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LONG TERM MATERIALS TEST PROGRAM MATERIALS
EXPOSURE TEST PLAN

December 1981

Work Performed Under Contract No. AC21-79ET15457

General Electric Company
Energy Systems Programs Department
Schenectady, New York

TECHNICAL INFORMATION CENTER
UNITED STATES DEPARTMENT OF ENERGY

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ABSTRACT

The Long Term Materials Test Program is designed to identify promising corrosion resistant materials for coal-fired gas turbine applications. Resistance of materials to long term accelerated corrosion will be determined through realistic PFB environmental exposure of candidate turbine materials for up to 14,000 hours. Selected materials also will be evaluated for their ability to withstand the combined erosive and corrosive aspects of the PFB effluent.

A pressurized fluidized bed combustor facility has been constructed at the General Electric Coal Utilization Research Laboratory at Malta, New York. The 12-inch diameter combustor will burn high sulfur coal with moderate-to-high chlorine and alkali levels and utilize dolomite as the sulfur sorbent. Hot gas cleanup is achieved using three stages of cyclone separators. Downstream of the cyclone separators, a low velocity test section (~ 30 ft/s) capable of housing 180 pin specimens $1/4$ " diameter has been installed to assess the corrosion resistance of the various materials at three different temperatures ranging from 1300° - 1600° F. Following the low velocity test section is a high velocity test section consisting of four cascades of airfoil shaped specimens, six specimens per cascade. This high velocity test section is being used to evaluate the combined effects of erosion and corrosion on the degradation of gas turbine materials at gas velocities of 800-1400 ft/s.

This report summarizes the materials selection and materials exposure test plan for the Long Term Materials Test.

MATERIALS TEST PLAN

INTRODUCTION

The Long Term Materials Test Program is designed to identify promising corrosion resistant materials for coal-fired gas turbine applications. Resistance of materials to long term accelerated corrosion will be determined through realistic PFB environmental exposure of candidate turbine materials for up to 14,000 hours. The ability of the candidate alloys to withstand the corrosive environment alone, as well as the ability of selected materials to withstand the combined erosive and corrosive aspects of the PFB effluent will be assessed.

Candidate base materials and protective systems for both vane and blade applications have been selected for exposure in the long term tests. Materials selection was based on the anticipated conditions in a PFB-powered gas turbine, the applicable experience related to exposure of high temperature materials to coal combustion environments, a fundamental understanding of erosion and corrosion mechanisms, and the applicability of the protective system and its processing requirements to commercial scale PFB-powered gas turbines.

The anticipated conditions in the PFB-powered gas turbine are based on the CFCC reference plant design, combusting a high sulfur coal with moderate-to-high chlorine and alkali levels. The reference system employs a bed temperature of 1750°F and the turbine inlet temperature is approximately 1650°F. Cooling of vane and blades is employed reducing metal operating temperatures to approximately 1100°-1450°F. The exhaust particulate loading and size distribution will be controlled by a series of hot gas clean-up devices. Approach velocities to the turbine components will be in the range of 800-1400 feet per second. Total alkali volatilized from the bed is calculated to be 1-10 ppm depending on the exact coal, dolomite, temperature of operation, and superficial fluidizing velocity.

As the result of these conditions in the turbines, the potential for erosion, corrosion and deposition are judged to be significant. The successful candidate material systems will therefore possess good resistance to both erosion and corrosion.

Published applicable experience in particle laden corrosive environments and coal burning turbines was thoroughly reviewed to identify promising materials. Among the more significant experience surveyed was (1) the early coal-burning turbine efforts by the Australian Aeronautical Laboratory, the Locomotive Development Committee and the U.S. Bureau of Mines, (2) gas turbine experience in sandy terrain and as expander turbines in fluidized catcracker units, (3) erosion-corrosion experiments performed by Curtiss-Wright, Westinghouse, United Technologies, NASA, and General Electric, and (4) recent fluidized bed corrosion/erosion tests performed by the General Electric Company and Curtiss Wright. In addition, the understanding of oil-fired erosion and corrosion mechanisms were used as a basis for establishing the relationship between this related experience and the contemplated PFB powered gas turbine.

