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EFFECTS OF SURFACE CHEMISTRY ON HOT CORROSION LIFE

FIRST ANNUAL REPORT

by

By

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**General Electric Company
Aircraft Engine Business Group
Cincinnati, Ohio 45215**

Prepared for

National Aeronautics and Space Administration

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ABS: Baseline burner rig hot corrosion with Udimet 700, Rene' 80; uncoated and with RT21, Codep, or NiCoCrAlY coatings were tested. Test conditions are: 900C, hourly thermal cycling, 0.5 ppm sodium as NaCl in the gas stream, velocity 0.3 Mach. The uncoated alloys exhibited substantial typical sulfidation in the range of 140 to 170 hours. The aluminide coatings show initial visual evidence of hot corrosion at about 400 hours, however, there is no such visual evidence for the NiCoCrAlY coatings. The turbine components show sulfidation. The extent of this distress appeared to be inversely related to the average length of mission which may, reflect greater percentage of operating time near ground level or greater percentage of operation time at takeoff conditions (higher temperatures). In some cases, however, the location of maximum distress did not exhibit

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16. Abstract Baseline burner rig hot corrosion tests are underway with Udimet 700, Rene' 80; uncoated and with RT21, Codep, or NiCoCrAlY coatings. Test conditions are: 900C, hourly thermal cycling, 0.5 ppm sodium as NaCl in the gas stream, velocity 0.3 Mach. The uncoated alloys exhibited substantial typical sulfidation in the range of 140-170 hours. The aluminide coatings showed initial visual evidence of hot corrosion at about 400 hours; at this stage there is no such visual evidence for the NiCoCrAlY coatings. Detailed evaluation of service-run turbine components revealed sulfidation in five of six examples. The extent of this distress appeared to be inversely related to the average length of mission which may, for example, reflect greater percentage of operating time near ground level or greater percentage of operating time at takeoff conditions (higher temperatures). In some cases, however, the location of maximum distress did not exhibit the structural features of hot corrosion.					
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FORWARD

This First Annual Report, covering the period 2 May 1983 to 2 May 1984, was prepared by the Engineering Materials Technology Laboratories (EMTL) of General Electric's Aircraft Engine Business Group, Cincinnati, Ohio, 45215, under NASA Contract NAS3-23926. R. E. Fryxell of General Electric is the Principal Investigator and J. P. Merutka of NASA-LeRC is the Project Manager for NASA.

This program involves extensive collaboration, under subcontract, of B. K. Gupta (and C. S. Kortovich) at the Materials and Manufacturing Technology Center of TRW Inc., Cleveland, Ohio, 44117.

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SUMMARY

This is the first annual report of a 3-year program. The objective is to determine surface chemistry effects of oxide scale and coating composition on the hot corrosion life of selected alloys in the coated and uncoated condition and to provide data for development of a hot corrosion life prediction model. The coated and uncoated alloys are to be evaluated by high velocity hot corrosion rig testing in the as-processed condition and after various aging treatments designed to simulate surface chemistry effects of in-service conditions. The alloys are Udimet 700 and René 80, both uncoated and with RT21, Codep, or NiCoCrAlY coatings. This testing is partially complete for specimens in the as-processed condition. Preliminary results demonstrate typical sulfidation microstructures and the expected relative performance of the various materials. All test specimens in this program are periodically removed from test for inspection and for nondestructive tracking of the progress of corrosion by measurement of 10 MHz coil inductance changes by the technique developed at NASA-LeRC. Later in the program, data from the evaluation of all rig-tested specimens will be used to develop a hot corrosion life prediction model and methodology for the specific alloys and coatings tested in the program. The capability of the life prediction model and methodology will then be tested by conducting additional hot corrosion tests on both the program coatings and alloys as well as other coatings and alloys.

Parallel with this laboratory test program is a comprehensive evaluation of six service-run turbine components to (1) establish extent and mechanism of hot corrosion degradation and (2) correlate observations to engine operating history, where possible. Sulfidation was observed in five of the six components.

Qualitatively, the extent of corrosion appears to be inversely proportional to average length of mission. However, with such a limited sampling it is not possible to determine whether this reflects fraction of time under takeoff conditions (higher temperatures), fraction of time near ground level (higher propensity for contamination) or some other related parameter.

INTRODUCTION

The program is a 36-month joint effort including the Engineering Materials Technology Laboratories of the General Electric Company Aircraft Engine Business Group and the Materials and Manufacturing Technology Center of TRW, Inc. It is divided into five tasks.

Task I involves the evaluation of six turbine components showing visual evidence of hot corrosion, and having known operating history, to establish the degree of degradation and mechanisms in the corroded areas. Correlation of the corrosion with operating conditions will be made to the extent possible. Evaluation includes chemical and X-ray diffraction analyses of surface scales/deposits as well as metallography, scanning electron microscopy, and electron microprobe examination. Both uncoated and coated hardware are included, and emphasis is placed on the cause and effect parameters which are associated with hot corrosion. The results of this study will serve as a point of reference in the remainder of this program which is based on laboratory testing. All burner rig tests are performed by TRW Inc., Cleveland, Ohio.

Task II will establish a hot corrosion baseline for the program alloys and coatings in the as-processed condition and involves up to 1000 hours exposure of duplicate specimens in a Mach 0.3 burner rig using Jet A fuel (0.045-0.065% sulfur) and in which 0.5 ppm sodium by weight, as NaCl, is added to the combustion air. The test cycle is 1 hour at 900° C, followed by 6 minutes of forced air cooling to ambient temperature. Specimens are to be inspected at approximately 20 cycle intervals, and removed from test when evidence of hot corrosion is noted in three successive inspections. Additional specimens will then be tested, as space is available, for time periods of 100, 300, and 500 hours if these time periods do not exceed 2/3 the time the original specimen of the same material was exposed. Specimens to be tested in this task include U700 and René 80, both uncoated and with the following coatings:

Pack aluminide RT21 (Chromalloy) and Codep (General Electric)
Low pressure plasma-sprayed NiCoCrAlY (Ni-23Co-18Cr-12Al-0.3Y)

In Task III, triplicate specimens of the above coated alloys will be aged at 1100° C under a variety of conditions, both oxidizing and inert environments, to induce coating-alloy interdiffusion with and without surface oxidation. These specimens will be hot corrosion tested in Task IV under the same conditions as in Task II to determine the effects of aging on hot corrosion behavior.

The aging treatments in Task III will be isothermal for 100 hours in a vacuum; isothermal air furnace oxidation for 100, 300, and 600 hours; 1 hour air furnace cycles for 100 hours; and cyclic burner rig oxidation for 100, 300, and 600 hours.

One specimen representing each of the above conditions will be evaluated using the methods outlined in Task I; the remaining duplicate specimens representing each condition will be used in Task IV.

At the conclusion of Task IV, all data will be consolidated, including the effects of aging on hot corrosion behavior, and an empirical hot corrosion life prediction model will be proposed.

Task V will be a hot corrosion cyclic burner rig test designed to check the validity of the proposed life prediction model. Duplicate specimens of a maximum of four alloys and five coatings will be tested up to a maximum of 1000 hours and evaluated as in Tasks II and IV. The alloys and coatings will be those from this program plus additional alloy-coating systems selected by the NASA Project Manager.

The Task V experiment will be designed to test two aspects of the life prediction model developed in Task IV.

1. The ability of the model to predict hot corrosion life under different rig test conditions will be evaluated using alloys and coatings previously tested in Task II and Task IV. The hot corrosion tests will use one or more variations of the test parameters used in Task II, III, and IV. Test parameter variations that will be considered include the following:
 - A different salt level
 - A different test temperature
 - Intermittent salt injection

- A different sulfur level in the fuel
- Additional or modified aging cycles.

The specific testing parameters will be derived from the results of Task II and Task IV, previous experience in evaluating field service hardware and previous experience in conducting hot corrosion testing under a variety of test conditions.

2. The ability of the model to predict hot corrosion resistance of new coatings and alloys will be evaluated using alloys and coatings that have not been evaluated previously in Task II and Task IV. Rig test conditions will be identical to those used in Task II and Task IV. The aging cycle, if any, will be chosen from the results of Task IV. Incorporated in the model will be a variety of information on the effect of compositional variables on hot corrosion.

TASK I - EVALUATION OF TURBINE COMPONENTS FROM FIELD SERVICE

This study involves a comprehensive evaluation of six turbine components, blades, and vanes, which show evidence of hot corrosion in addition to normal oxidation degradation. These were selected from uncleaned engines for which operating history was reasonably well defined, to provide a basis for estimating cause and effect parameters.

In the course of searching for suitable examples, it became very clear that, although uncleaned components were readily available, they generally have suffered some handling. This reduces to a certain extent the reliability of surface analysis for airborne contamination which may either be high from handling contamination or low from losses of relatively nonadherent surface deposits. This, however, in no way jeopardizes metallographic evaluation and correlation with operating history. Actually, evidence of corrosive contamination such as sodium compounds on airfoil surfaces is of second order importance in that ingestion of such contamination in an airborne turbine is intermittent and the amount found may only represent ingestion during the final landing before engine teardown.

It should be pointed out that in the high pressure turbine (HPT) of the engine models available for this study, hot corrosion is not frequently observed. The great majority of the engines run for considerably longer times

than the selected examples before first removal without life-limiting environmental degradation. The five HPT examples selected were obtained only after examining blades from a large number of disassembled engines. On the other hand, hot corrosion is more frequently encountered in the low pressure turbine (LPT) since, at the lower temperatures involved, corrosive contaminants are more likely to accumulate in significant amounts and/or be present a greater percentage of the time. The sixth component selected is a LPT vane.

Identification of the six components with their provenance and summary of available operating history is given in Table I. Four of these are the same component from engines of the same model to afford maximum opportunity to make cause and effect correlations, thus eliminating design and operational differences known to be significant. Evaluation of these components followed the sequence:

1. Examine visually and photograph.
2. For the three components for which duplicate parts are available, rinse concave and convex airfoil surfaces separately for the duplicates. Analyze for water soluble sodium, calcium, magnesium, cobalt, nickel, and sulfate.
3. Based on visual examination, select suitable locations for cutting which represent regions of maximum distress. Cut dry, prepare metallographic mounts, and polish under kerosene.
4. Examine mounts in the as-polished condition up to 500X for evidence of sulfidation.
5. Selectively analyze scale and underlying metal by scanning electron microscopy (SEM-EDAX) and by electron microprobe (EMP).
6. Etch mounts and examine for evidence of alloy depletion.
7. Measure maximum distress at various locations, including metal loss and alloy depletion.
8. Define type of attack, whether oxidation, Type 1 hot corrosion, Type 2 "intermediate temperature" hot corrosion.
 - Type 1 - A zone of alloy depletion containing sulfide particles.
 - Type 2 - No alloy depletion in the affected area, and sulfides only at the scale-alloy interface. The scale is dense and layered.

Table I. Components Selected for Evaluation.

<u>Carrier Base</u>	<u>Aircraft</u>	<u>Component</u>	<u>Total Hours</u>	<u>Number of Cycles</u>	<u>% Takeoffs at Coastal Airports</u>
Southern Asia	A300B	(1)*	2,694	1560	67
Northern Africa	A300B	(1)	4,641	2169	77
Southern Africa	A300B	(1)	1,830	1471	50
Western Europe	DC10-30	(1)	2,625	610	46
South America	DC10-30	(2)*	15,155	2987	72
U.S. Military	F4	(3)*	2,700		

(1) CF6-50 Stage 1 high pressure turbine blade, Codep coated Rene' 80.

(2) CF6-50 Stage 1 low pressure turbine vane, uncoated Rene' 77.

(3) J79 Stage 1 high pressure turbine blade, Codep coated Rene' 80.

* Duplicate parts available for determining composition of water soluble deposits.

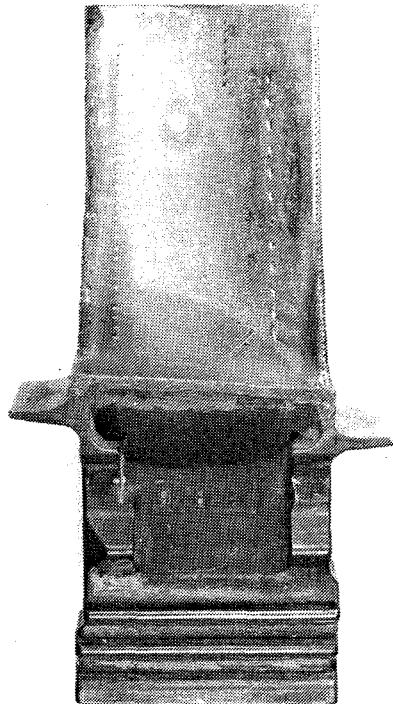
9. Document typical regions of distress with photomicrographs.
10. Analyze by X-ray diffraction selected samples of scale removed from areas as close as possible to locations of maximum distress.

The four CF6-50 turbine blade evaluations first will be described individually and then as a group to indicate correlation to the extent possible with operating history. Metallographic mounts were prepared of three transverse cuts of each airfoil. After preliminary examination, one mount from each blade was eliminated from detailed study since environmental degradation was clearly less. The intent of the study was to identify locations of maximum distress and to establish mechanisms, whether oxidation or hot corrosion. As is typical with high pressure turbine blades, the convex airfoil surfaces (CVX) exhibit minimal degradation; thus the following discussion refers only to concave/pressure surfaces (CCV), in some cases including the leading edge (LE). Specific locations are designated by % span (radial)/% chord (distance from leading edge to trailing edge). Photographs of the concave surfaces of the four CF6-50 turbine blades are shown in Figures 1a and 1b.

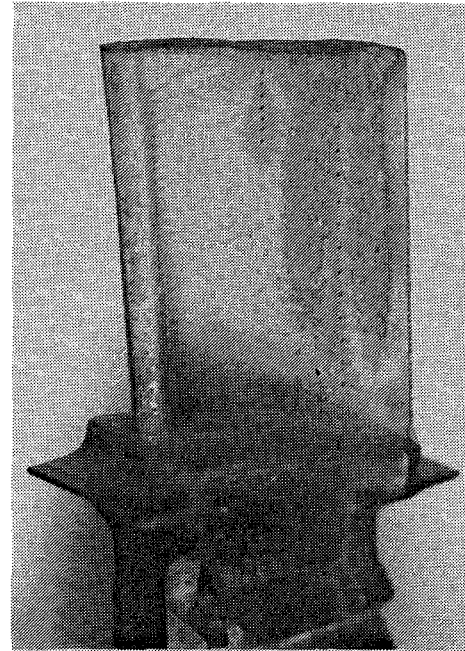
Southern Asia

At the 40%/70-90% location, the coating was breached and some attack of the substrate was evident. Maximum distress was about 100 μm (0.004 inch). Type 1 subsurface sulfides were clearly visible, as shown in Figure 2, within remaining coating at the edge of the distressed region. Confirmation that these particles were sulfides was obtained by EMP as shown in Figure 3.

