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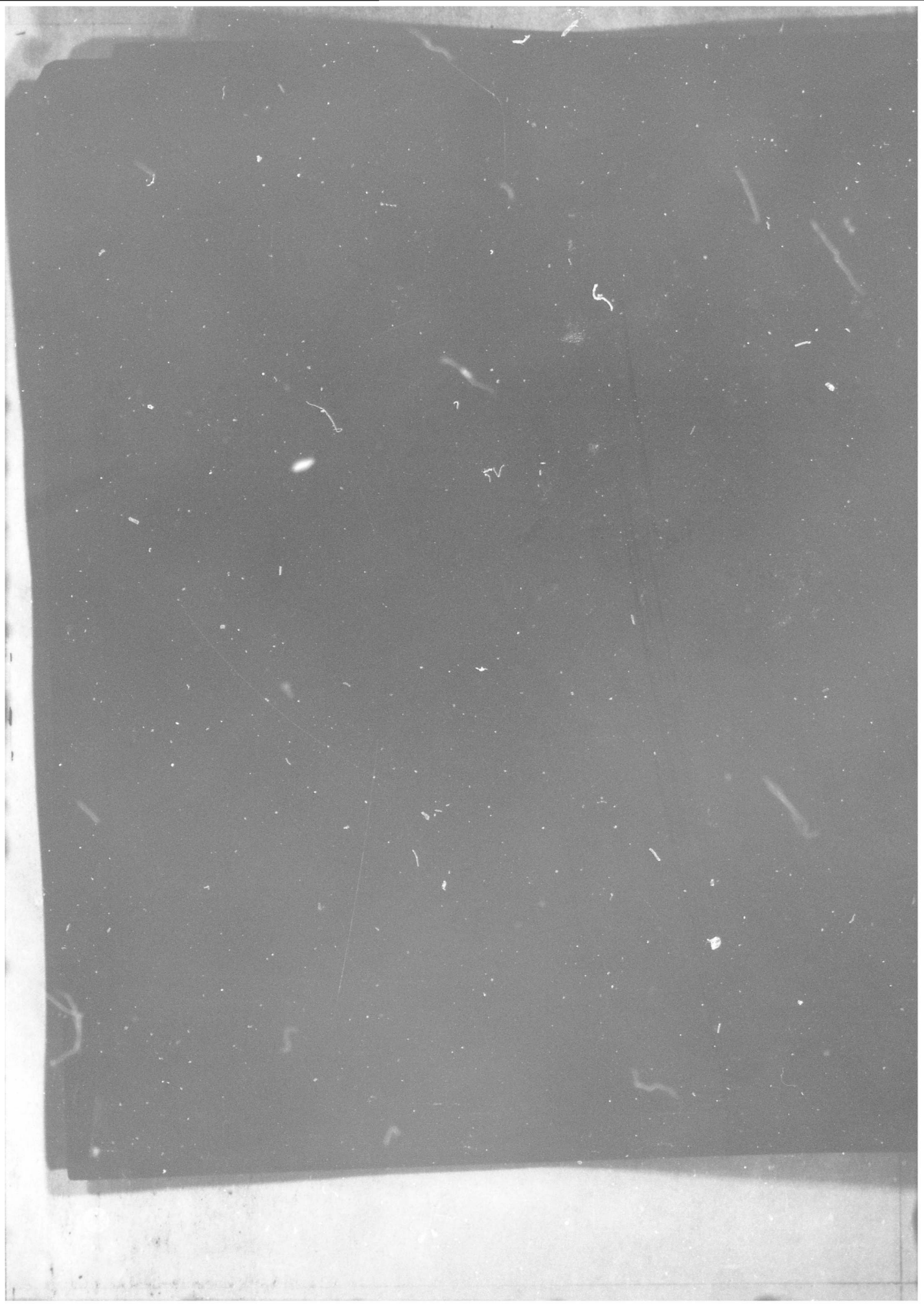

EVALUATION AND IMPROVEMENT OF COATINGS FOR COLUMBIUM ALLOY GAS TURBINE ENGINE COMPONENTS

H. A. Hauser and J. F. Holloway, Jr.

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FOREWORD

This report discusses the work performed by Pratt & Whitney Aircraft Division of United Aircraft Corporation, East Hartford, Connecticut on evaluation and improvement of coatings for columbium alloy gas turbine engine components during the period from 1 April 1965 to 30 April 1966. The work was performed in accordance with Contract No. AF33(615)-2117 under the direction of Mr. Norman M. Geyer (MAMP), Metals Branch, Metals and Ceramics Division of the Air Force Materials Laboratory. This contract was conducted under Project 7312, "Metal Surface Deterioration and Protection", Task 731201, "Metal Surface Protection", and was supported in part by the AFML Director's Fund. The report was submitted 31 May 1966 in compliance with Line Item 1c of DD Form 1423, dated 13 February 1965, attached to the original contract, and with Exhibit C, Part I, paragraph C of Contract Modification No. S2(66-1258), dated 1 April 1966. The contractor has assigned the number PWA-2809 to the report.

This technical report has been reviewed and is approved.