A fundamental understanding of the PFB degradation processes includes consideration of the corrosion and erosion resistance of the candidate systems. Key variables including both gas and metal temperatures, gas/particle velocities, alkali level in the system, particulate loading and particulate size distribution must be considered in selection and evaluation of materials for their performance in the PFB environment. A key material property is the ability of the alloy to form and maintain a continuous oxide film for good resistance to corrosion. However, the influence of this oxide film on erosion resistance is unknown, as well as, the influence of the erosion process on the maintenance of the oxide scale. The experience, to date, indicates that superior corrosion resistance also increases erosion resistance in the PFB environment.

Selection of a protective coating/cladding system for a given substrate material must also take into consideration the following:

- The coating should form a thin, adherent oxide scale, free from defects such as cracks, blisters, etc., and exhibiting minimum mass transport

coefficients. The oxide scale should not spall off, vaporize, undergo transformation, or react with salt deposits in any damaging manner.

- The coating should remain metallurgically bonded to the substrate during both processing and service. Flaws such as cavities and holes should neither be present nor form later at the coating/substrate interface.
- The coating should exhibit diffusional stability with the substrate so that during service, the two do not undergo excessive reaction to lose the composite nature of the system.
- Coatings should not adversely affect the mechanical properties of the substrate.
- The substrate and the coating must have similar heat transfer and thermal expansion characteristics.
- The coating should be resistant to stress rupture, thermal fatigue, erosion, and foreign object damage.
- The coating should be ductile at all temperatures, or should undergo brittle-to-ductile transition at a temperature lower than the actual service temperature.
- Coatings should not promote liquid phase formation at or under the coating/substrate interface during high temperature exposure.
- The coating should be thin, uniform, free from flaws, and relatively easy to apply and repair if necessary.
- The method of application of the protective system must be suited for use with PFB-powered gas turbines.

It is unlikely that any one coating or cladding protective system will satisfy all of these requirements as well as provide resistance to erosion. A variety of protective systems and methods of application have been selected for evaluation in the long term materials test. The following sections present details on both the

materials selection and the exposure strategy to be used during the validation test and the long term tests.

MATERIALS SELECTION

The selection of base materials, coatings and claddings for evaluation during the long term materials test is based on all available information regarding the erosion and corrosion behavior of materials in PFBC exhaust gases. Many of these materials already have been tested in the Exxon and/or CURL 1000-hour test. Table 1 lists the materials selected including base alloy, coating/cladding designation and the method of application for the various protective systems. Chemical compositions are listed in Table 2.

All coatings and claddings will be applied to IN-738 substrate with selected coatings also applied to FSX-414. Reasons for selection of each material are as follows:

1. IN-738LC - Common corrosion resistant nickel-base superalloy used as a first stage blade material in industrial gas turbines. This material is used as the base alloy for coatings and claddings in this test.
2. FSX-414 - Cobalt-base superalloy most widely used as a vane material in petroleum fired industrial gas turbines. In petroleum fired turbines its corrosion resistance is superior to IN-738. Results of cascade tests to date suggest that it may be prone to corrosion attack in the effluent from a PFBC. This material also is used as a base alloy for selected coatings.
3. U-500 - Nickel-base superalloy with corrosion resistance as good or better than IN-738LC but lower strength. It has been used as a first stage blade material. It may also be used as a vane alloy to replace FSX-414 which has experienced significant corrosion in the 1000-hour cascade tests run to date. This is the key reason for its inclusion in the test.

TABLE 1MATERIALS SELECTION

<u>Base Alloy</u>	<u>Protective System</u>	<u>Identification</u>	<u>Process</u>
IN-738	FeCrAlY	GE-2541	Clad
IN-738	FeCrAlY	ATD-17	PVD Coating*
IN-738	CoCrAlY	PFB-6	Clad
IN-738	CoCrAlY	ATD-14	PVD Coating
IN-738	CoCrAlY	ATD-2	PVD Coating
IN-738	CoCrAlY	PS-1	Plasma Spray
IN-738	Aluminide + Pt	RT-22	Pack Coating
IN-738	Aluminide	RT-19	Pack Coating
IN-738	Thermal Barrier	PS-TBC	Plasma Spray
IN-738	Ni-48Cr	IN-671	Clad
IN-738	None	-	-
FSX-414	Aluminide + Rh	RT-44	Pack Coating
FSX-414	CoCrAlY	ATD-2	PVD Coating
FSX-414	CoCrAlY	ATD-14	PVD Coating
FSX-414	FeCrAlY	ATD-17	PVD Coating
FSX-414	None	-	-
U-500	None	-	-
IN-739	None	-	-