In the 60% radial section, similar Type 1 structures were observed with a maximum distress of about 50 μm (0.002 inch) at the LE; the diffusion zone of the coating was still intact. EDAX examination confirmed the presence of subsurface sulfides, predominantly of Ti and Ni. The scale was rich in Al with lesser amounts of Cr and Ti, and some Ca. Calcium sulfate was identified by X-ray diffraction. However, the greatest distress occurred at 60-80% chord, about 200 μm (0.008 inch); thorough EDAX examination failed to reveal sulfides, and the scale was Cr rich with lesser amounts of Ti and Al. Thus in this blade, although Type 1 hot corrosion was strongly evident, the most distressed region had both structural and compositional features of oxidation.

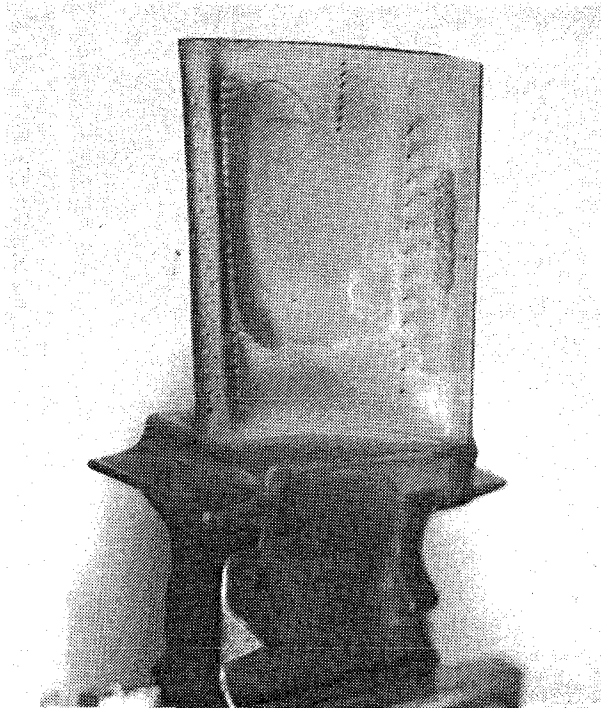


Southern Asia: A300B
2694 hours, 1560 Cycles



Nothern Africa: A300B
4641 hours, 2169 Cycles

Figure 1a. Stage 1 High Pressure Turbine Blades from CF6-50 Engines.

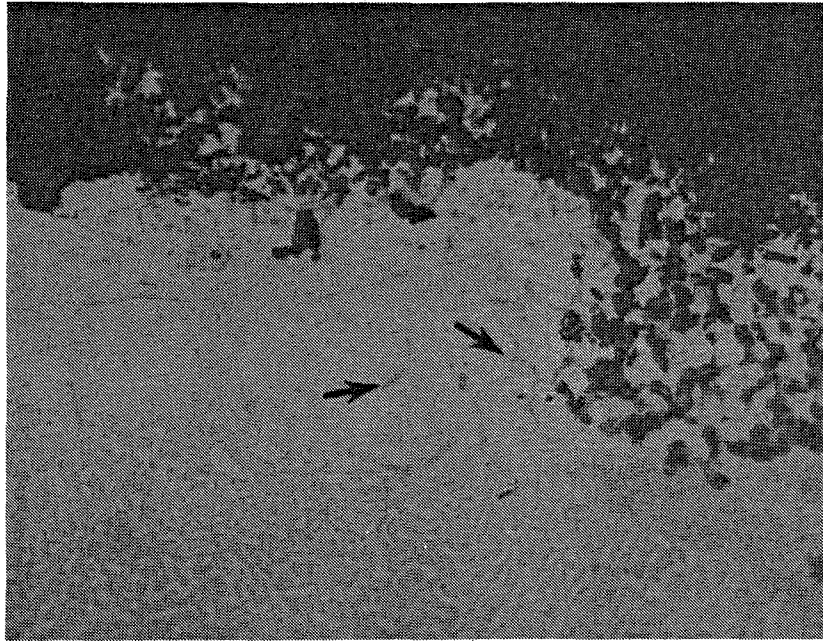


Southern Africa: A300B
1830 hours, 1471 Cycles



Western Europe: DC10-30
2625 hours, 610 Cycles

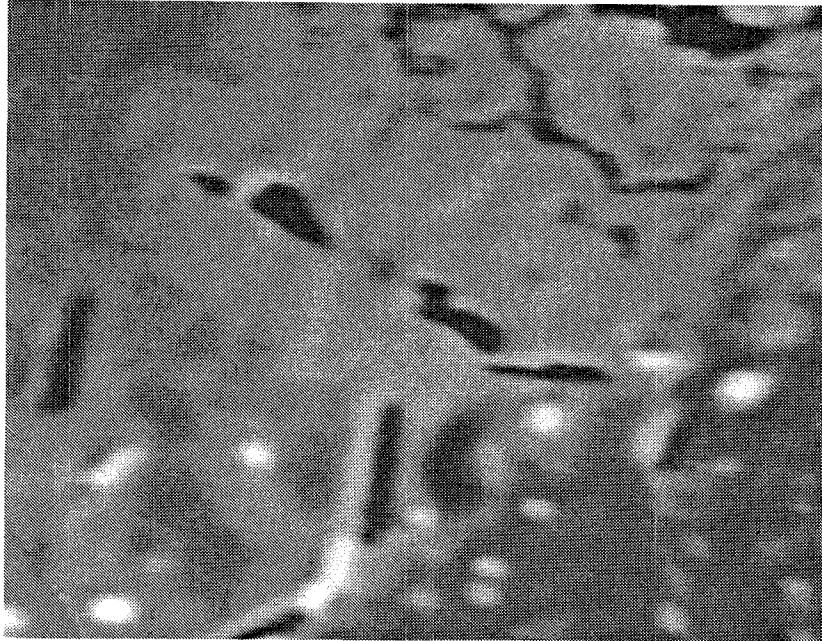
Figure 1b. Stage 1 High Pressure Turbine Blades from CF6-50 Engine.



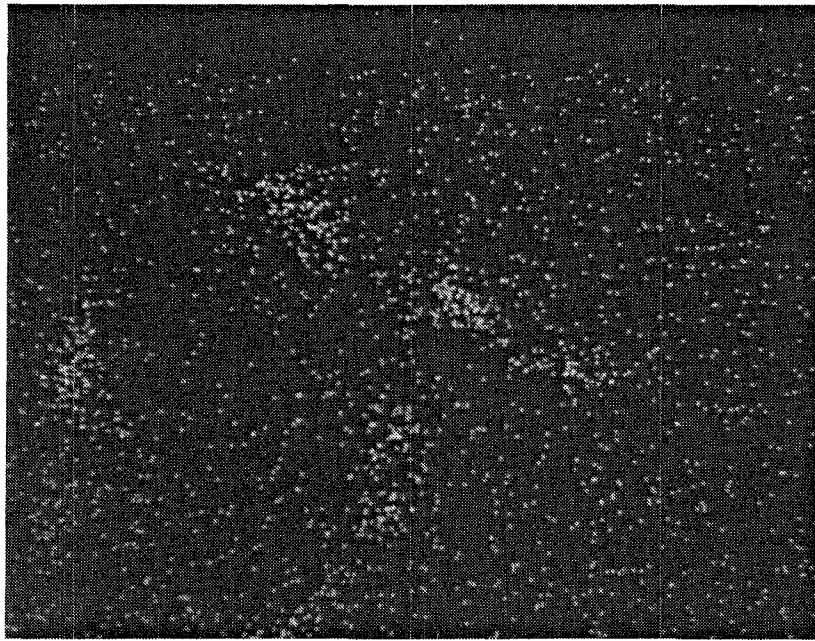
As-Polished

100X

Figure 2. Photomicrograph at the 40%/80% Location on the Concave Airfoil of a Stage 1 High Pressure Turbine Blade from a Southern Asia CF6-50 Engine. Type 1 Sulfides are Present (Arrows).



BSE



Sulfur

Figure 3. EMP Configuration of Sulfides in the Gray Subsurface Particles Shown in Figure 2. Stage 1 High Pressure Turbine Blade from a Southern Asia CF6-50 Engine (5000X).

Northern Africa

At the 50%/60-90% location, the coating was breached and considerable loss of the substrate alloy had occurred to a total depth of about 230 μm (0.009 inch) including a depletion layer. Type 1 subsurface sulfides were clearly visible as shown in Figure 4, together with strong EDAX confirmation of sulfur, combined with Ti, Cr, and Ni. The scale was Cr and Al rich with some Ti. Although Ca was not observed by EDAX, CaSO_4 was identified by X-ray diffraction from a scale specimen adjacent to the cut region. The depletion zone in the substrate showed lower levels of Cr, Al, and Ti than in the unaffected René 80. At the 80%/50-80% location, similar structures were observed, again to a maximum depth of about 230 μm (0.009 inch).

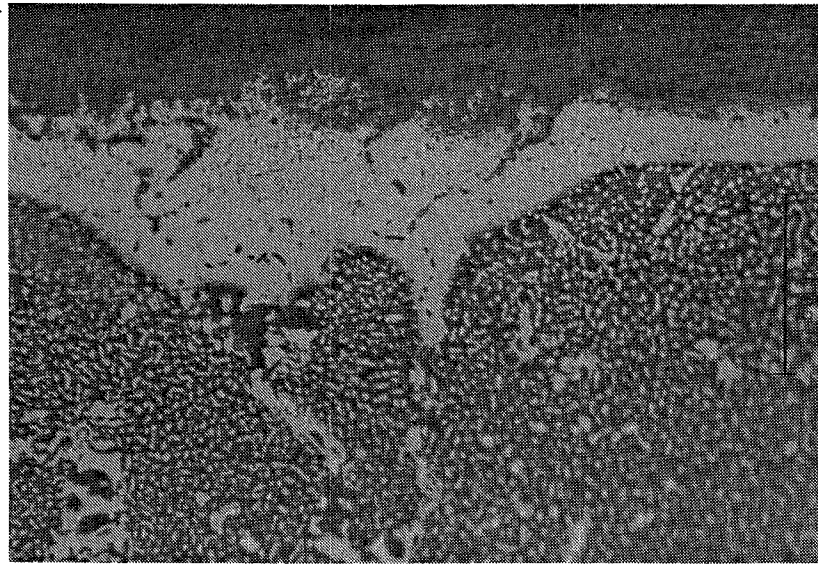
Southern Africa

For this blade, the maximum distress of about 180 μm (0.007 inch) was observed at the 60%/90% location and showed the structural features of oxidation only (Figure 5a). No sulfides were found by EDAX examination. The scale contained substantial amounts of Ca and some Fe (from the environment) as well as Al.

Type 1 sulfidation was confirmed by both EMP and EDAX in the 10% radial section at the leading edge (Figure 5b) and at 60-80% chord. Maximum distress was about 50 μm (0.002 inch) and 75 μm (0.003 inch) at these two locations, respectively. The outer scale was rich in Al with a lesser amount of Ti (LE) and the depletion zone in the exposed substrate at 60-80% chord again was lower in Cr, Al, and Ti than in the unaffected René 80.

Western Europe

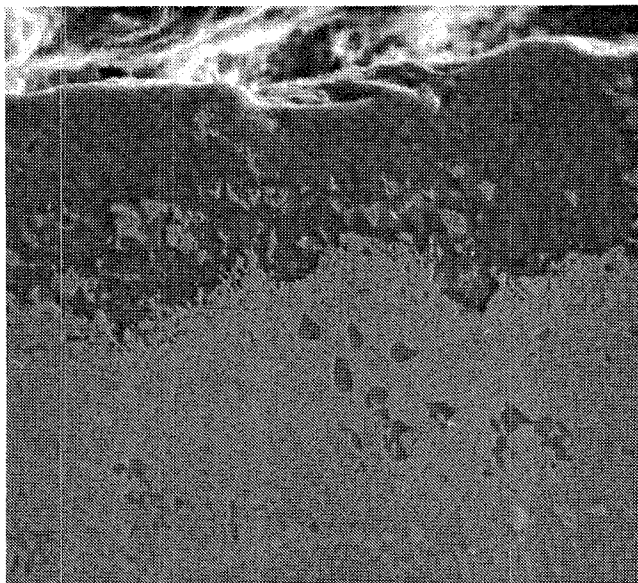
This was the least distressed of the four CF6-50 turbine blades. There was faint visual evidence for Type 1 sulfidation within the coating (which was not breached) in the 10% section, at the leading edge and at 60-80% chord, maximum penetration about 45 μm (0.0018 inch). EDAX analysis of the scale showed strong Al and lesser amounts of Ti and Cr. Sulfur was, however, not confirmed.



Etched

(a)

500X



(b)

1450X

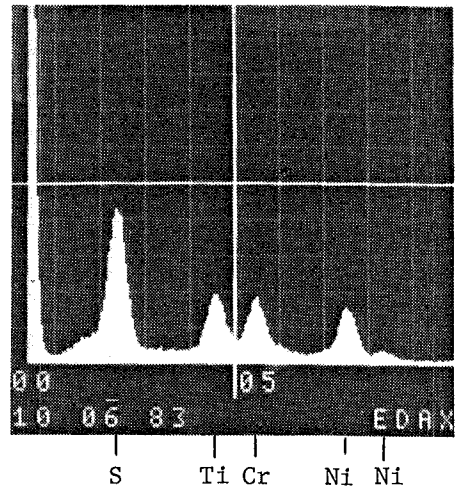


Figure 4. Photomicrographs at the 50%/60-90% Location on the Concave Airfoil of a Stage 1 High Pressure Turbine Blade from a Northern Africa CF6-50 Engine. The Gray Subsurface Particles in (b) were Identified by EDAX as Sulfides Containing Ni, Cr, and Ti.