I Perlmutter

I. Perlmutter
Chief, Metals Branch
Metals and Ceramics Division
AF Materials Laboratory

ABSTRACT

Data generated using laboratory tests such as oxidation-erosion, thermal fatigue, ballistic impact, and wear-galling to simulate turbine engine environments are reported for eight coated columbium alloy composites. Applications considered were turbine vane and turbine blade airfoils operating in the 1800°-2500°F temperature range and turbine blade roots at 1300°-1600°F. Five of the eight coatings were modified after preliminary screening tests and re-evaluated. Complete evaluation of TRW TiCr-Si (vacuum) pack coated D-43 alloy, including stress-rupture testing, established the system as a turbine vane candidate for engine testing in the last phase of the program. Preliminary results on Sylcor SiCrTi slurry coated D-43 alloy indicated promise for turbine vane use and continued testing was recommended. TRW TiCr-Si (vacuum) pack coated Cb-132M alloy demonstrated potential as a blade airfoil composite and is being fully evaluated. The Naval Research Laboratory zinc coating selected for possible blade root application was deleted from the program because of nonreproducibility. Sylcor Ag-Al-Si (508-F) coated Cb-132M was the most promising turbine blade root composite in screening tests and will be fully investigated for that purpose. Performance of all candidate blade root coatings was marginal, and emphasis on this area during the next yearly effort was recommended.

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SECTION I

INTRODUCTION

The rapid oxidation rate of columbium alloys has prevented extensive application in high-temperature oxidizing environments. Since protection from oxidation is necessary to maintain desirable mechanical properties, coating development efforts have been extremely active in recent years. Most development programs, however, have been concerned with applications such as re-entry vehicle and rocket engines operating for short times in the 2400° to 3000°F range to take maximum advantage of the high melting point, relatively low density, and high strength and ductility of columbium alloys at these temperatures.

As a result, the evaluation of coated columbium alloys has been very limited under those conditions found in gas turbine engines, where operating temperatures are currently not as extreme, but where reliability must be measured in hundreds or thousands of hours and/or cycles (both thermal and mechanical). In addition to the high-temperature oxidizing environment, turbine blade and vane components must withstand thermal fatigue from cyclic operation, impact damage from ingested foreign objects, high steady-state and vibratory stresses from centrifugal loading (in the case of blades), and corrosion and erosion from the combusted fuel and air stream.

In the gas turbine engine field, lift or vertical takeoff powerplants are potentially a most attractive application area for coated columbium alloy blades and vanes. These engines must be high in performance and of minimum weight to accomplish the desired missions, thereby requiring high turbine inlet temperatures (2000°-2700°F). Adequate blade and vane lives under these conditions are possible with existing superalloys only if complex cooling schemes which compromise performance are employed, while advanced materials such as coated columbium alloys might be used uncooled or with considerably less cooling air.

The subject test and evaluation program is concentrated in two distinct areas: 1) turbine blade airfoil and turbine vane and 2) turbine blade root. Tentative operating temperature requirements for the blade airfoil and vane are 1800° to 2500°F. Although blade root temperatures are only 1300° to 1600°F, the coating/columbium substrate composite must operate under conditions of high stress and mechanical wear.

The program conducted during this annual report period was divided into three phases as tabulated below:

Phase No.	Contractual Item No.	Title
I	1	Literature Survey
I	2	Preliminary Screening Evaluation
II	3	Coating Improvement
III	4	Advanced Evaluation, Coating Life Properties
III	5	Advanced Evaluation, Composite Properties

Flow charts showing the progression of tests within the three phases are presented at the beginning of each evaluation section in this report.

A similar evaluation of additional coating/columbium substrate systems will be conducted during the coming year; a description of this part of the program is included in Section V, Future Work. The composites for subsequent engine testing will be selected from those systems which were evaluated during the first year and those to be evaluated during the second year.

SECTION II

SUMMARY

Selection of the coating-substrate systems tabulated below was based on the results of the literature survey and of discussions between the contractor and the Air Force Materials Laboratory Project Engineer. These systems were evaluated through preliminary screening tests for gas turbine engine vane and blade applications.

Application	Coating	Columbium Alloy Substrate
Turbine Vane	TRW TiCr-Si (vacuum) pack	C-129Y
	Sylcor Ti-CrTi-Si (triplex) pack	D-43
	Sylcor SiCrTi slurry	D-43
Turbine Blade Airfoil	TRW TiCr-Si (vacuum) pack	Cb-132M
	TRW TiCr-Si (slip) pack	Cb-132M
Turbine Blade Root	Sylcor Sn-Al	Cb-132M
	Sylcor Ag-Al-Si	Cb-132M
	Zn	Cb-132M

Based on the results of oxidation-erosion and thermal fatigue testing at 2200°-2400°F, ballistic impact testing at 70°-2200°F, and previous data, the TRW TiCr-Si (vacuum) pack/D-43 (vane) and TRW TiCr-Si (vacuum) pack/Cb-132M (blade airfoil) systems were selected for advanced evaluation. The former system, although not among the above eight initially included in the program, was chosen for advanced evaluation based on available comparative data and discussions with the Air Force Materials Laboratory Project Engineer. Prestrain/oxidation and wear-galling screening tests on the blade root systems demonstrated that Sylcor Ag-Al-Si coated Cb-132M alloy was the best of those investigated and was qualified for advanced evaluation. During the preliminary screening portion of the program, each coating, with the exception of the TRW TiCr-Si (slip) pack and Sylcor SiCrTi slurry, was modified to improve performance and re-evaluated.

