali species.

Corrosion on the airfoil pressure surface and leading edge results from particle impaction which disrupts any protective oxide layer, exposing bare metal to the hot gas environment. The disruption of the protective oxide layer allows further oxidation of the metal, or intimate contact of the metal surface with corrosive alkali species carried by the particulate.

#### **Erosion/Corrosion**

The relative performance of the six materials evaluated in this 1150-hour test period can best be assessed by comparing the amount of total affected metal, including metal loss due to erosion plus internal corrosion penetration. Table 3-4 lists the range of these measurements for the three airfoils (two impulse, one reaction of each material which were destructively evaluated).

#### TABLE 3-4

### HVTS Erosion/Corrosion Data (Mils)

|                        | Maximum                      | Coating/Cladding |
|------------------------|------------------------------|------------------|
| Material               | Total Affected Metal (Mils)* | Thickness (Mils) |
| IN-738                 | 2.4-4.2                      |                  |
| IN-939                 | 1.6-3.0                      |                  |
| RT-19 (aluminide)      | 1.2-2.2                      | 2-3              |
| Co-23Cr-10A1-0.4Y (PVD | ) 0.5-1.2                    | 6-8              |
| FeCrAly (PVD)          | 0.3-1.0                      | 8-12             |
| RT-44 (rhodium-alumini | de) 0.5-0.8                  | 3-4              |

\*Range of maximum measurements on 3 airfoils

The nickel-base alloys, IN-738 and IN-939, showed the most overall materials degradation with 4.2 and 3.0 mils of maximum total affected metal, respectively. Corrosion of the nickel alloys was particularly severe as evidenced by the extensive sulfidation attack shown in Figure 3-4. This internal sulfidation morphology is similar to the type of attack observed on these same materials at 1300°F in the LVIS, but is somewhat accelerated due to the com bined effects of erosion and corrosion introduced by the high-velocity particle impaction.

The RT-19 aluminide coating was penetrated by corrosion to the interdiffusion zone in the 1150-hour exposure, although metal loss due to erosion was not particularly severe. This aluminide coating has shown a high susceptibility to hot corrosion attack at all three temperatures, 1100°. 1300° and 1500°F in the LVTS.

The rhodium-aluminide coating, RT-44, was clearly the best material tested in this group of six materials, exhibiting no corrosion attack and relatively minor erosion of the coating.

The MCrAlY coatings were significantly more resistant to degradation than the base alloys, but not as resistant as the RT-44 coating. The FeCrAlY and CoCrAlY coatings experienced internal oxidation attack in areas of particle impaction. This mode of attack is unique to the high-velocity test section, as the MCrAlY materials showed only minor surface oxidation or no distress at all at 1500°F, and pitting attack at 1100° and 1300°F in the LVTS. Although the MCrAlY coatings showed somewhat more total degradation than the RT-44 coating, the MCrAlY materials can be applied in thicknesses of 8 to 12 mils, while the aluminide coatings are generally 2-4 mils in thickness.

#### HVTS - Materials Selection for Long Term Exposure

Table 3-5 presents a ranking of the relative erosion/corrosion resistance of the 12 materials evaluated during the two nominal 1000-hour screening tests. The ranking was obtained by simply rating the leading edge metal loss, pressure surface metal loss, and maximum corrosion penetration on a 1 to 12 basis (l=least metal loss or corrosion penetration), and summing the three ratings.

Based on the actual erosion/corrosion data and the relative ranking, the following assessments were made:

1. Unprotected base alloys are susceptible to unacceptably high rates of erosion/corrosion degradation (>2-3 mils/1000 hrs).