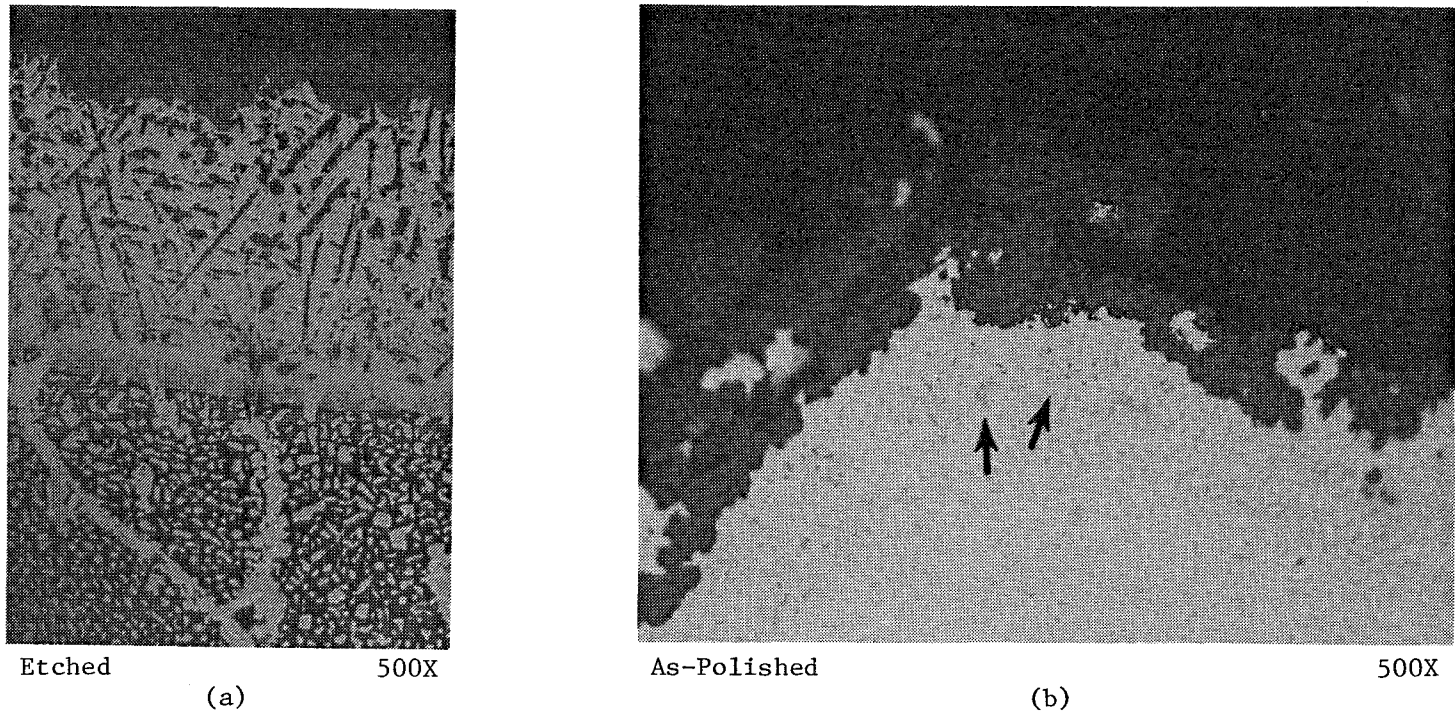


Figure 5. Photomicrographs at the (a) 60%/90% Location on the Concave Airfoil and (b) 10%/LE Location of a Stage 1 High Pressure Turbine Blade from a Southern Africa CF6-50 Engine.

(a) Conventional Oxidation with No Evidence of Sulfides

(b) Considerable Subsurface Sulfides Within Residual Coating (Arrows)

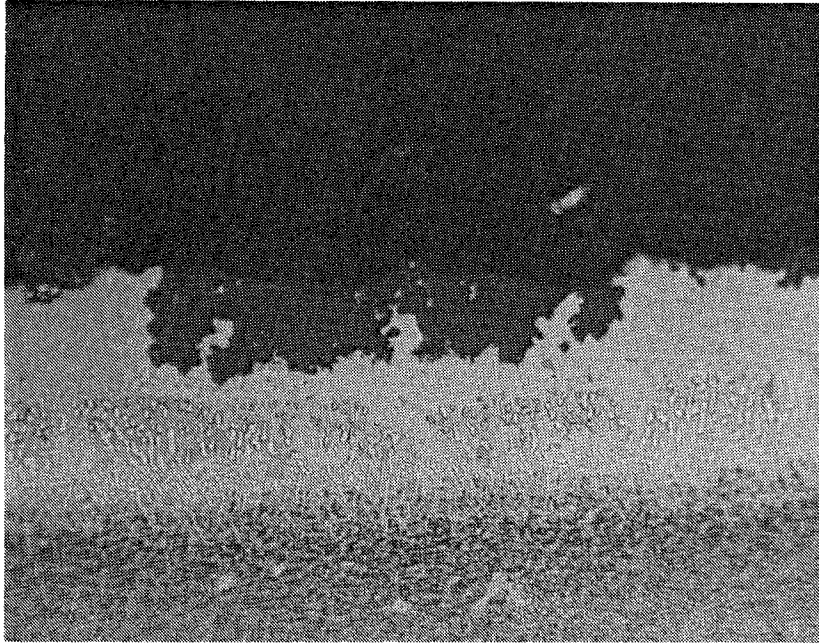
At the 20%/60-80% location, the structure was quite similar and is shown in Figure 6. Maximum attack was about 50 μm (0.002 inch), and EMP analysis did provide evidence for sulfur, although weak, in the small particles within the residual coating barely visible in the photomicrograph. EDAX analysis of the scale showed considerable Ca as well as Al.

The above information is summarized in Table II. The listed temperatures refer to the blade surface at the location of maximum degradation; these must be regarded as approximate for two reasons: (1) the observed distress occurs over some distance along the surface, and (2) assigned temperatures are calculated and will, in general, change somewhat as the turbine ages.

The depletion zone which develops in an alloy such as René 80 is largely a loss of aluminum which manifests itself in the disappearance of the γ' phase. This was confirmed by EDAX analysis of several of the etched metallographic mounts, as well as the presence of subsurface sulfides. However, sulfides were found in the most distressed areas in only two of these blades. In the other two blades, although sulfides were found, the most severely distressed regions did not contain any detectable sulfides.

This points up a primary difficulty in interpretation, namely that contaminant ingestion occurs only when the aircraft is near ground level; during most of a mission, simple oxidation is probably to be expected. Table II exhibits one attempt to relate the observed degradation to operating history. Using flight pattern information and the criterion given in footnote (c), total times were estimated during which each engine might be considered vulnerable to contaminant ingestion: total hours near coastal airports, and total hours (in life of engine). One way of displaying this information is shown in Figure 7. There is in fact a trend showing increased hot corrosion with increased low altitude operating time; the curvature is upwards as is not unreasonable, i.e., above about 60-80 μm (nominal original coating thickness) the substrate alloy is exposed. However, with this limited sampling, it is not possible to make inferences concerning coastal versus total low altitude exposure.

Alternatively, the trends shown in Figure 7 may reflect total times at takeoff/thrust reverse (higher) temperatures, a parameter which is clearly related to total hours at low altitudes. This is shown in rate units (Figure



Etched

500X

Figure 6. Photomicrograph at the 20%/70% Location on the Concave Airfoil of a Stage 1 High Pressure Turbine Blade from a Western Europe CF6-50 Engine. A Few of the Subsurface Particles Within the Residual Coating were Confirmed as Sulfides by EMP Examination.

Table II. Summary of Metallographic Evaluations and Operating History for Four Stage 1 High Pressure Turbine Blades from CF6-50 Engines.

Carrier Base	Total Hours	No. of Cycles	Hours/ Cycle	Hours <2000' El. (a)		Location (b)	Temp. ° C (Est.)		Maximum Depth of Attack, μm (c)		Type 1 Sulfides
				Near Coastal AP	Total		Max.	Avg.	Total	per 1000 Hours	
Southern Asia	2694	1560	1.72	350	520	40/70-90	1090	820	100	37	Yes
						60/LE	980	740	50	20	Yes
						60/80	1090	820	200	75	No
Northern Africa	4641	2169	2.14	560	720	50/60-90	1090	820	230	50	Yes
						80/50-80	1090	820	230	50	Yes
Southern Africa	1830	1471	1.24	240	490	10/LE	910	680	50	27	Yes
						10/60-80	1020	760	75	40	Yes
						60/90	1090	820	180	100	No
Western Europe	2625	610	4.30	95	200	10/60-80	1020	760	45	17	No
						20/60-80	1020	760	50	20	Yes

(a) Estimated, using 20 minutes as approximate total time per cycle; gate to takeoff, climb to 2000 ft.

(b) % Span/% Chord.

(c) Including depletion zone.

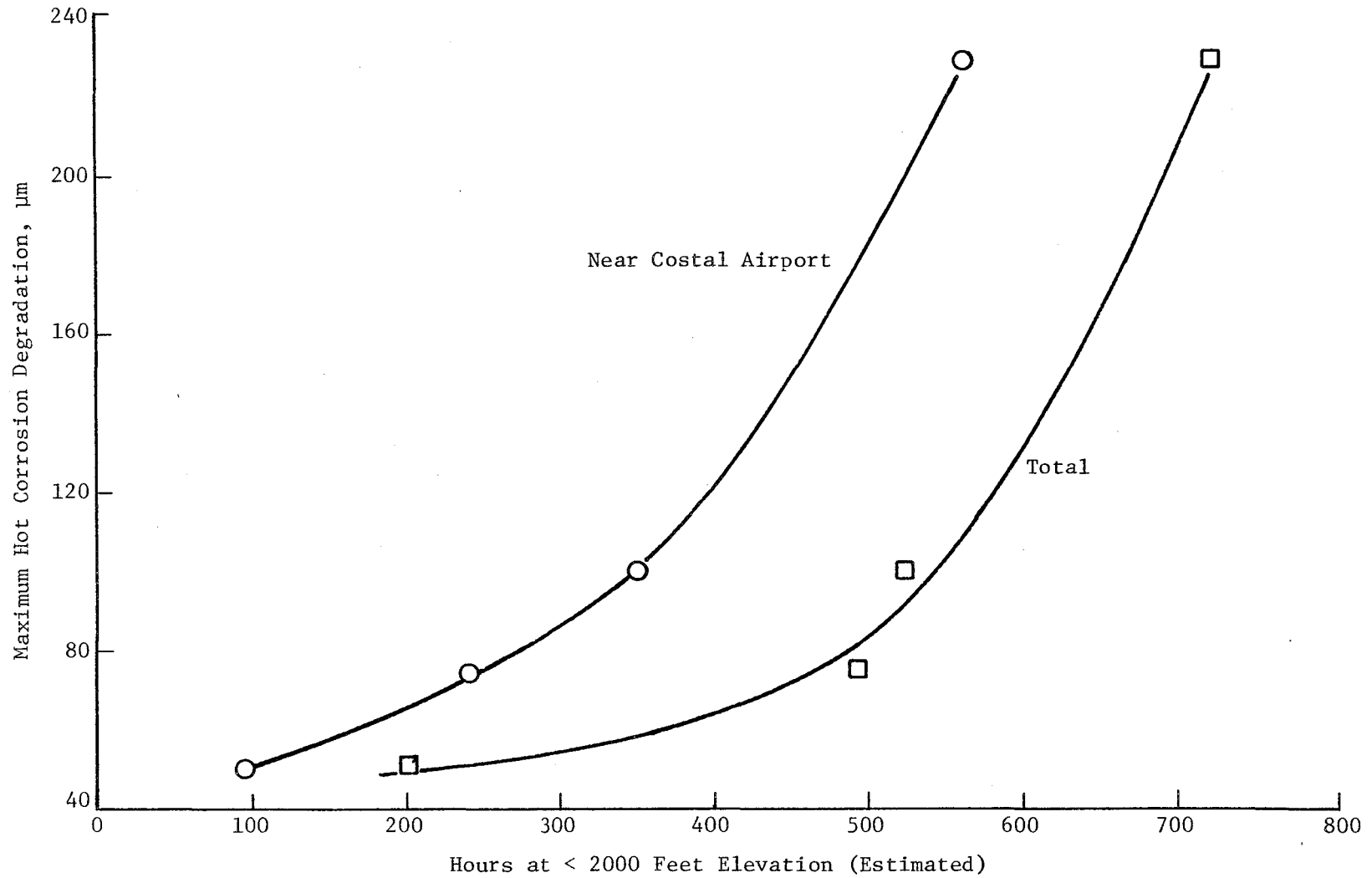


Figure 7. Hot Corrosion Degradation for CF6-50 Stage 1 High Pressure Turbine Blades as Function of Time at Less Than 2000 Feet Elevation.