Complete evaluation of TRW TiCr-Si (vacuum) pack coated D-43 alloy for turbine vane application by oxidation-erosion and thermal fatigue testing at 2000°-2500°F, ballistic impact testing at 70°-2600°F, and stress-rupture testing at 2200° and 2500°F demonstrated that this system is a candidate for engine testing. Test results are summarized below.

Test	Life
Oxidation-erosion	410 hours at 2000°F, 220 hours at 2200°F, 60 hours at 2400°F, and 40 hours at 2500°F; transient overheating to 2600°F reduced the 2200°F life by approximately 50 percent.
Thermal fatigue	1200-1600 cycles at 2000°F, 800 cycles at 2200°F, 400-1200 cycles at 2400°F, and 1600-2200 cycles at 2500°F
Ballistic impact	1-3 hours at 2200°F after 70°-2200°F impact
Stress-rupture	100 hours at 2200°F and 12,000 psi or 2500°F and 5600 psi

Advanced evaluation of the TRW TiCr-Si (vacuum) pack/Cb-132M system for blade airfoil application was not conducted because the low ductility of the available bar prevented meaningful mechanical fatigue and creep testing. Additional material is presently being procured for this purpose.

Sylcor Ag-Al-Si coated Cb-132M alloy was partially evaluated through the advanced evaluation phase for blade root application. Through static oxidation testing for general integrity, airfoil-root coating compatibility testing, and 2000°F transient overshoots, the system demonstrated adequate performance. Mechanical fatigue testing of both Ag-Al-Si coated specimens and specimens containing airfoil-root coating overlaps is awaiting the receipt of Cb-132M alloy bar now on order.

A contract extension of one year duration has been awarded to evaluate seven additional coating-substrate systems. These systems, as well as those evaluated thus far, are candidates for engine testing in the last phase of the program.

SECTION III

TECHNICAL DISCUSSION

1. ITEM 1

LITERATURE SURVEY

In accordance with the requirements of Item 1 of the contract work statement, a literature survey was conducted to determine the current state of the art of coatings for the protection of columbium alloys in the 1300° to 2500°F temperature range. In the course of this survey, data were collected on the performance of over 40 combinations of coatings and columbium substrate materials. The general guide lines for this investigation are listed below:

- The literature search was concentrated on recent development and evaluation programs.
- Special attention was concentrated on temperature and environmental conditions approximating those of a gas turbine engine.
- Each test was objectively evaluated to determine whether the specific test conditions involved tended to favor certain types of coatings.
- In evaluating each coating, consideration was given not only to its performance at high temperatures but also to the properties which would be applicable to the lower temperature blade root area as well. Such properties include oxidation life (the length of accumulated test time before failure due to oxidation of substrate) at low temperatures, pest behavior (catastrophic low temperature oxidation), and ductility.

A list of references for the literature reviewed during the survey starts on page 211. A discussion of the results available from these investigations at the time of the survey is presented below.

Wurst and Cherry (Ref. 1) have reported good oxidation life for the TRW TiCr-Si (vacuum) pack coating at 1600°, 2400°, and 2600°F on B-66 and D-43 columbium alloys. Maximum life of the coating at 2400°F was found to be 124 and 113 one-hour cycles on the B-66 and D-43 substrates, respectively. (Each cycle consisted of 1 hour in 2400°F static air followed by a period at ambient temperature.) For both alloys, some embrittlement and degradation in strength were noted as a result of the coating and/or coating application process. A difference in B-66 alloy substrate hardness between

two coating application batches was detected. Adequate stress-rupture life and resistance to creep were also reported (Ref. 1). Serious problems were encountered, according to Wurst and Cherry, on another system, TiCr-Si on C-129Y columbium alloy (Ref. 2). Losses in ultimate strength, yield strength, and oxidation life were reported for this system.

Stetson (Ref. 3) confirmed the Wurst & Cherry oxidation life results for the TiCr-Si/B-66 system and reported similar data for the TiCr-Si/Cb-752 system. It was noted that the reliability of the coating on D-43, B-66, and Cb-752 columbium alloys had decreased the most between 2400°F and 2600°F; the 95-percent probability limit dropped from 51 hours at 2400°F to 6 hours at 2600°F for all gage thicknesses. Special attention was called to the fact that the TiCr-Si/D-43 system suffered a significant loss in ultimate and yield strengths with an attendant increase in elongation as a result of coating. At 2200°F, Stetson reported that, for the TiCr-Si coating on D-43 alloy, the ultimate tensile strength was 45 percent, the yield strength 40 percent, and the elongation 235 percent of that of the uncoated alloy, while on B-66 and Cb-752 alloys the ultimate tensile strength was 80 percent, the yield strength 75 percent, and elongation from 35 to 80 percent of those of the uncoated alloys. It was noted that the losses found in the TiCr-Si/D-43 system probably exist also for other alloys which utilize carbide strengthening. This reasoning followed an extensive investigation by Stetson which revealed that this behavior apparently resulted from the coating constituents having a greater affinity for the carbon in these substrate alloys than do the alloy constituents, thus disturbing the substrate chemistry (Ref. 3).