* Physical Vapor Deposition

TABLE 2
COMPOSITION OF MATERIALS

<u>Material</u>	<u>Ni</u>	<u>Cr</u>	<u>Co</u>	<u>Mo</u>	<u>W</u>	<u>Ta</u>	<u>Cb</u>	<u>Al</u>	<u>Ti</u>	<u>Fe</u>	<u>C</u>	<u>B</u>	<u>Zr</u>	<u>Y</u>	<u>Other</u>
In-738	Bal.	16.0	8.5	1.7	2.6	1.7	0.4	3.4	3.4	--	0.17	0.01	0.10	-	-
In-739/939	Bal.	22.0	19.0	-	2.0	1.4	1.0	1.9	3.7	--	0.15	0.008	0.10	-	-
U-500	Bal.	18.0	18.0	4.0	-	-	-	2.9	2.8	1.0	0.10	0.006	-	-	-
FSX-414	10.0	29.0	Bal.	-	7.0	-	-	-	-	1.0	0.25	0.01	-	-	0.50 Mn, 1.0 Si
IN-671 (CLAD)	Bal.	48.0	-	-	-	-	-	-	-	-	-	-	-	-	-
PFB-6 (Clad)	10.0	25.0	Bal.	-	-	-	-	5.0	-	-	-	-	-	0.15	-
ATD-2 (PVD)*	-	23.0	Bal.	-	-	-	-	12.0	-	-	-	-	-	0.35	-
ATD-14 (PVD)	-	30.0	Bal.	-	-	-	-	5.0	-	-	-	-	-	0.40	-
PS-1 (PS) ⁺	-	30.0	Bal.	-	-	-	-	5.0	-	-	-	-	-	0.40	-
GE-2541 (Clad)	-	25.0	-	-	-	-	-	4.0	-	Bal.	-	-	-	1.0	-
ATD-17 (PVD)	-	25.0	-	-	-	-	-	4.0	-	Bal.	-	-	-	0.3	-
RT-19 (PACK)"	Aluminided														
RT-22 (PACK)	Aluminided + Cr + Pt														
RT-44 (PACK)	Aluminided + Cr + Rh														
PS-TBC (PS)	MgO Stabilized ZrO ₂														

* PVD - Physical Vapor Deposition

+ PS - Plasma Sprayed

" PACK - Pack Cementation

4. IN-739 - New nickel-base superalloy for first stage blades. It is reported to be more corrosion resistant than IN-738.
5. FeCrAlY Cladding/Coating - GE-2541 is an iron-base material which will be clad as 10 mil thick sheet on the IN-738 substrate. This material has demonstrated excellent corrosion resistance in both the Exxon and CURL 1000-hour PFBC cascade tests as well as several atmospheric bed tests and in turbines fired on contaminated petroleum fuels.

ATD-17 is an iron-base coating comparable in composition to GE2541. The coating is applied in a 6-10 mil thick layer by a physical vapor deposition (PVD) process. This FeCrAlY coating is applied to both IN-738 and FSX-414 substrates as a candidate for both blade and vane applications. FeCrAlY coatings have shown good resistance to corrosion/erosion degradation in both the CURL 1000-hour test and the Curtis Wright PFB tests.

6. CoCrAlY Cladding/Coatings - PFB-6 (low Al CoCrAlY Cladding) - This material will be applied as a 10 mil sheet cladding to the IN-738 substrate. It has been tested in both the Exxon and CURL 1000-hour cascade tests, and shown to have excellent corrosion resistance.

ATD-14 (low Al CoCrAlY Coating) - This coating is comparable in composition to the PFB-6 cladding. It will be applied in a 7-10 mil thick layer by the PVD process.

PS-1 (low Al CoCrAlY Coating) - This coating composition again is similar to the PFB-6 cladding alloy, but will be applied by a plasma spraying process to a thickness of 10 mils.

ATD-2 (high Al CoCrAlY Coating) - This cobalt-base coating is a 7-9 mil thick layer applied by a physical vapor deposition process.