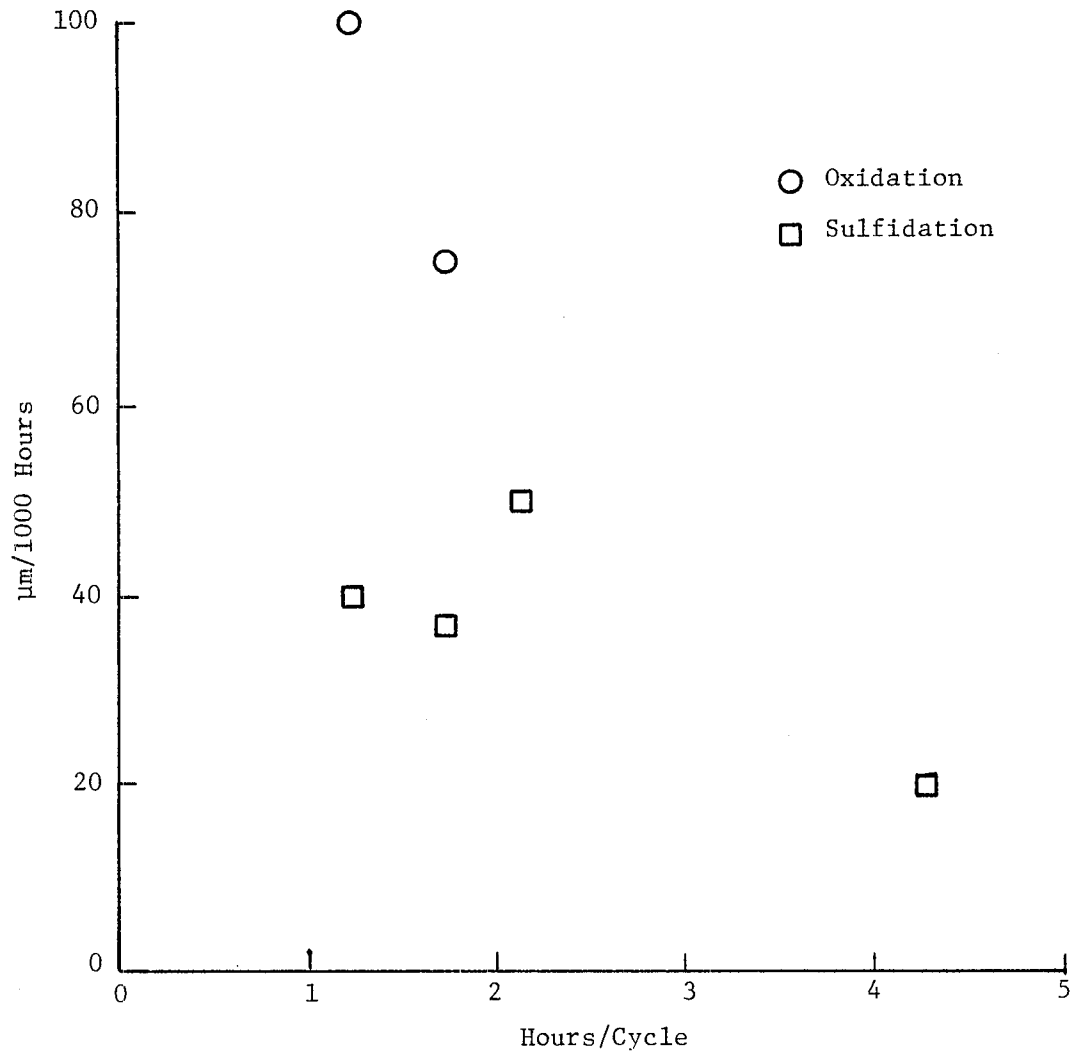


Figure 8. Maximum Degradation Rates for CF6-50 Stage 1 High Pressure Turbine Blades as Function of Average Mission Duration.

8). The highest rates correspond to the shortest lengths of mission for which the percentage of time at higher temperatures (or lower altitude) is greater.

These are in fact interesting and instructive exercises, but remembering that the present four blades were selected as rather extreme examples, the actual amounts of corrosive degradation (Table II and Figures 7 and 8) do not define possible curves which other examples should be expected to match. Further, since oxidation and hot corrosion rates are competitive in some instances (Table II), the cause and effect relationships with respect to environmental degradation have that additional degree of complexity.

The trend shown in Figure 8 has, however, long been recognized in a qualitative sense and hopefully continued quantitative evaluations coupled with compilations of operating histories will lead to a reliable choice between various possible correlations such as those depicted in Figures 7 and 8.

With respect to the ultimate blending of this type of information with the burner rig studies to be performed in this program and application to life prediction methodology, it should be pointed out that previous burner rig corrosion tests at General Electric with very low salt ingestion levels (at barely the threshold level for salt condensation) produced conventional Type 1 sulfidation but at rates indistinguishable from oxidation. This may relate to the results in Table II, namely that oxidation and corrosion rates may vary relative to each other, depending on the circumstances. Possibly as well there are implications with respect to the design of somewhat more complex burner rig tests; i.e., temperature cycling, intermittent salt ingestion, etc.

Remaining to discuss are evaluations of two additional components and a variety of chemical and structural identifications on scales and deposits.

South America

This Stage 1 LPT vane was selected as an extreme example of severe hot corrosion. In the LPT where the temperatures are considerably lower than in the high pressure turbine, salt deposits are more likely to accumulate and

may not evaporate during high altitude operation in subsequent missions. This is particularly true of the relatively stagnant internal cavities. In the present example, inside-out corrosion from such deposits was so severe as to penetrate through the airfoil in some locations. The present evaluation, however, is confined to external surfaces which can be unambiguously related to operating parameters and to degradation of other components elsewhere in the engine. Photographs of two adjacent vanes are shown in Figure 9.

Metallographic sections were prepared for the 80% span of both of these vanes (measured outwards from the inner flange). Type 1 sulfidation was prevalent and extensive at many locations on both concave and convex surfaces - unlike high pressure turbine blades where very little degradation occurs on the convex surface. Maximum attack was about 400 μm (0.016 inch), including depletion zone, at several locations on both sections examined. There was no evidence of Type 2 corrosion. Typical microstructures are shown in Figure 10, and EMP evidence for subsurface sulfides in Figure 11. The scale is rich in Ni and Cr, low in Al, and the γ' -free region of the René 77 is typically depressed in Cr, Al, and Ti.

Pitch line temperatures for this component are about 890° C at takeoff and 775° C at cruise. At the 80% span, temperatures are perhaps 25-50° C lower. It is interesting to note that the sulfidation rate of about 25) $\mu\text{m}/1000$ hours from this low pressure turbine vane - 5.06 hours/cycle - falls in the same low range as that for the Western Europe long mission HPT blade (see Table II). Although conclusions cannot be drawn from a single example, this observation does include the counteracting effects of lower temperature (lower rate) and greater salt retention time (greater effective rate). Also, it should be pointed out that the low pressure turbine vane is not coated which makes it more vulnerable to corrosion, at least for Type 1 attack.

U.S. Military

This Stage 1 high pressure turbine blade is the least distressed in the present study. Detailed examination of 60% and 80% sections failed to reveal sulfides. The maximum distress was observed to be about 25 μm (0.001 inch) at the 80%/80% location for which the maximum operating temperature is estimated

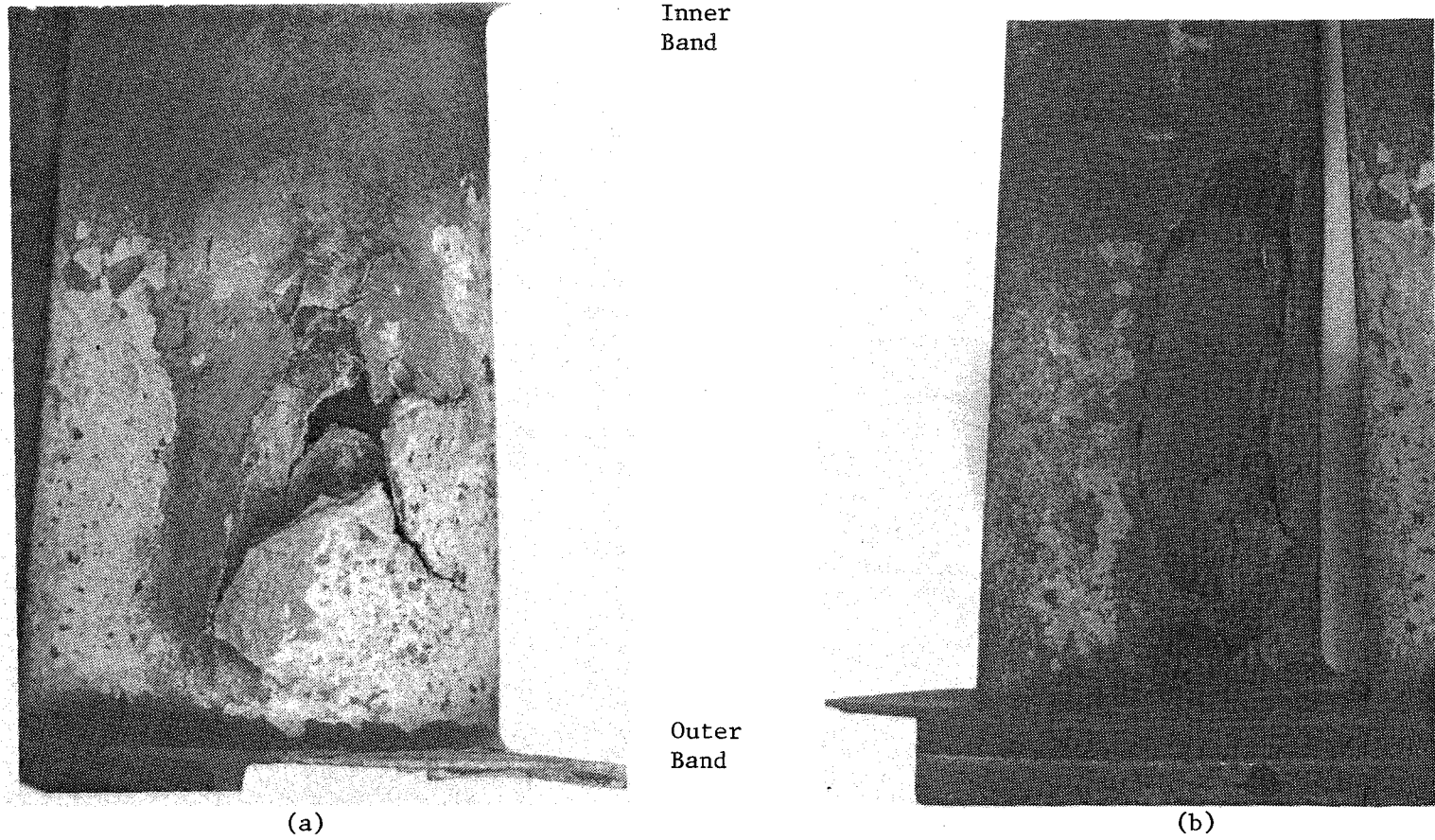
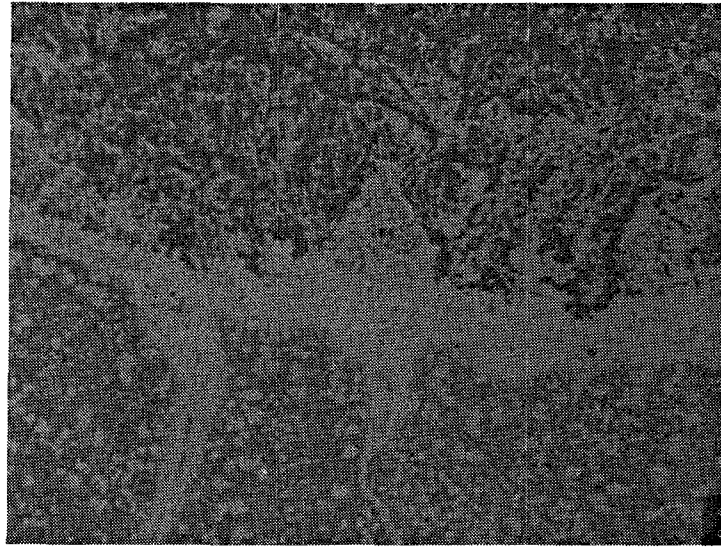
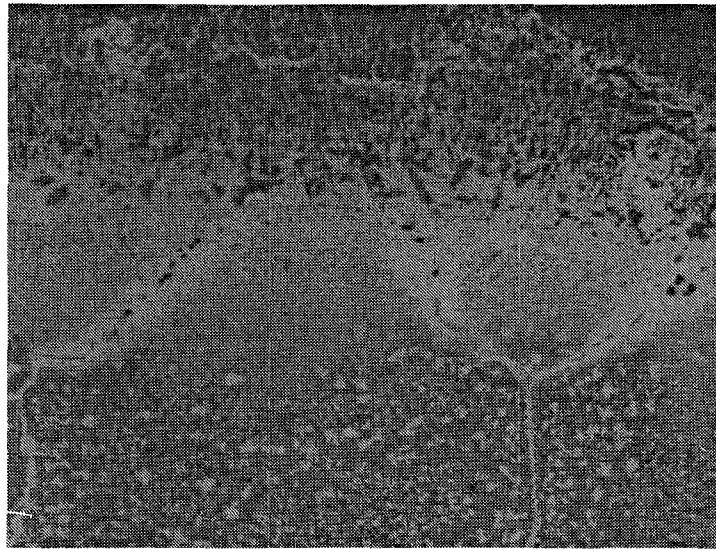


Figure 9. Stage 1 Low Pressure Turbine Vanes from a South America CF6-50 Engine, 15,155 hours of Service.



Etched

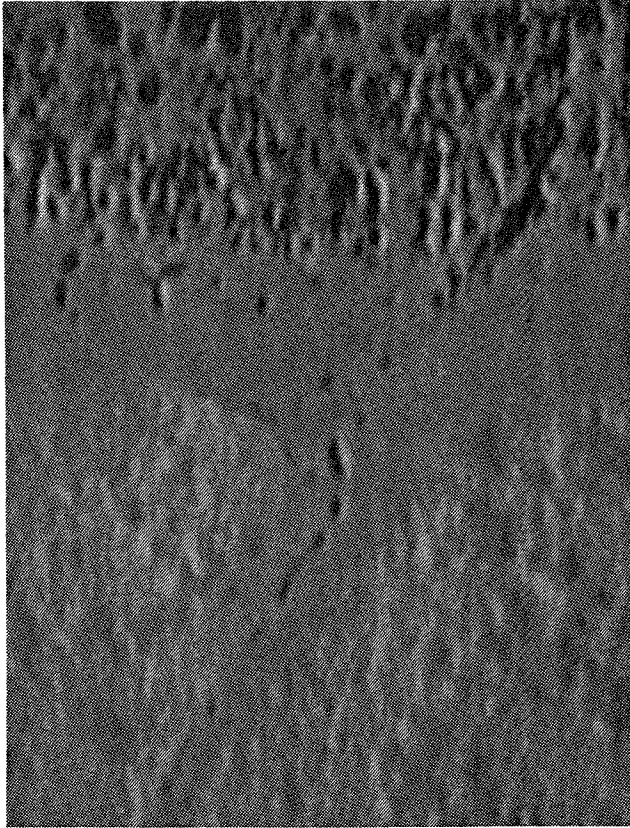
500X



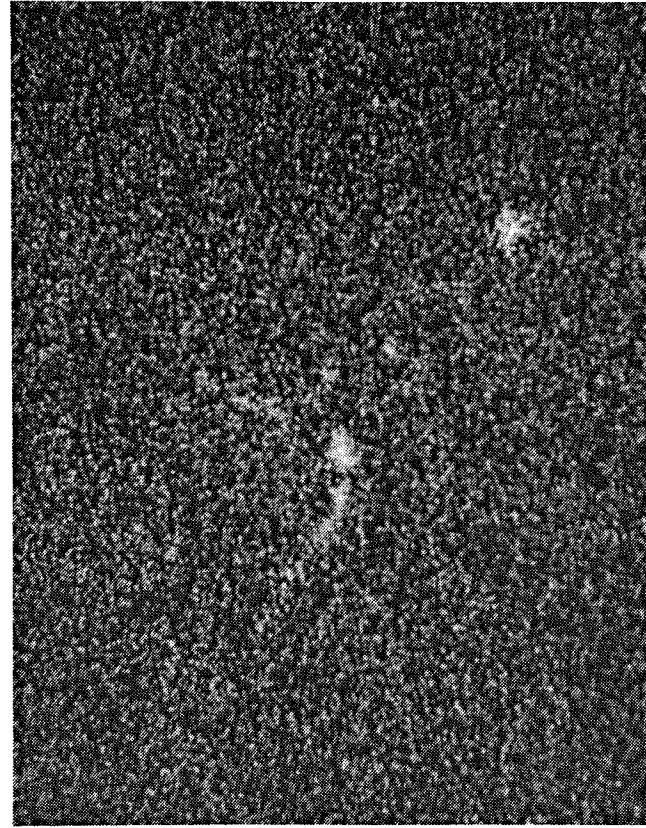
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500X

Figure 10. Photomicrographs Showing Type/Sulfidation on a Stage 1 Low Pressure Turbine Vane from a South American CF6-50 Engine.