Other oxidation life data for the TRW TiCr-Si coating on Cb-752 (Ref. 4, 5), D-43 (Ref. 2, 4) D-41 (Ref. 6), and Cb-132 (Ref. 4) columbium alloys were reviewed. Although some variations were reported, coating protectiveness on these alloys did not differ significantly from that reported for the TiCr-Si coating on B-66 alloy (Ref. 1, 2, 3).

Previously unreported oxidation-erosion tests conducted by Pratt & Whitney Aircraft showed good results for the TRW TiCr-Si/D-43 system during dynamic oxidation-erosion as tabulated below.

Temperature	Test Time	Coating Condition
2200°F	100 hours	Small spot failure at the base
2200°F	100 hours	No failure
2400°F	100 hours	Leading edge failure at 100 hours
2600°F	80 hours	Leading edge failure at 80 hours

During these tests the specimens were rotated at 1750 rpm in a gas stream consisting of a mixture of combusted JP-5 fuel and air. This mixture closely approximates the environment encountered in a gas turbine engine. An evaluation of the TiCr-Si coating on D-14 and D-31 columbium alloys was also obtained.

In a rather unique test, Pietromonaco and associates (Ref. 7) found that the oxidation life of the TRW TiCr-Si coating on D-36 and Cb-752 alloys was inadequate when tested in a "re-entry flight profile simulator." This test incorporated a Mach 1 airflow over the test specimens while the pressure and temperature were varied to simulate a re-entry condition. The peak temperature was 3000°F. Although the low pressures are not applicable to a turbine engine environment, this was one of few reported tests which evaluated the performance of refractory metal coatings in a high-velocity gas stream under conditions approaching those present in an advanced turbine engine.

One coating developed by the Sylvania Sylcor Division of General Telephone and Electronics, Inc. was a Cr-Ti-modified-silicide. This coating demonstrated very good oxidation life on B-66 alloy. Wurst and Cherry (Ref. 1) reported that, in terms of oxidation life on this particular alloy, the coating performed better than any coating tested to date, with typical lives ranging up to 199 one-hour cycles at 2400°F and up to 156 cycles at 2600°F. Some of the specimens, tested at 2400°F, withstood 225 cycles (maximum duration of the test) without failure.

A disadvantage of the Cr-Ti-modified-silicide coating was a detrimental effect of the coating and/or coating application process on the ductility of the substrate alloy. One specimen evaluated by Wurst and Cherry (Ref. 1) exhibited a high degree of embrittlement, while other, more ductile specimens demonstrated mechanical failure after less than 1.0 percent elongation. Typical ultimate and yield strengths of the more ductile specimens were 96 and 80 percent, respectively, of the uncoated-specimen strengths.

The protective life of the Boeing Company's Disil coating has been reported on B-66 (Ref. 1), C-129Y (Ref. 2), and D-36 (Ref. 8) columbium alloys. At 2400°F, typical oxidation lives from 15 to 53 one-hour cycles have been observed, depending on which columbium alloy was used (Ref. 1, 2). Wurst and Cherry (Ref. 1) reported that the coating did not appear to alter the B-66 alloy microstructure and the room temperature mechanical properties were the best of any system tested. They reported further, however, that, on D-43 alloy, not only was there a large degradation in strength, but the coating exhibited serious low-temperature failures (four to eight one-hour cycles at 1600°F) in addition to poor life at 2400° and 2600°F. Testing in the "re-entry flight profile simulator", in a manner similar to the previously mentioned testing of the TiCr-Si coatings, demonstrated that the Disil coating possessed inadequate life for the re-entry application (Ref. 7).

The Chromizing Corporation's Durak KA coating has been reported to afford reasonable protection on B-66 (Ref. 1, 9) and C-129Y (Ref. 2) alloys. Good tensile property retention has been reported as well as good creep resistance and stress-rupture life on B-66 alloy (Ref. 1). The coating has appeared compatible with D-43 alloy (Ref. 2), although only limited data have been reported. No effects from thermal shock were noted for the coating (Ref. 9).

The results of tests on a Cr-B-Si coating developed by Ling-Temco-Vought have been erratic, the coating life depending on the composition of the columbium alloy substrate (Ref. 2, 8, 9). On B-66 alloy, the coating was questionable in terms of oxidation life. Although the coating does not appear to alter the base metal structure, severe losses in room temperature tensile properties were experienced (Ref. 1). Some losses in mechanical properties were also experienced on D-43 and C-129Y alloys, and the oxidation life at 1600°F was very erratic, as evidenced by failure in a range of 4 to 24 one-hour cycles (Ref. 2). The reliability of the coating appears questionable due to wide variations in measured oxidation life (Ref. 2). Severe embrittlement of D-36 foil by the coating or coating process has been experienced (Ref. 8, 9).

A Sylcor-developed Ag-Al-Si coating was the only coating tested by Wurst and Cherry (Ref. 2) which exhibited useful life at 3000°F (11 to 13 one-hour cycles on D-43 alloy and 3 to 7 cycles on C-129Y alloy). Some batch variations (in both mechanical properties and oxidation life) were noted; however, the coating exhibited little effect on the room temperature strength of D-43 alloy (Ref. 2). Controlling the thickness of the coating appeared to be a problem, with reported values ranging from 2.4 to 13.6 mils. There was some alteration of the substrate structure when the coating was applied to C-129Y (Ref. 2). In one instance, the coating was reported to have performed for 1000 hours at 1100° to 2000°F without problems, although the substrate alloy was not mentioned (Ref. 10).