#### TABLE 3-5

Relative Erosion/Corrosion Resistance of Gas Turbine Materials in HVTS

| RT-44 (rhodium-aluminide)  | Most Resistant  |
|----------------------------|-----------------|
| Co-23Cr-10A1-0.4Y          | 1               |
| FeCrAly (PVD)              |                 |
| Co-30Cr-5A1-0.4Y           |                 |
| RT-19 (aluminide)          |                 |
| FeCrAly (Clad)             |                 |
| CoNiCrAly (Clad)           |                 |
| U-500                      |                 |
| IN-939                     |                 |
| RT-22 (platinum-aluminide) |                 |
| In-738                     | $\checkmark$    |
| FSX-414                    | Least Resistant |

- 2. RT-19 aluminide coating is susceptible to severe corrosion attack (in both the HVTS and the LVTS), and therefore, although ranked relatively high in Table 3-5, is unacceptable for PFB fired gas turbine applications,
- 3. MCrAlY and rhodium-aluminide systems show the best overall erosion/ corrosion resistance. The CoNiCrAlY and FeCrAlY cladding materials appear to be less resistant to degradation than the physical vapor deposition coatings. (The cladding alloys will be dropped from testing at the recommendation of the Materials Advisory Committee.)

Therefore, the following materials have been selected for long-term exposure in the HVTS:

FeCrAlY (PVD) Co-23Cr-10Al-0.4Y (PVD) Co-30Cr-5Al-0.4Y (PVD) RT-44 (rhodium-aluminide) RT-22 (platinum-aluminide)

RT-22, platinum-aluminide coating is being included as this coating is similar to RT-44 and should show similar performance.

In addition to the five materials listed above which will be tested in multiple locations, one airfoil of each of the base alloys and the RT-19 coating will be included for comparison with these 1000-hour test results. Table 3-6 shows the entire HVTS-materials test matrix currently in test. One airfoil of each of the five best materials will be removed after 2500 hours for metallurgical evaluation, with the remaining airfoils continuing to be tested for the duration of the long-term test.

### TABLE 3-6

### High Velocity Test Section -Materials Test Matrix

| <u>Position</u> | Material                     |
|-----------------|------------------------------|
| I-1             | Co-23Cr-10A1-0.4Y/FSX-414    |
| I-2             | RT-22/In-738                 |
| I-3             | Co-30Cr-5A1-0.4/IN-738       |
| I-4             | Co-23Cr-10A1-0.4Y/IN-738     |
| I-5             | FeCrA1Y/IN-738               |
| I-6             | IN-738                       |
| II-1            | FSX-414                      |
| II-2            | FeCrA1Y/IN-738               |
| II-3            | Co-23Cr-10A1-0.4Y/IN-738     |
| II-4            | Co-30Cr-5A1-0.4Y/IN=738      |
| II-5            | RT-22/IN-738                 |
| II-6            | U-500                        |
| III-1           | RT-44/FSX-414                |
| III-2           | Co-23Cr-10A1-0.4Y/IN-738     |
| III-3           | RT-22/IN-738                 |
| III-4           | FeCrA1Y/IN-738               |
| III-5           | Co-30Cr-5A1-0.4Y/IN-738      |
| III-6           | IN-939                       |
| IV-1            | FeCrAly/IN-738               |
| IV-2            | Co-30Cr-5Al-0.4Y/IN-738      |
| IV-3            | IN-939                       |
| IV-4            | RT-44/FSX-414                |
| IV-5            | Co-23Cr-10Al-0.4Y/IN-738     |
| IV-6            | RT-19/IN-738                 |
| V-1             | IN-738 )                     |
| V-2             | FeCrAly (PVD) (Erosion Pins) |

### SECTION 4 PROGRAM MANAGEMENT

On March 22, 1983, a meeting of the Long Term Materials Test Advisory Committee was held at Malta. Participants, in addition to GE personnel, included Jim McClung and Ralph Scott (METC), Sandy Dapkunas (DOE, Germantown), Ron Bradley (ORNL), John Stringer (EPRI), and Mike Klett and Lynn Rubow (Gilbert Associates). The objectives of the meeting were to review results from the first 1000 hours of testing and discuss any other matters relating to the conduct of the program. On April 4, 1983, a letter was issued to the participants which summarized the action items and agreements from the meeting and requested comments and recommendations on the conduct of the program. No responses were received.

During the period, the need to change dolomite produced results which showed a relationship between dolomite particle size and erosion of the in-bed heat exchanger. Besides the results for long-term exposure of turbine materials, the testing in the Malta PFBC continues to produce significant results regarding the optimum operating envelope for a PFBC. Through the end of the period a total of 2687 hours of testing has been completed. The Malta PFBC facility continues to set the standard for long-term reliable operation.