BSE



Sulfur

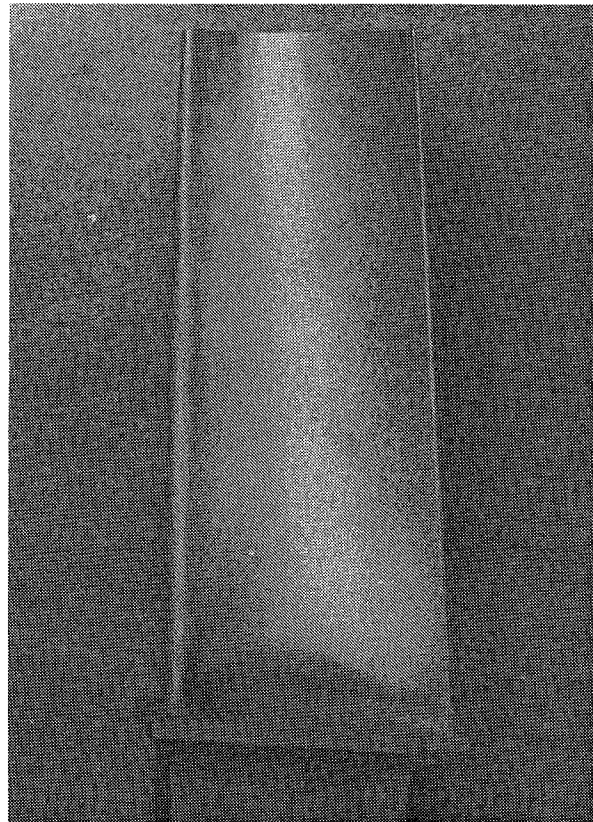
Figure 11. EMP Confirmation of Sulfides in the Gray Subsurface Particles Shown in Figure 10. Stage 1 Low Pressure Turbine Vane from a South America CF6-50 Engine (2000X).

as about 870° C. At this location, where the coating is less than half consumed, the scale is predominantly aluminum oxide, and contains small amounts of Ca and Fe derived from the environment. A photograph of this blade and a typical microstructure are shown in Figure 12. (Sulfidation has occasionally been identified on other J79 HPT blades. Thus the one depicted in Figure 12 is not an extreme example as is the case for the other five components in this survey.)

Structural and Chemical Analyses - X-ray diffraction identification of CaSO_4 in scales has been cited in several instances. These patterns also invariably reveal NiO and a variety of spinel structures which cannot be explicitly identified. These compounds are considered inert and not involved in the hot corrosion process. It should be pointed out that in early stages of life (or relatively benign operating conditions) the scale which forms is nominally pure (and protective) aluminum oxide as described above for the J79 turbine blade. As the source of Al for reformation of this oxide is depleted - either with time or with more severe environmental parameters - the scale which forms becomes mixed with NiO and spinels. Such scales are far less protective, and once they have formed, degradation proceeds at faster rates. The scales analyzed by X-ray diffraction in this study fall in the latter category.

Analyses of water soluble constituents in deposits on the three duplicate parts identified in Table I are summarized in Table III. For one of these, results for inside surfaces are given. These deposits were removed ultrasonically in distilled water after scrubbing all outer surfaces.

Water-soluble Co and Ni (as sulfates) are formed in Type 2 hot corrosion which occurs at temperatures (e.g., $\leq 750^\circ \text{C}$) appreciably lower than the range in which Type 1 hot corrosion occurs. The absence of these species in deposits on the two HPT blades is consistent with the operating temperatures involved and the Type 1 microstructures observed in the metallographic evaluations. On the other hand, significant amounts of these species were found on the surface of the LPT vane which operates at lower temperatures. Apparently, the operating temperatures for this component are borderline, i.e., some chemical evidence for the possibility of Type 2 hot corrosion, yet microstructural evidence



Etched

200X

Figure 12. Stage 1 High Pressure Turbine Blade from a U.S. Military J79 Showing Degradation of the Codep Coating at the 80% Location on the Concave Airfoil.

only for Type 1. Details of the differences in these two mechanisms are amply discussed elsewhere (References 1 and 2).

Table III. Analyses of Water Soluble Deposits on Surfaces of Turbine Components From Field Service.

Component	Surface	Milligrams					
		Na	Ca	Mg	Co	Ni	SO ₄
Southern Asia Stage 1	Concave	0.24	0.32	0.09	0.01	0.01	2.39
	Convex	0.21	0.14	0.09	0.01	0.01	1.93
HPT Blade	Inside	0.25	5.84	0.12	0.01	0.01	40.6
South America Stage 1	Concave (2)	0.49	0.22	0.18	0.01	0.04	2.65
	LPT Vane Convex (2)	0.48	0.28	0.19	0.04	0.07	3.21
U.S. Military Stage T	Concave	0.09	0.02	0.02	0.01	0.01	0.94
	HPT Blade Convex	0.23	0.04	0.06	0.01	0.01	1.12

The other species listed in Table III are derived from the environment, specifically on the runway and at low elevations during takeoff and landing. The roughly equal amounts of these species on the concave and convex surfaces of the two HPT blades are unlike those found in marine engines (i.e., the convex surface generally has far less) and supports the view that the contamination observed represents that ingested during final landing. The larger amounts found internally are to be expected, i.e., accumulate during the use period of the engine. Calcium and magnesium compounds are known to be inert in hot corrosion and in fact act as inhibitors. The primary culprit is Na₂SO₄ for which there is ample evidence. On average, the metallic species listed in Table III account for about two-thirds of the measured sulfate, on a molar basis; possibly some other metals are present in the water soluble deposits for which analyses were not made. Qualitative analyses for chloride were made but no significant amounts detected. It should also be mentioned that the amounts of Na present were too small to be detected by EDAX in the metallographic sections discussed earlier, and which were prepared by nonaqueous procedures.

In summary, Type 1 hot corrosion was clearly identified in five of the six components evaluated in this study. Preliminary attempts have been made to correlate the extent of such degradation with operating histories. The observed microstructures will be used for comparison with those observed in the following burner rig tests which are designed to induce Type 1 hot corrosion.

TASK II - BASELINE HOT CORROSION TESTS

All hot corrosion testing in this program is being performed at TRW, Inc., Cleveland, Ohio, in high velocity (0.3 Mach) burner rigs operating at one atmosphere. In this Task II, data are being obtained on uncoated and coated alloys to be compared later (Task IV) with data obtained under the same test conditions, and on the same materials, but with specimens previously subjected to a variety of aging conditions (Task III).

Materials for this Task II burner rig hot corrosion test include Udimet 700 and René 80 in the uncoated condition and with the following coatings:

RT21 aluminide (Chromalloy American Corp.)

Codep aluminide (General Electric Co.)

Ni23Co18Cr12Al0.3Y applied by vacuum (low pressure) plasma spraying.

Nominal major constituents of the substrate alloys in weight percent are:

Udimet 700 - 14.5 Cr, 4.3 Al, 3.4 Ti, 15.0 Co, 4.2 Mo, balance Ni

René 80 - 14.0 Cr, 3.0 Al, 5.0 Ti, 9.5 Co, 4.0 Mo, 4.0 W, balance Ni

Heat treatment sequences for these alloys are:

U700

1. 1163° C (2125° F)/4 hours in vacuum/fast cool
2. 1080° C (1975° F)/4 hours in vacuum/fast cool
3. 927° C (1700° F)/24 hours in vacuum/fast cool
4. 760° C (1400° F)/16 hours in vacuum/fast cool

René 80

1. 1093° C (2000° F)/2 hours in vacuum/helium quench
2. 1052° C (1925° F)/4 hours in vacuum/furnace cool to 649° C (1200° F) in 20 minutes/air quench
3. 843° C (1550° F)/4 hours in vacuum/air quench

Alloys were cast per Figure 13 (slightly oversize) and specimens individually stamped with a serial number on the bottom end. The two series were coded "X" for U700 and "L" for René 80. All specimens were given the first heat treatment and then centerless ground to a 32 AA finish. Top ends were also machined to a 32 AA finish and the corner slightly chamfered to minimize the possibility of poor coating integrity.

At this point, coatings were applied to a sufficient quantity of specimens for the entire program. For the two aluminides, the coating process fulfilled the function of the second step in the heat treatment; remaining heat treatments were performed and the specimens were then ready for test. Diameters of each specimen were measured before and after coating.

The plasma-sprayed coatings were applied to a slightly greater thickness than desired. Specimens were given the second heat treatment which served also to provide a diffusion bond, and subsequent aging as well. The excess coating was then machined off to give the desired nominal thickness of 0.10 mm (4.0 mils), surface finish 32 AA.

Photographs of typical specimens of all six coating/substrate systems are shown in Figure 14. Photomicrographs are shown in Figure 15; these structures are normal and the two plasma-sprayed coatings exhibit excellent density. Thicknesses of these coatings, including diffusion zone, are:

Codep/René 80	50 μ m
Codep/U700	84
RT21/René 80	81
RT21/U700	79
NiCoCrAlY/René 80	98
NiCoCrAlY/U700	98

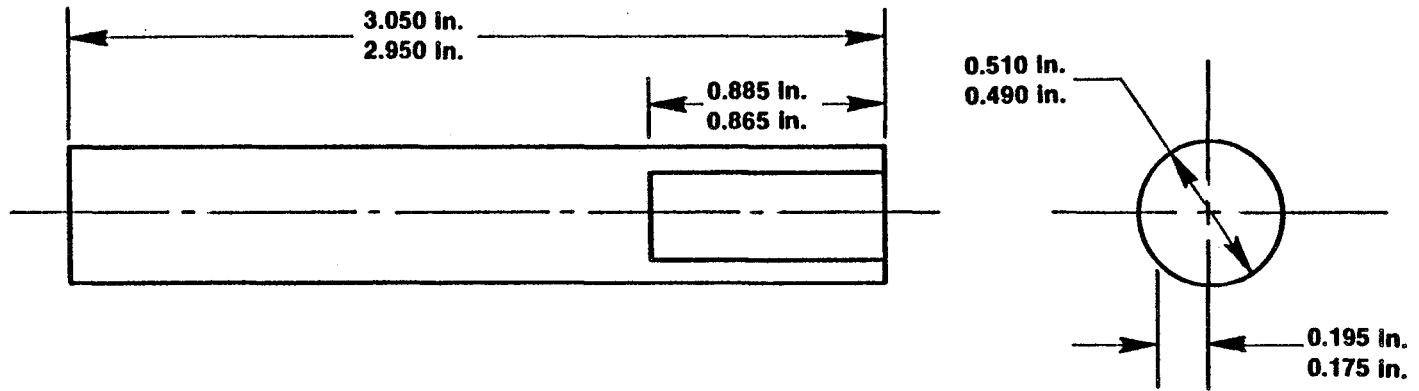
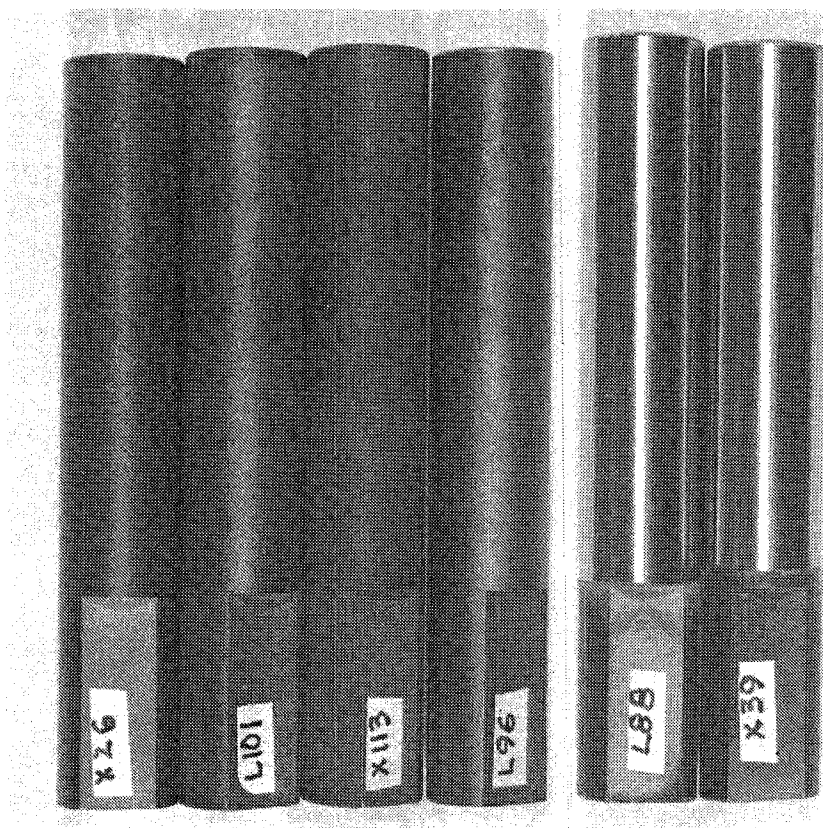


Figure 13. Hot Corrosion Test Specimen.