The Sn-Al coating developed by Sylcor has demonstrated good oxidation life in a static environment up to 2300°F on Cb-132M columbium alloy (Ref. 11). Although thickness uniformity was questionable, the coating possessed good diffusional stability and did not appear to seriously alter the Cb-132M microstructure.

For operation up to 1800°F, the zinc-base coating developed by the U.S. Naval Research Laboratories has been reported to provide oxidation life for hundreds of hours (Ref. 12, 13). The coating appeared to possess remarkable self-healing properties resulting from the high vapor pressure of the zinc-columbium compounds. No effects on substrate mechanical properties have been reported, but it has been stated that the coating has actually semipurged contaminated columbium alloy of oxygen. Thickness of the coating was subject to some variations depending on the coating application method.

The NAA-85 coating developed by North American Aviation, Inc, is an aluminide type of coating system. Good ductility was reported for as-coated specimens (Ref. 8, 14), but questionable oxidation life was obtained at 2000°F (Ref. 8). There is little published data available concerning coating reliability versus operating temperature.

The MAC TS-137 coating developed by McDonnell Aircraft Corporation has demonstrated good protective qualities from 1400° to 3000°F for re-entry application (Ref. 15). The oxidation life of the coating as tested on D-14 alloy is too low for turbine engine application, with a reported oxidation life from 22 to 34 hours in the 1400° to 2800°F range.

Other coatings which received attention during the course of the literature survey are listed below.

Coating	Vendor	Alloy	Reference
PFR-30	Pfaudler Company	B-66	1
PFR-32	Pfaudler Company	D-43 C-129Y	2
Pt & Pt-Rh	Metals and Controls, Inc.	FS-85	16
G.T.C.	General Technologies Corp.	D-36	8
Disil 2	Boeing Company	D-36	8
Vanadium Modified Disil	Boeing Company	C-129Y	7
AMFKOTE-3	American Machine & Foundry	D-36	8
AMFKOTE-30	American Machine & Foundry	D-36	8
Du Pont 1	Du Pont	D-36	8
Ti-Si	Sylcor	D-36	8

It is of particular interest to note the research and development efforts currently being carried out to develop new or modified coating systems. The vanadium modified Disil coating being developed by the Boeing Company

appears to offer significant promise for high-temperature application. Although data is still quite limited and preliminary in nature, evaluation of the coating on C-129Y columbium alloy shows very good protective qualities during the "flight profile simulator" test (Ref. 7, 17).

A joint study by the Solar Division of International Harvester Company and the Illinois Institute of Technology Research Institute is currently being conducted to develop coatings for columbium alloys (Ref. 18, 19). Solar, responsible for the program, is studying the development of modified silicide systems as based on the general success of the (Cr-Ti)-Si coating. Preliminary data have been obtained on a number of disilicides from the Cb-Ti-Mo-Si, Cb-Ti-Cr-Si, and the Cb-V-Cr-Si systems.

The IIT Research Institute is conducting development studies on aluminide systems based on the compound (Ti-Cb)-Al, improved $CbAl_3$ compounds, and bcc coating systems (multilayer coatings consisting of body-centered cubic metals and solid solutions). Promising results have been reported (Ref. 18, 19), though data is presently quite preliminary in nature.

Sama and associates (Ref. 20, 21, 22) are conducting a study to determine pertinent phase and compositional changes in several coating systems. This analytical study is oriented toward determining how these changes affect the oxidation lives of various coatings.

The limitations of the Sn-Al coating on columbium are being studied by Priceman and Sama (Ref. 23). In connection with this study, development of slurry-applied silicide systems containing chromium, titanium and vanadium is also underway. Work with a Si-20Cr-5Ti coating on D-43 alloy indicates no serious loss of mechanical properties as a result of the coating heat-treat cycle or the presence of the coating itself, nor did it indicate that there was inadequate protection by the coating during subsequent oxidation testing. Thus far, pest failures do not appear to be a problem; the typical oxidation life has been reported to be greater than 150 hours at 1600° and 2000°F and 47 to 72 hours at 2300°F.