ATD-2 is a high aluminum content CoCrAlY composition and will be compared to the low Al CoCrAlY materials to establish the effects of low and high concentrations of aluminum on corrosion and erosion.

7. PS-TBC - PS-TBC is a two layer plasma sprayed (PS) thermal barrier coating (TBC). The inner layer is a high aluminum CoCrAlY bond coat covered by a ceramic outer layer of MgO-stabilized ZrO_2 . The CoCrAlY provides corrosion protection to the substrate while the ceramic layer insulates the base alloy from the high temperature gases so the parts operate at lower effective temperatures. This is representative of a new class of protective coatings developed to protect turbine blades.

8. Pack Aluminide Coatings - Three different aluminide diffusion coatings will be evaluated in the long term testing. Each is applied by the pack cementation process in a 1-3 mil thick layer. RT-22 is an aluminide diffusion coating on a chromium layer with an overlayer of platinum. This coating is presently in service on blades of industrial gas turbines and has excellent corrosion resistance in turbines burning contaminated oils. It is the most advanced and widely used aluminide system commercially available. It has shown superior corrosion resistance in the Exxon 1000-hour test.

RT-19 is a straight aluminide diffusion coating. This coating is less expensive than the RT-22 coating and is generally less corrosion resistance. RT-19 and RT-22 will be applied to IN-738 substrates.

RT-44 is similar to the RT-22 coating except rhodium is substituted for platinum in the coating process. This aluminide coating will be applied to FSX-414 substrates.

9. IN-671 (Ni-48Cr Cladding) - IN-671 is a Ni-48Cr cladding applied as a 10 mil thick sheet which has shown poor corrosion resistance in both the Exxon and CURL-1000 hour PFBC cascade tests. It is included as an indicator of the severity of the environment.

As indicated, four methods of applying the protective systems are being evaluated in the long term tests; pack cementation, plasma spraying, physical vapor

deposition, and cladding via a hot isostatic pressure process. Table 3 lists the advantages and disadvantages of each of these processes.

MATERIALS EXPOSURE STRATEGY

The materials exposure strategy for the long term test is based on a presupposed corrosion curve of the general form shown in Figure 1. Experience has shown that high temperature corrosion of many materials is characterized by parabolic oxidation initially, followed by an extended period of little additional attack with eventual "breakaway" or accelerated hot corrosion. The actual time for breakaway corrosion to occur is a function of the temperature, chemical makeup of the environment and the material. The long term materials test is designed to provide breakaway type corrosion data by exposing a variety of materials to a representative PFB environment over a range of temperatures.

Prior to initiating the long term testing, a 500-hour corrosion validation test is being performed to establish the corrosive conditions in the low velocity and high velocity test sections. A variety of materials also will be evaluated during this 500-hour test. Details of both the 500-hour corrosion test and the long term tests are presented in the following sections.

Low Velocity Test Section - Corrosion

The low velocity test section can accommodate up to 180 test specimens (0.25 inch diameter pins), 60 of which are cooled. This design will permit the testing of up to 12 different materials in the cooled configuration and up to 20 different materials uncooled (18 materials have currently been selected for uncooled test specimens). Table 4 lists the overall test matrix for both the 500-hour validation test and the long term tests.

During the 500-hour validation test, fifteen uncooled materials and eleven cooled materials will be evaluated in the low velocity test section. The uncooled specimens will operate at the gas temperature (1550°-1600°F) and the cooled pins

TABLE 3

APPLICABILITY OF COATING PROCESSES

<u>Materials Applied</u>	<u>Methods of Application</u>	<u>Advantages</u>	<u>Disadvantages</u>
Metals	Pack Cementation	Complex Shapes Coatable	Thin, composition limitation
Ductile Metals	Cladding	Thick overlays - Some compositional flexibility	Complex Parts - Restricted to low Al (< 5%)
Metals, Oxides, Carbides	Thermal Spraying	Cheap, fast, complex shapes coatable, compositional flexibility	Flaws, thickness control difficult
Metals	Physical Vapor Deposition	Complex shapes coat-able, reliable	Expensive

FIGURE 1

PHILOSOPHY OF LTMT - BREAKAWAY CORROSION

will run at 1450°F. 1450°F was selected to test the cooled pins as this temperature represents the maximum metal operating temperature for a PFB fired gas turbine based on the CFCC design. Two specimens of each material will be tested at each temperature, one of which will be removed after 250 hours and the other after completion of 500 hours. This test matrix will provide 250- and 500-hour corrosion data on fifteen materials at 1600°F and 11 materials at 1450°F.