Coating:	Codep	RT21	RT21	Codep	NiCoCrAlY
Substrate:	U700	René 80	U700	René 80	René 80 U700

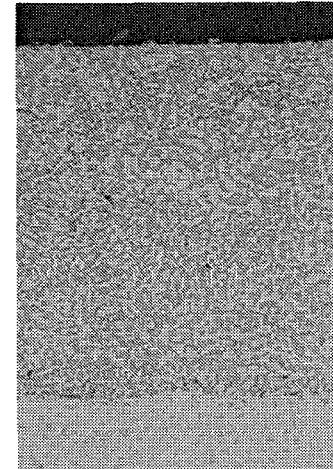
Figure 14. As-Coated Burner Rig Specimens of Codep, RT21, and NiCoCrAlY to U700 and to René 80 (1.4X).



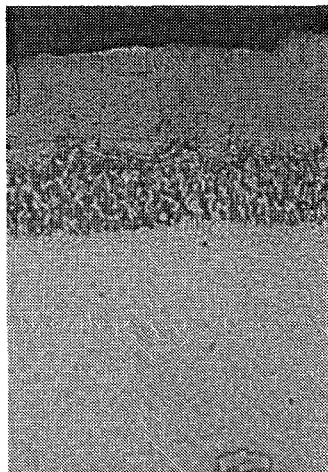
Codep
Coating
on U700



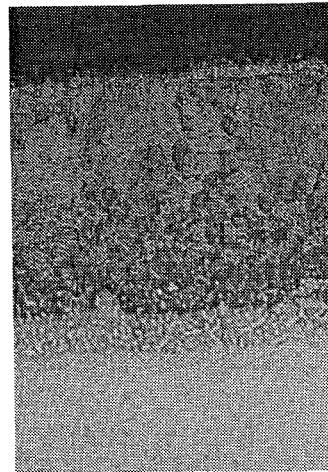
RT21
Coating
on U700



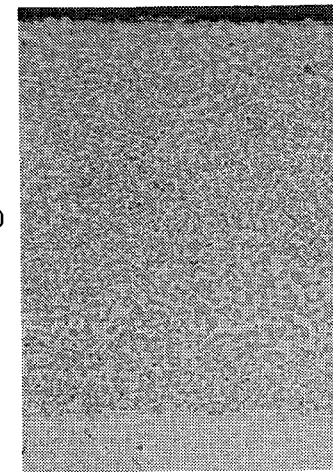
NiCoCrAlY
Coating
on U700



Codep
Coating
on René 80



RT21
Coating
on René 80



NiCoCrAlY
Coating
on René 80

Figure 15. Microstructures of Codep, RT21, and Plasma-Sprayed NiCoCrAlY Coatings on U700 and René 80 (500X).

The four aluminide-coated systems were analyzed by quantitative EMP in a series of five locations normal to the surface; three in the additive layer, one each in the diffusion zone and the substrate alloy. The results for the four systems were reasonably consistent in several respects.

1. Cr, Ti, and Mo are somewhat enriched in the diffusion zone relative to concentrations in the substrate. These elements are present in the additive layer, rather uniform across the thickness, averaging approximately 4%, 1%, and 1%, respectively.
2. Cobalt is present in the diffusion zone at about the same concentration as in the substrate, and is roughly constant across the additive layer at about two-thirds the level in the substrate.

Concentrations of Ni and Al may be considered in terms of compound formation of β NiAl. Noting that CoAl also can form, and that the concentration of Co in the additive layer is substantial, atomic ratios of Co+Ni/Al were calculated. This ratio averaged approximately 0.80 for the four systems at the outer surface and increased to approximately 0.95 near the diffusion zone.

The above is recorded as baseline information and will be available for comparison with EMP measurements on tested specimens.

Burner rigs with rotating specimen holders in test are shown in Figure 16. Testing was performed under the conditions listed in Table IV. JP5 fuel (essentially the same as Jet A) was used. The sulfur content was 0.035% adjusted by the addition of ditertiary butyl sulfide to the as-received fuel. Burner pressure and the throat diameter requirements were achieved by fitting the burners with specially designed exhaust nozzles shown in Figure 17. These are the same as used in the burner rigs at NASA-LeRC, Cleveland, Ohio.

The test cycle consisted of 1 hour at temperature followed by a minimum of 6 minutes of forced cooling to ambient temperature. Every effort was made to adhere to this test cycle. However, sometimes because of rig malfunction, a cycle was interrupted resulting in (1) less than 1 hour at temperature and (2) ambient air cooling rather than a forced blast.

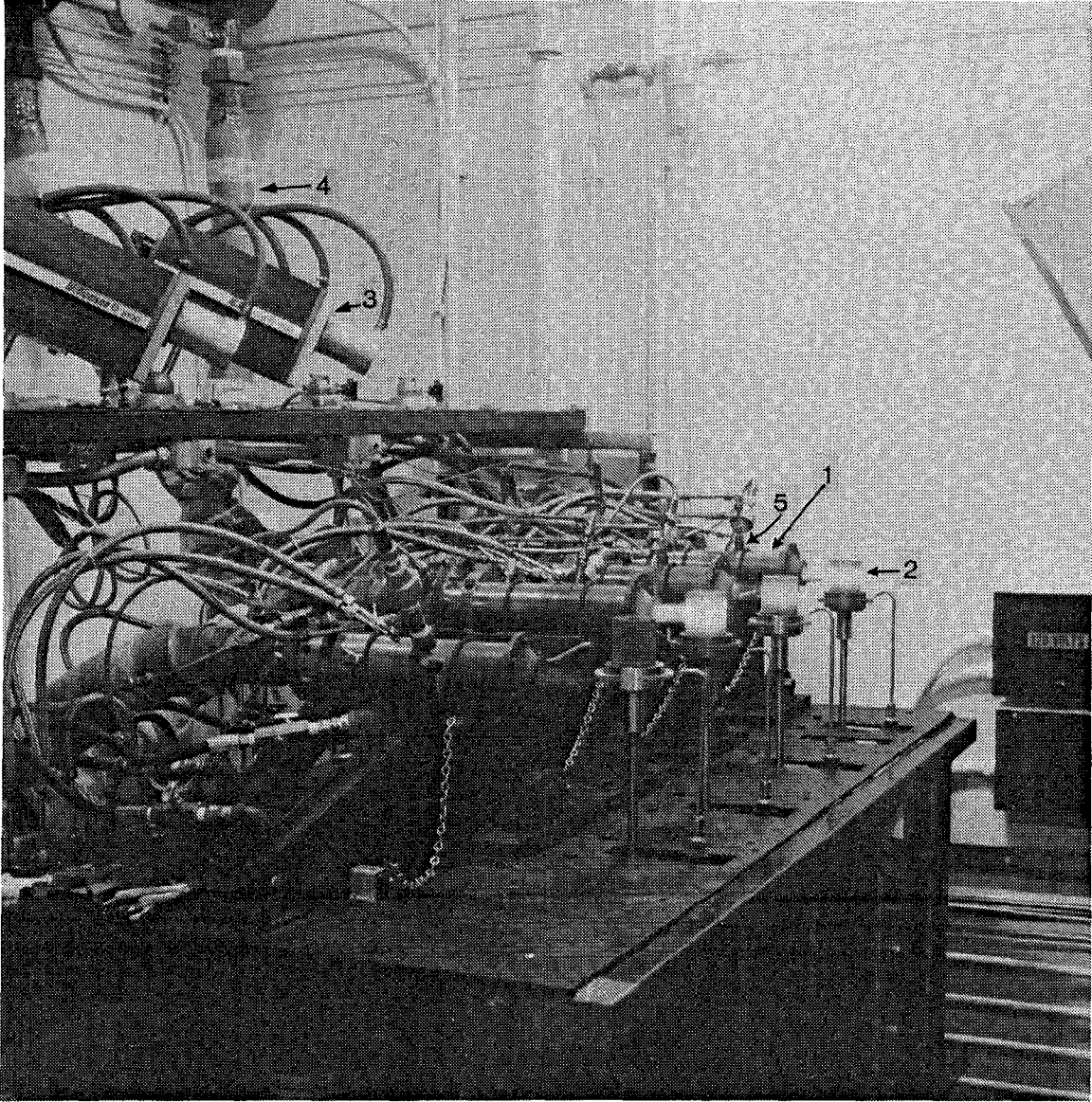


Figure 16. Corrosion Rig Setup.

1. Burner
2. Specimens on Holder Rotating at 600 rpm
3. Optical Pyrometer
4. Preheated Combustion Air
5. Salt Solution Spray Nozzle

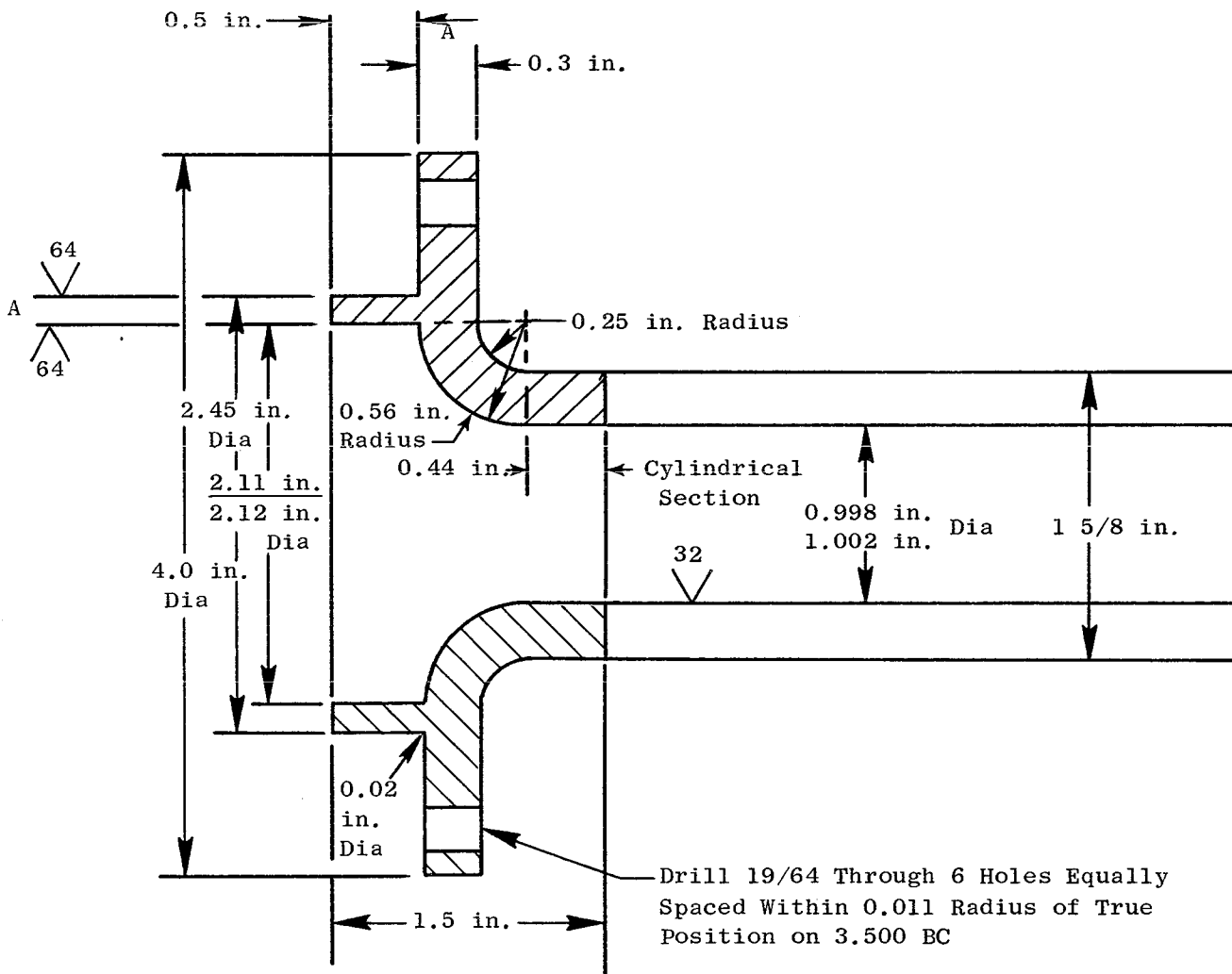


Figure 17. Thin Wall Convergent Burner Nozzle (MAR-M-302).

Table IV. Burner Rig Operating Conditions.

Specimen Temperature - $900^{\circ}\text{C} \pm 9^{\circ}\text{C}$

Test Cycle - 1 hour at temperature followed by 6 minutes of forced air cooling

Sodium Concentration - 0.5 ppm sodium (+10%) in the combustion gases introduced as aqueous NaCl

Combustion Air Preheat Temperature - $232^{\circ}\text{C} \pm 10^{\circ}\text{C}$

Specimens - Eight positioned equally on a 4.2 cm (1.64 inch) diameter circle of a holder rotating at 600 rpm

Burner Nozzle Throat Diameter - 2.54 cm (1.0 inch)

Burner Pressure - 1.0 psig

Nozzle Throat to Nearest Specimen - 4.45 cm (1.75 inch)

Pertinent burner rig operating parameters are recorded during the tests at least twice in each test interval (nominally 20 cycles between inspections). Typical flow rates for combustion air and fuel are 32-34 standard cubic feet per minute and 1.3-1.5 gallons per hour respectively. In order to ensure continued reliability of the optical pyrometers used for specimen temperature control, the specimen temperature was independently measured with a hand-held optical pyrometer. This independent temperature check was used as a rough indicator of gross deviation in calibration of the control optical pyrometer. The control pyrometers were calibrated with the optical pyrometer calibration setup at NASA-LeRC, Cleveland, Ohio. A slip-ring thermocouple arrangement attached to a specimen in a rotating holder was used in this calibration.

The sodium in the combustion air is maintained at the specified level of 0.5 ppm by adding salt-water solution in the combustion products. This solution is introduced in the combustion air in the form of an atomized spray. Figure 18 shows the salt solution spray nozzle used.