2. ITEM 2

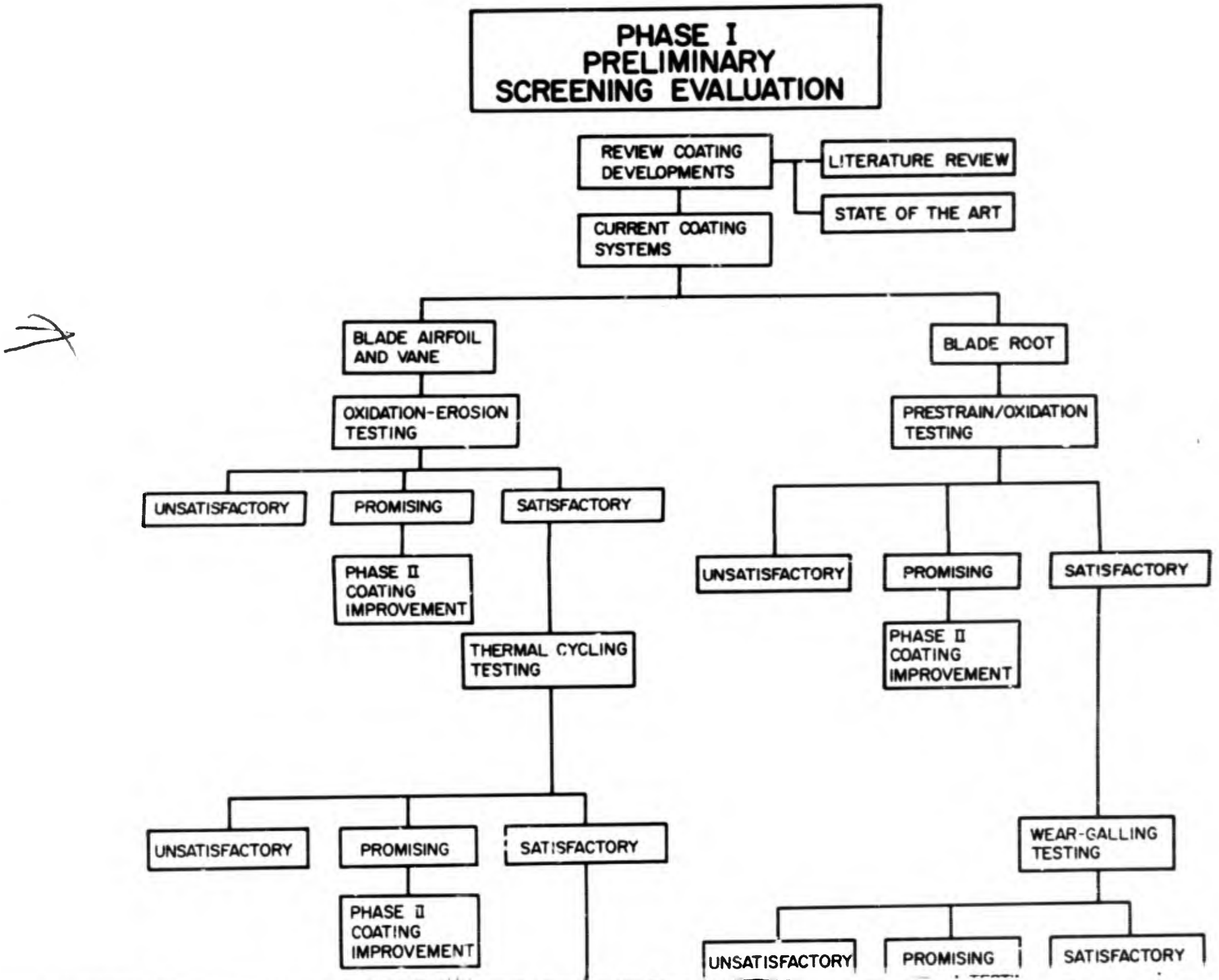
PRELIMINARY SCREENING EVALUATION

Item 2 requires that the contractor perform preliminary screening tests (Phase I) on the most promising coating-substrate combinations selected as a result of the literature survey (Item 1) and on at least one new coating-substrate system developed under Air Force sponsorship or elsewhere, the total not to exceed eight systems. Six systems were selected in coordination between the Air Force Materials Laboratory Project Engineer and the contractor in the first quarter. In the third quarter, two additional systems were defined. The additional systems were classified as Phase I supplemental systems and were not candidates for Phase II improvement or Phase III advanced evaluation. A technical flow chart showing the progression of tests through Phase I of the program is shown in Figure 1.

2.1 MATERIALS

Turbine vane alloys must possess moderate strength (about 5000 psi minimum rupture strength) at operating temperatures, thermal fatigue resistance, weldability, ductility for fabrication into sheet and subsequent forming, and ductility after recrystallization. After discussions between the AFML Project Engineer and the contractor, D-43 and C-129Y alloys were selected as the vane substrate materials. Chemical analyses and hardness data for the as-received alloys are presented in Table I. Representative microstructures of each heat (three heats per alloy) are shown in Figures 2 through 5. The vane alloy thermal fatigue test necessitates construction of a sheet material airfoil specimen with a leading edge radius of two and one-half times the thickness (2.5T) for 0.050-inch-thick material. Results of bend tests, conducted to assure this capability prior to airfoil forming, are summarized in Table II. Both the D-43 and C-129Y as-received materials met the ductility requirement normal to the sheet rolling direction.

Turbine blade alloys must have high stress-rupture and fatigue strength, good creep resistance, and the ability to absorb impact forces without fracture. Cb-132M alloy was selected since it possesses properties which more nearly fulfill these requirements when compared to other commercially available columbium alloys. Three-inch-diameter extruded billets were obtained from E.I. du Pont de Nemours & Co. The supplied chemical analyses of the two different heats appear in Table III. Five of the six procured billets were extruded at Nuclear Metals, Inc. using the following parameters:


PHASE I
PRELIMINARY
SCREENING EVALUATION
REVIEW COATING DEVELOPMENTS
LITERATURE REVIEW
STATE OF THE ART
CURRENT COATING SYSTEMS
BLADE AIRFOIL AND VANE
BLADE ROOT
OXIDATION-EROSION TESTING
PRESTRAIN/OXIDATION TESTING
UNSATISFACTORY
PROMISING
SATISFACTORY
PHASE II COATING IMPROVEMENT
THERMAL CYCLING TESTING
UNSATISFACTORY
PROMISING
SATISFACTORY
PHASE II COATING IMPROVEMENT
WEAR-GALLING TESTING
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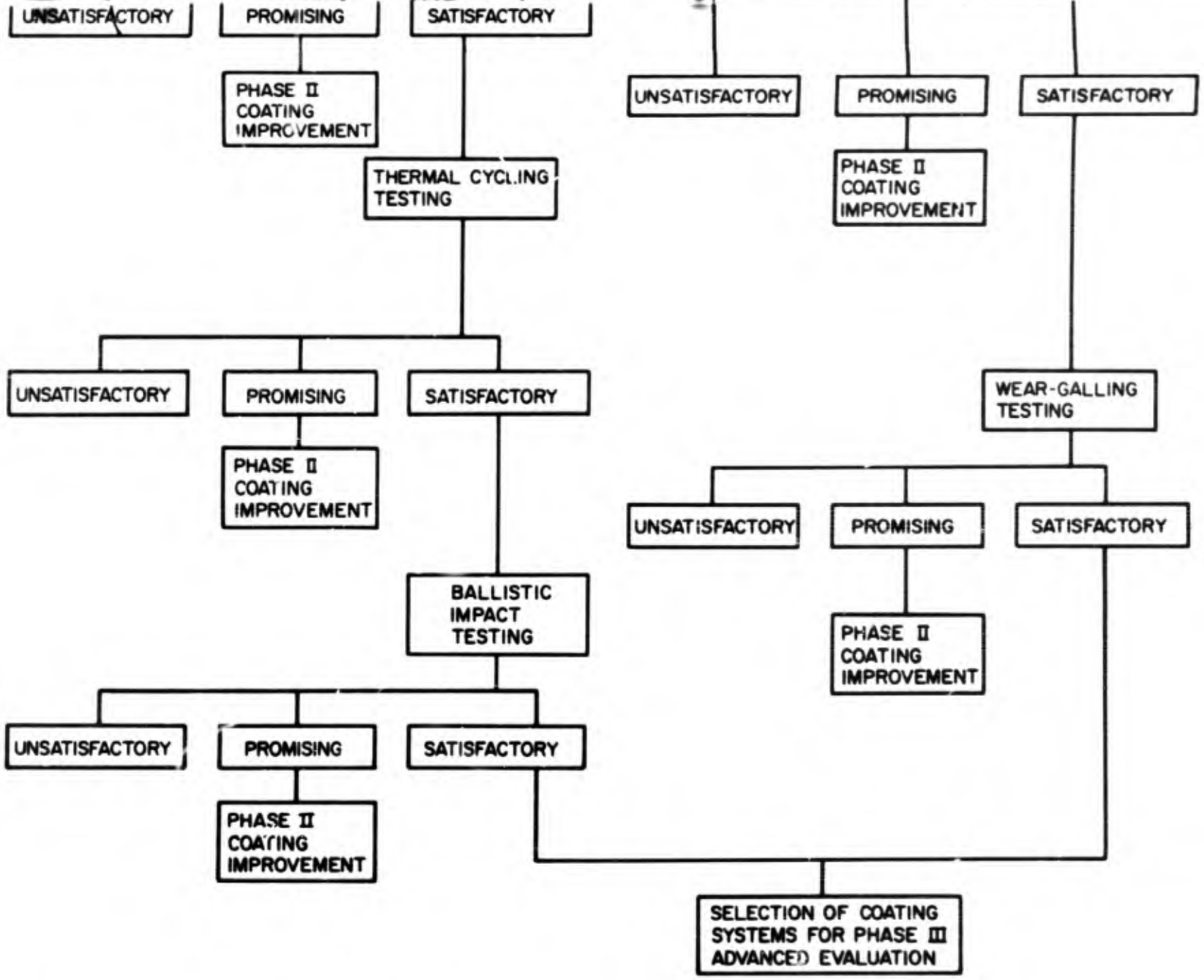

Figure 1. Flow Chart for Phase I of Program (Items 1 and 2)

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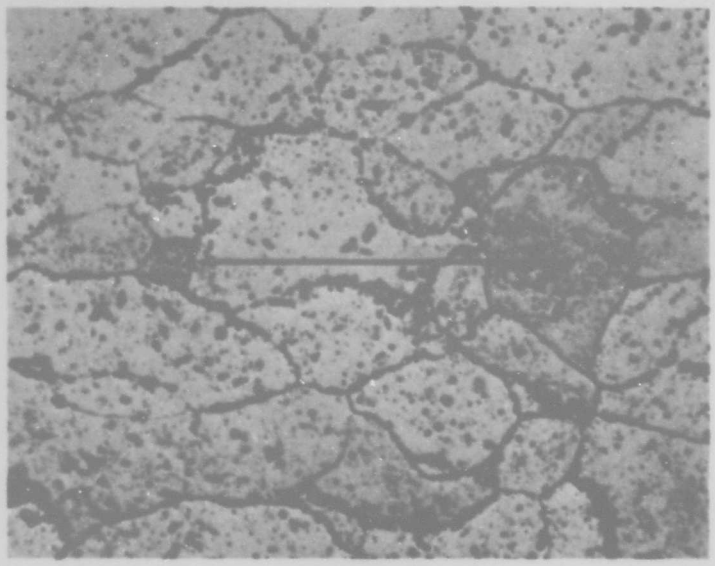
TABLE I