For the long term test, the matrix will be expanded to three temperatures, 1300°, 1450°, and 1600°F. The second cooled pin temperature of 1300°F will be utilized to study the susceptibility of the various candidate materials to low temperature corrosion. Low temperature corrosion in the temperature range of 1100-1400°F has been observed in gas turbines operating in marine environments. Further, recent results from tests in a high temperature, high velocity erosion/corrosion rig, performed in conjunction with this program, indicate that some materials may be susceptible to rapid degradation at 1350°. (Testing at 1300°F is not being carried out during the 500-hour test as a second cooling air manifold is required on the low velocity test section.) Twelve materials will be evaluated at each cooled temperature, 1300° and 1450°F, using 30 of the 60 cooled positions for each temperature. Figure 2 shows the planned specimen replacement schedule for any two materials to be tested at either 1300° or 1450°F. Thus, after 14,000 test hours, corrosion data will be available for twelve materials at 1300° and 1450°F for the following exposure intervals: 250, 500, 1000, 2500, 7000, 9750 and 14,000 hours.

The 120 positions available for uncooled test specimens will provide more extensive mapping of the corrosion behavior of each material. Currently, eighteen materials have been selected for evaluation which will utilize 108 of the available positions. The twelve remaining positions will be used for thermo couples to monitor the gas temperature and for installation of test specimens fabricated from

SPECIMEN POSITION

A, B – TWO DIFFERENT MATERIALS

FIGURE 2

COOLED SPECIMEN REPLACEMENT SCHEDULE

SPECIMEN POSITION

FIGURE 3

UNCOOLED SPECIMEN REPLACEMENT SCHEDULE

alternate materials. Figure 3 shows the planned replacement schedule for a single material exposed at 1600°F. Detailed examination of this exposure plan will reveal that each replacement interval is repeated during the long term test thereby providing duplication of the corrosion data for each material at 1600°F; 250, 1000, 2500, 5000, 7000, 9000 and 14000 hours. The spare specimen will be used for discretionary purposes to monitor specimen condition in case of an upset in the test conditions.

It is anticipated that some of the materials to be exposed at each temperature will not survive the entire 14,000 hours of testing. When a material fails due to corrosion at any of the three test temperatures, the specimen positions occupied by that material (only at the temperature where breakaway corrosion occurred) will be used for one of the following purposes: 1) to test a new material not included in the initial test matrix, 2) to expose specimens of a "good" material for additional intervals, or 3) to repeat the exposure intervals of the material which exhibited breakaway corrosion.

High Velocity Test Section - Erosion/Corrosion

The materials selected for evaluation of their erosion/corrosion resistance in the high velocity test section are listed in Table 4. Figure 4 shows a schematic of the high velocity test section. The 24 airfoil specimens operate at the gas temperature which will be approximately 1300°-1400°F based on early test operation data. The first three cascades are impulse cascades designed to turn the flow without accelerating the gas, except for small frictional losses. Approach gas velocities to the first three cascades will be 800-900 ft/s. The fourth cascade consists of reaction airfoils which accelerate the flow from about 900 ft/s to 1700 ft/s. Downstream of the reaction airfoil are two pin specimens which will be exposed to gas velocities of about 1400 ft/s. One pin specimen will operate at the gas temperature 1300°-1400°F, and the other will be air cooled to approximately 1100°F. (This is the only cooled specimen in the high velocity test section.)

During the 500-hour validation test one impulse airfoil of each of twelve materials will be tested. In addition, one reaction airfoil of each base alloy, IN-738, IN-739, U-500 and FSX-414, will be tested along with two FSX-414 pin specimens. Table 5 shows the test matrix for the high velocity test section. Exposure of these specimens will provide 500-hour erosion/corrosion data on all the materials.