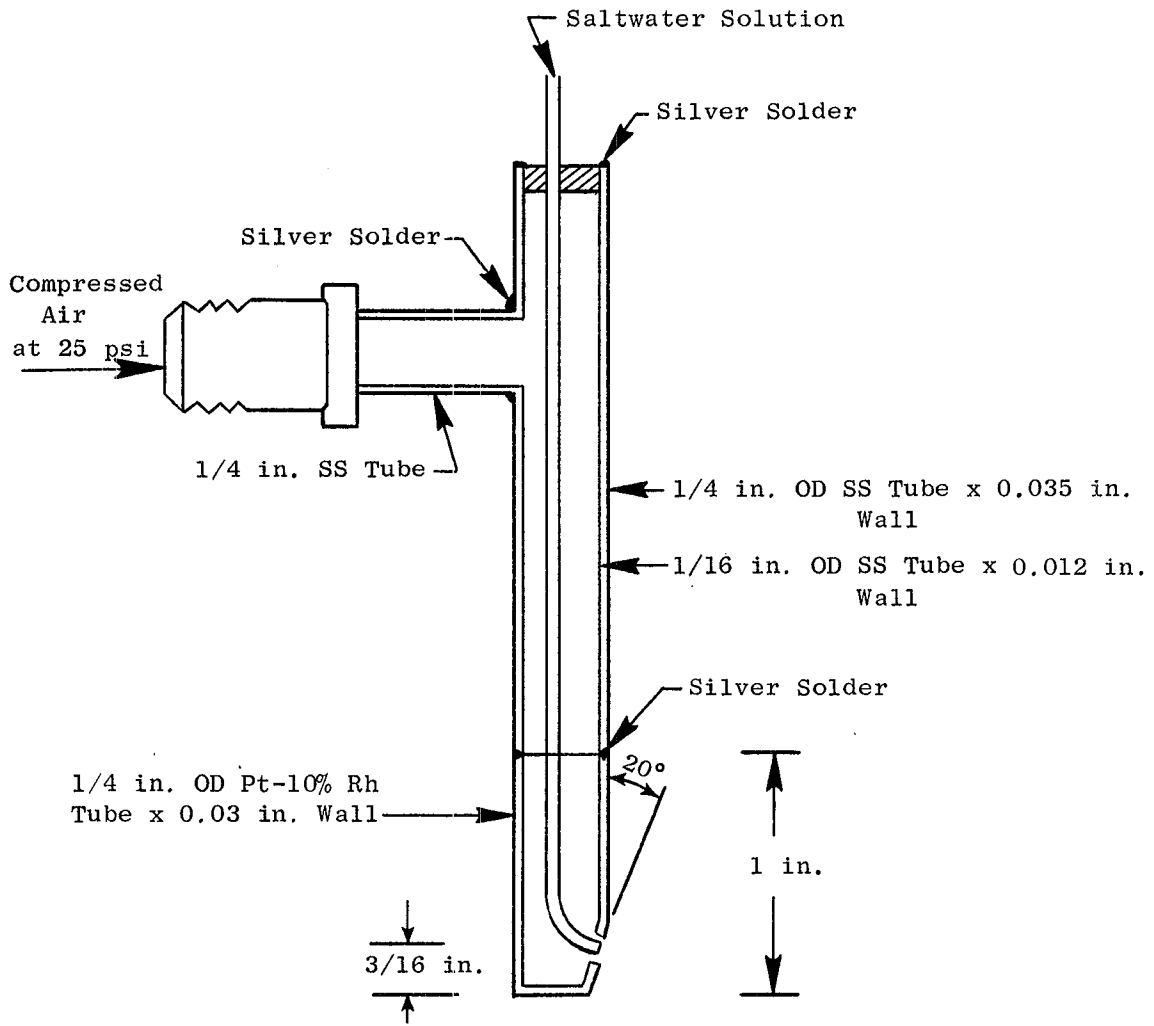


Figure 18. Salt Solution Spray Nozzle.

The test plan includes duplicate specimens of U700 and Rene 80, both uncoated, and with RT21, Codep, or NiCoCrAlY coatings. Specimens are removed from test at approximately 20 cycle intervals for inspection (in practice about 18 to 24 cycles). When visual evidence of hot corrosion is apparent at three successive inspections, the specimen is removed. The visual appearance of all specimens is documented photographically at each inspection. The plan also includes the testing of single specimens of each material for time periods of 100, 300, and 500 hours, if these time periods do not exceed 2/3 the time the original specimens of the same material were exposed. Thus, for a material which is not removed until at least 750 hours for visual reasons, the test will provide evaluation of the extent of corrosion at four different time intervals.

Posttest evaluation of specimens is performed by the traditional destructive techniques used in Task I. In addition, this program provides an opportunity to extend the recent pioneering studies at NASA-LeRC in which the progress of hot corrosion (or oxidation) is monitored, nondestructively, by the measurement at each inspection interval of the inductance of a coil at high frequency with the test specimens being a core (Reference 3). Under carefully controlled conditions, and noting that at high frequencies only the outer surface of the metallic core will influence the measurement, the coil inductance can respond either to a change in metallic core volume (metal converted to oxide scale) or to a change in composition of the metallic surface (formation of a depletion zone within the alloy as scale forms). In either event, results from such measurements afford high sensitivity in nondestructively following the changes which occur during environmental degradation. It is anticipated that results in this program will contribute significantly to an understanding and interpretation of such measurements.

Coil inductance measurements are made with the burner rig specimen positioned in a coil (see Figure 19) such that the center of the flame impingement zone, 2.5 cm from the top of the specimen, is in the center of the coil. Measurements are made in series mode with a multifrequency LCR meter at a frequency of 10 MHz (see Figure 20). At least two measurements are taken with each specimen core. For each measurement, the specimen is removed from the coil and replaced randomly to average the core-coil axial alignment.

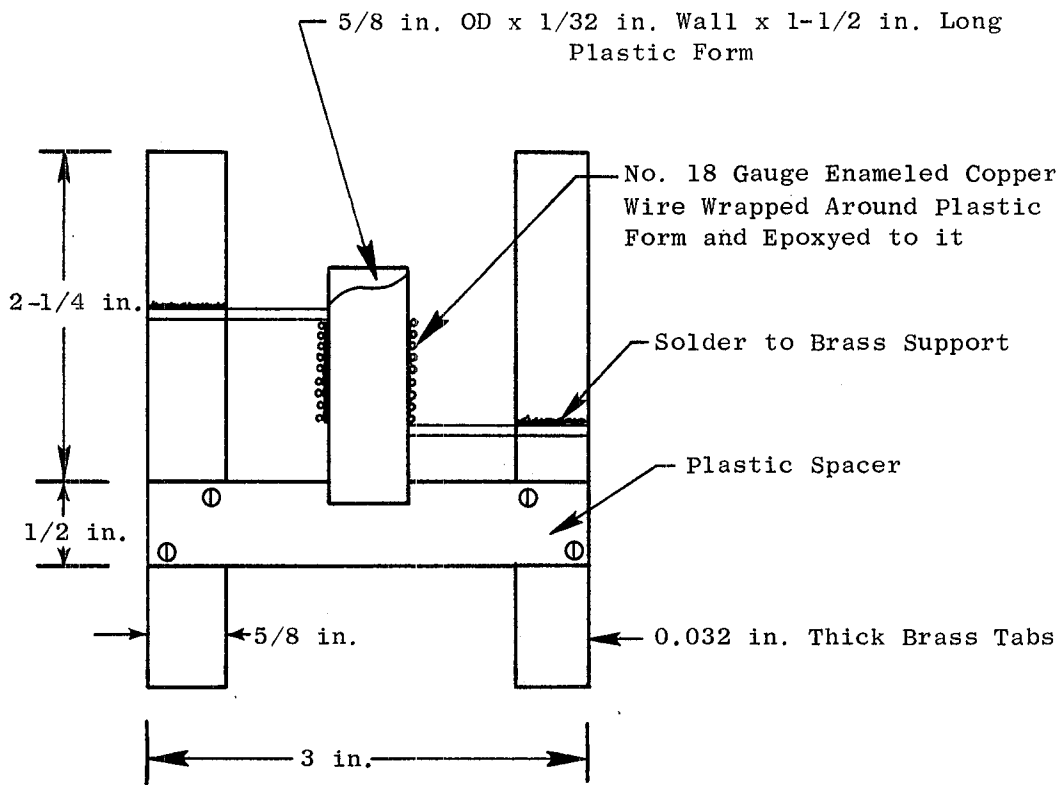


Figure 19. Inductance Coil and Support.

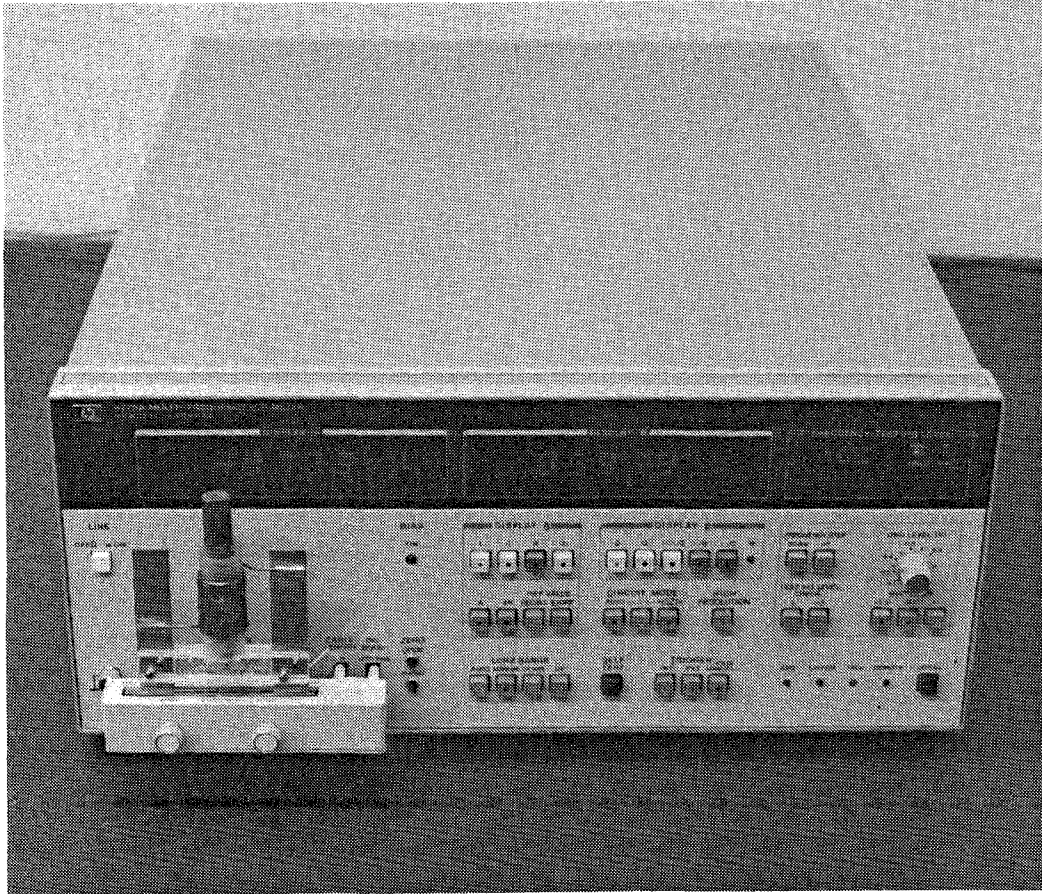


Figure 20. Inductance Rig/Coil with Specimen Core.

Results and Discussion

Duplicate specimens of the eight coating/substrate combinations initially in test are identified in Table V. The intent was that these all run full term, i.e., to the third inspection at which visual evidence of hot corrosion was apparent. The only exception is specimen L-8 (RT21/René 80) which was removed at 300 hours since a small amount of mechanical damage occurred to the coating at the top of the specimen, out of the hot zone. This specimen serves for one of the fractional exposures per the test plan described above; metallographic examination showed essentially no metal loss.

Other specimens removed from test thus far are listed in Table VI, together with total exposure times and the numbers of thermal cycles which did not receive forced air cooling. Graphs of coil inductance measurements for the six uncoated specimens are shown in Figure 21. Curves for the U700 specimens, although not identical with that reported by Deadmore under comparable test conditions (Reference 3), show strong similarities in both shape and the magnitude of changes. Curves for the René 80 specimens show smaller changes, as expected for this more corrosion resistant alloy. Typical specimen macrophotographs are shown in Figures 22 and 23.

Metallographic sections of the uncoated specimens listed in Table VI have been prepared. Although the extent of attack was not uniform around the circumference of these specimens, Type I sulfidation was apparent at essentially all locations. Typical microstructures for U700 and René 80 showing a depletion zone in the alloy, containing subsurface sulfides, are shown in Figure 24. That the nature of this degradation is similar to that observed in field experience is apparent by comparison with Figures 4 and 10.

Uncoated specimens X-19 (U700) and L-77 (René 80) were rinsed in distilled water and analyzed for total sodium; values of 7.9 and 8.3 milligrams were obtained, respectively. The rinsings showed strong evidence for sulfate but none for chloride. Thus the measured values correspond to about 1.2 and 1.3 mg of Na_2SO_4 per square centimeter, noting that the cylindrical exposed surface of the specimen is about 20 cm^2 .

Table V. Specimen Identification.