TABLE 4
MATERIALS TEST MATRIX

Base Alloy	Protective System	Identification	Process	Low Velocity Test Section				High Velocity Test Section	
				Uncooled		Cooled		500 hr	Lg Trm
				500 hr	Lg Trm	500 hr	Lg Trm		
IN-738	FeCrAlY	GE2541	Clad	x	x	-	x	x	x
IN-738	FeCrAlY	ATD-17	Coating	x	x	x	x	x	x
IN-738	CoCrAlY	PFB-6	Clad	x	x	-	x	x	x
IN-738	CoCrAlY	ATD-14	Coating	x	x	x	x	x	x
IN-738	CoCrAlY	ATD-2	Coating	x	x	x	x	x	x
IN-738	CoCrAlY	PS-1	Plasma Spray	x	x	-	-	-	-
IN-738	Aluminide+Pt	RT-22	Coating	x	x	x	x	x	x
IN-738	Aluminide	RT-19	Coating	x	x	x	x	x	x
IN-738	Thermal Barrier	PS-TBC	Plasma Spray	x	x	-	-	-	-
IN-738	Ni-48Cr	IN-671	Clad	x	x	x	-	-	-
IN-738	None	-	-	x	x	x	x	x	x
FSX-414	Aluminide + Rh	RT-44	Coating	x	x	x	x	-	-
FSX-414	CoCrAlY	ATD-2	Coating	-	x	-	x	x	x
FSX-414	CoCrAlY	ATD-14	Coating	-	x	-	-	-	-
FSX-414	FeCrAlY	ATD-17	Coating	-	x	-	-	-	-
FSX-414	None	-	-	x	x	x	x	x	x
U-500	None	-	-	x	x	x	x	x	x
IN-739	None	-	-	x	x	x	x	x	x

x = material to be tested

FIGURE 4

SCHEMATIC OF HIGH VELOCITY TEST SECTION

TABLE 5
HIGH VELOCITY TEST SECTION
MATERIALS TEST MATRIX FOR 500-HOUR VALIDATION TEST

	<u>Material</u>	
I-1	*	
I-2	U500	
I-3	FSX-414	
I-4	IN-738	
I-5	IN-739	
I-6	*	
II-1	*	
II-2	GE2541/IN-738	(FeCrAlY clad)
II-3	ATD-2/IN738	(CoCrAlY PVD) Hi Al
II-4	ATD-14/IN-738	(CrCrAlY PVD) Lo Al
II-5	ATD-17/IN-738	(FeCrAlY PVD)
II-6	*	
III-1	*	
III-2	PFB-6/IN-738	(CrCrAlY Clad) Lo Al
III-3	RT-22/IN-738	Pt-Cr-Al
III-4	RT-19/IN-738	Al
III-5	ATD-2/FSX-414	(CoCrAlY PVD) Hi Al
III-6	*	
IV-1	*	
IV-2	U-500	
IV-3	FSX-414	
IV-4	IN-738	
IV-5	IN-739	
IV-6	*	
V-1	FSX-414	
V-2	FSX-414 (cooled)	

*Specimen previously used during qualification test.

The long term test will consist of a series of four 1000-hour tests designed to screen 12 materials (three at a time). At the end of these tests (total 4000 hours), three or four materials will be chosen and tested for the remaining 10,000 hours. During this 10,000 hour test specimens of each material will be removed at 2500 hours and replaced, thus producing erosion/corrosion data for each material (three or four different materials) at 1000, 2500, 7500 and 10,000 hours.

The high velocity erosion pins will be inspected periodically and replaced as required based on the condition of the specimens.

MATERIALS EVALUATION

Pre-Test Characterization

Prior to exposure in the test facility each specimen will be weighed, visually inspected for surface defects or irregularities, and dimensionally checked. The dimensional checks will consist of diametrical measurements of the pin specimens, and profile contour tracing of the airfoil specimens. The contour tracing is a 20X magnification profile of the airfoil accurate to ± 0.001 inch. In addition to these non-destructive characterizations selected specimens of each base alloy and coating or cladding material will be metallographically evaluated for microstructure, bond integrity and internal defects.

Post-Test Evaluation

Upon removal from the test, each specimen will be examined and photographed to characterize the nature and extent of erosion, corrosion and deposition on a gross basis. Next the specimens will be cleaned of deposits, which will be analyzed, followed by another visual inspection and dimensional check similar to the pre-test checks. Each specimen will be weighed, followed by sectioning for metallographic evaluation. These sections will be examined microscopically for total corrosion penetration and metal loss. If required, scanning electron microscopy and/or electron microprobe analysis will be used to study the corrosion and/or erosion morphology.