(At Start of Test)

<u>Specimen Number</u>	<u>Coating</u>	<u>Alloy</u>
<u>Rig 1</u>		
X-3	---	U700
X-8	Codep	U700
X-66	RT21	U700
X-30	NiCoCrAlY	U700
L-54	---	René 80
L-3	Codep	René 80
L-8	RT21	René 80
L-9	NiCoCrAlY	René 80
<u>Rig 2</u>		
X-21	---	U700
X-14	Codep	U700
X-73	RT21	U700
X-56	NiCoCrAlY	U700
L-77	---	René 80
L-7	Codep	René 80
L-36	RT21	René 80
L-41	NiCoCrAlY	René 80

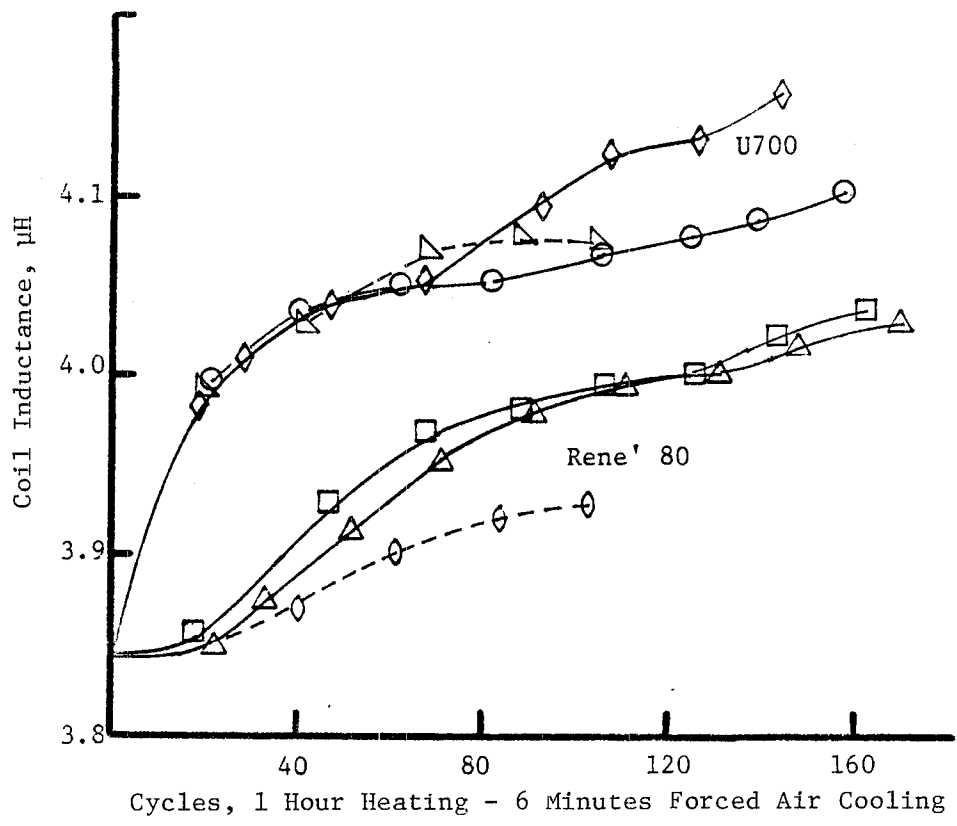


Figure 21. Uncoated Alloy Coil Inductance Changes with Hot Corrosion at 900° C, Cyclic Exposure.

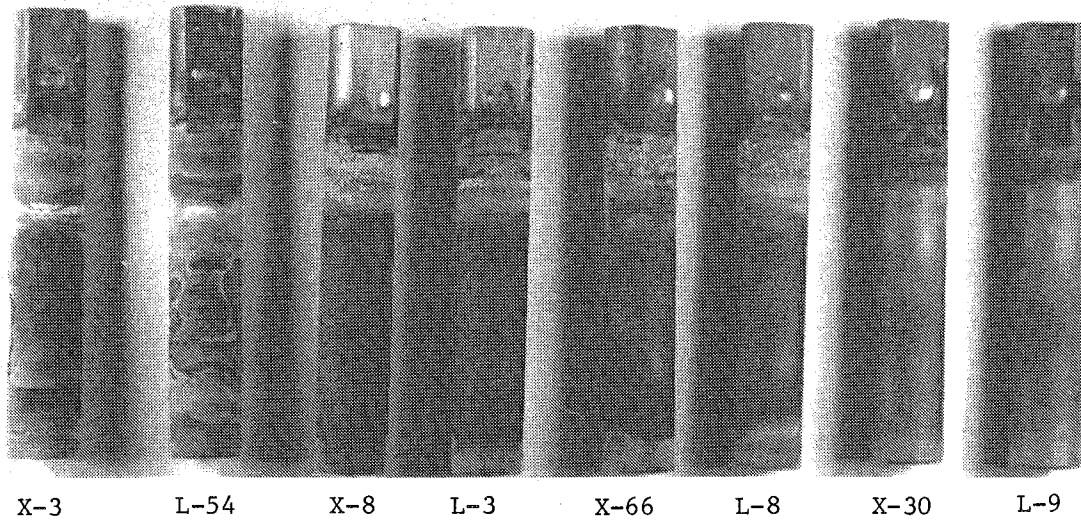
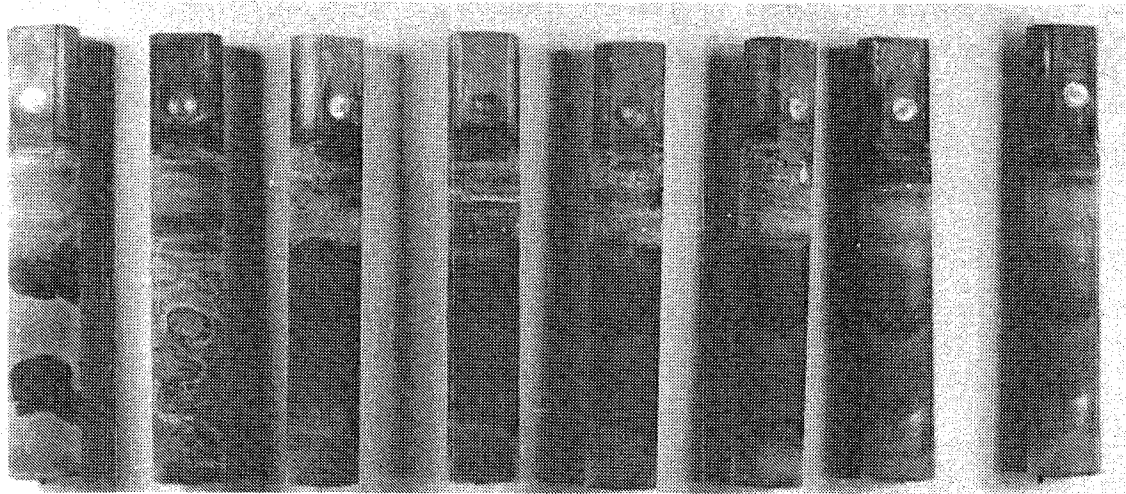
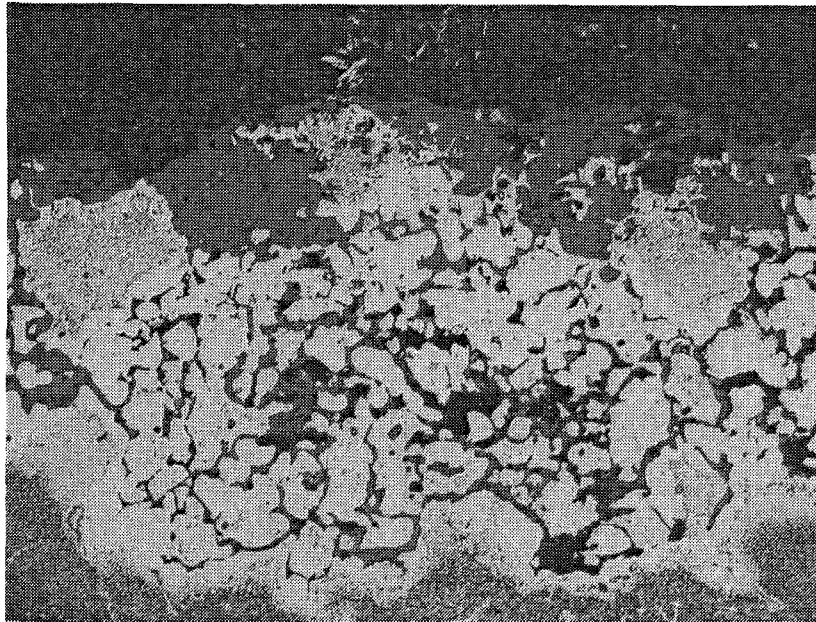


Figure 22. Uncoated U700 (X-3) Removed from Test After 142.65 hours, Rig 1.

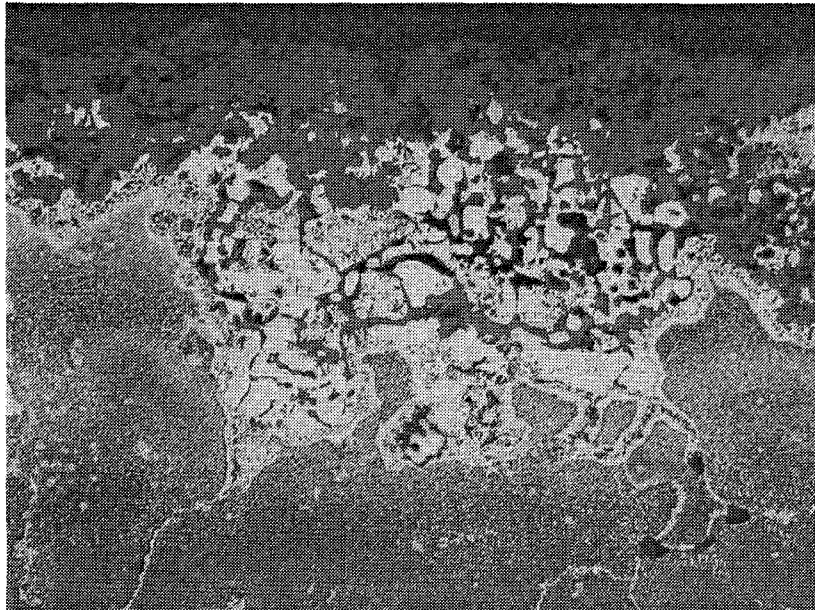


X-25 L-77 X-14 L-7 X-73 L-36 X-56 L-41

Figure 23. Uncoated René 80 (L-77) Removed from Test after 167.7 hours, Rig 2.



(a) U700, 142.65 hours



(b) René 80, 167.7 hours

Figure 24. Photomicrographs of Specimens from Burner Rig Tests. Type 1 Sulfides are Present (Etched, 200X).

Table VI. Test Information for Removed Specimens.

Specimen			Time At Removal, Hours	Number of Thermal Cycles ^(a)
Code	Alloy	Coating		
X-3	U700		142.65	141 (8)
X-19	U700		157.6	155 (4)
X-108	U700		105.5	105 (1)
L-54	René 80		162.65	161 (8)
L-77	René 80		167.7	167 (6)
L99	René 80		104.1	104 (1)
X-8	U700	Codep	424.8	420 (13)
L-7	René 80	Codep	438.9	436 (9)

(a) Normal cycle consists of 1 hour at test temperature and 6 minutes forced air cooling. Numbers in parentheses indicate cycles with less than 1 hour at temperature followed by ambient air cooling.

Selected coil inductance measurements obtained to date for all of the coated specimens are listed in Table VII. Only three have thus far been removed. Two of the Codep-coated specimens were removed for the standard criterion that visual evidence of corrosion was apparent at three successive inspections. However, coating corrosion generally follows the course of localized pitting (unlike more frontal attack of uncoated alloys) and thus the changes in coil inductance are minimal. Metallographic evaluation of these specimens needs to be performed.

Hot corrosion testing will be continued to a maximum of 1000 hours. Concurrently, the Task III aging exposures will be performed (as described earlier in this report), and these specimens subsequently hot corrosion tested in Task IV under the same conditions as in Task II.

Table VII. Change of Coil Inductance With Coated Specimens (Micro Henry).

Specimen Number	Material	0	106.6	Coil Inductance at Time in Hours				
				203.3	300.3	405.8	424.8	438.9
<u>Rig 1</u>								
X-8	Codep/U700	3.7956	3.7901	3.7980	3.8005	3.8089	3.8117(a)	
X-30	NiCoCrAlY/U700	3.7570	3.7575	3.7555	3.7601	3.7590		
X-66	RT21/U700	3.7945	3.7785	3.7813	3.7858	3.7891	Continuing	
L-3	Codep/René 80	3.8035	3.8117	3.8189	3.8220	3.8246		
L-8	RT21/René 80	3.7912	3.7750	3.7692	3.7709(a)		Continuing	
L-9	NiCoCrAlY/René 80	3.7585	3.7562	3.7550	3.7588	3.7588		
L-44	RT21/René 80	3.7920	(3.7850) (65 Hr)	(3.7870) (124.5 Hr)	Continuing			
		0	109.7	208.7	314.0	399.5		
<u>Rig 2</u>								
X-14	Codep/U700	3.7892	3.7842	3.7853	3.7947	3.7957	Continuing	
X-56	NiCoCrAlY/U700	3.7572	3.7553	3.7550	3.7576	3.7575		
X-73	RT21/U700	3.7935	3.7840	3.7758	3.7765	3.7805	Continuing	3.8282(a)
L-7	Codep/René 80	3.8030	3.8082	3.8150	3.8195	3.8219		
L-36	RT21/René 80	3.7918	3.7791	3.7710	3.7695	3.7705	Continuing	
L-41	NiCoCrAlY/René 80	3.7593	3.7572	3.7575	3.7611	3.7592		
L-49	NiCoCrAlY/René 80	3.7580	(3.7608) (105.8 Hr)	(3.7594) (167.2 Hr)	Continuing			

(a) Removed from test.

CONCLUSIONS

This study involves the determination of surface chemistry effects of oxide scale and coating composition on the hot corrosion life of selected alloys in the coated and uncoated conditions in burner rig tests. The burner rig data will be used to develop a hot corrosion life prediction model, to embrace both coating/alloy composition variables and environmental variables. Evaluation of service-run turbine components, which have suffered hot corrosion, establishes a baseline of microstructures and the extent of metal degradation as a function of operating history.

The evaluation of six service-run turbine components has been completed. In five of these, sulfidation was observed and qualitatively the extent of corrosion appears to be inversely related to the average length of mission. In some cases, however, the location of maximum distress did not exhibit the structural features of hot corrosion.

The first phase of the hot corrosion burner rig program is underway. Test conditions are: 900° C, hourly thermal cycling, 0.5 ppm sodium as NaCl in the gas stream, velocity 0.3 Mach. The alloys are Udimet 700, René 80, uncoated and with RT21, Codep, or NiCoCrAlY coatings. At the close of this reporting period, nearly 500 hours of testing had been accumulated. The two uncoated alloys were removed in the range of 140 to 170 hours and exhibited typical sulfidation, structurally similar to that observed for service-run turbine components. The aluminide coatings began visually to exhibit hot corrosion at 400+ hours; the NiCoCrAlY coatings, although somewhat discolored, continue in test with no clear evidence of hot corrosion.

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