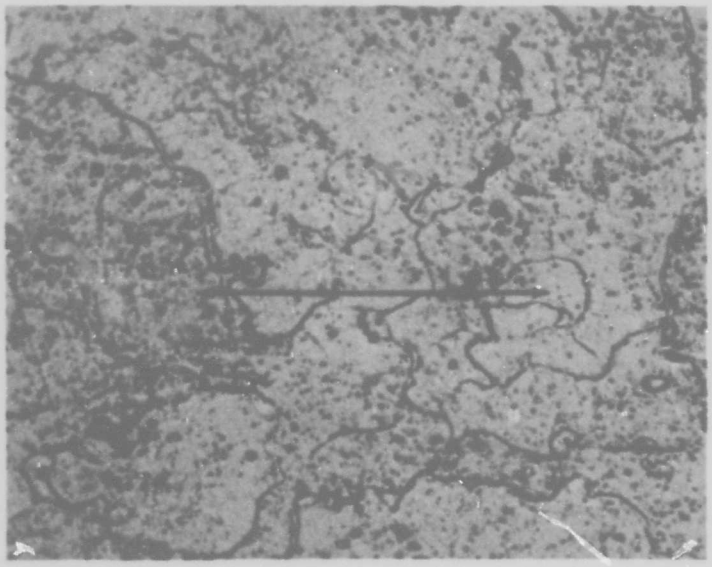
CHEMICAL ANALYSES AND HARDNESS DATA FOR THE COLUMBIUM ALLOYS EVALUATED FOR VANE APPLICATION

Supplier	Alloy	Nominal Composition	Heat No.	Source of Analysis	Composition (percent)						Interstitial (Gas) and Alloy Impurities (parts per million)						Hardness[c] (DPH)
					Cb	W	Zr	Hf	Y	C	O	N	C	H	Zr	Ta	
Du Pont	D-43	Cb-10W-1Zr-0.1C	423	P&WA	Balance	9.14	2.32[b]	---	---	---	---	---	---	10	---	---	180
			487	Supplier	Balance	9.30	1.10	---	---	0.094	103	43	---	11	---	---	216
			488	Supplier	Balance	9.80	1.00	---	---	0.104	175	75	---	7	---	---	189
Wah Chang	C-129Y	Cb-10W-10Hf-0.1Y	57216	Supplier[a]	Balance	9.85	---	10.45	0.19	---	85	47.5	75	4.6	4050	750	---
			57216	P&WA	Balance	9.44	---	10.51[b]	0.14	---	50	---	---	3.3	---	---	210
			57230	Supplier[a]	Balance	9.95	---	10.20	0.105	---	50	36.5	85	4.3	2800	750	---
			57230	P&WA	Balance	9.44	---	9.73	0.14	---	---	---	---	3.0	---	---	210
			57259	Supplier[a]	Balance	9.99	---	9.30	0.115	---	70	31.5	75	3.75	2550	1150	---
			57259	P&WA	Balance	9.72	---	9.41	0.13	---	80	---	---	3.1	---	---	194

Notes: a. Average of two analyses
b. Hafnium plus zirconium
c. Hardness determined at P&WA using 50-gram load

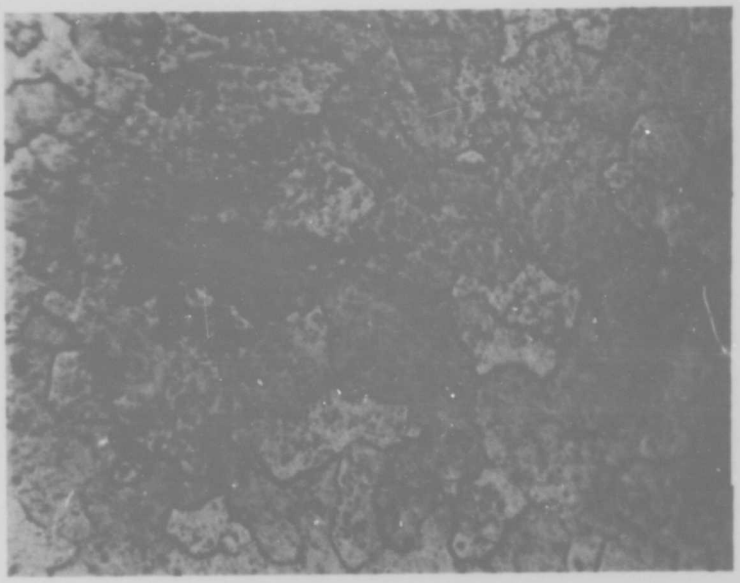


ALLOY: D-43, SHEET VENDOR HEAT: NO. 487
PLANE: PARALLEL TO ROLL DIRECTION (ARROWS) MAG: 500X
ETCH: 60% LACTIC ACID, 20% HYDROFLUORIC ACID, 20% NITRIC ACID



ALLOY: D-43, SHEET VENDOR HEAT: NO. 488
PLANE: PARALLEL TO ROLL DIRECTION (ARROWS) MAG: 500X
ETCH: 1st, 60% LACTIC ACID, 20% HYDROFLUORIC ACID, 20% NITRIC ACID,
2nd, 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER

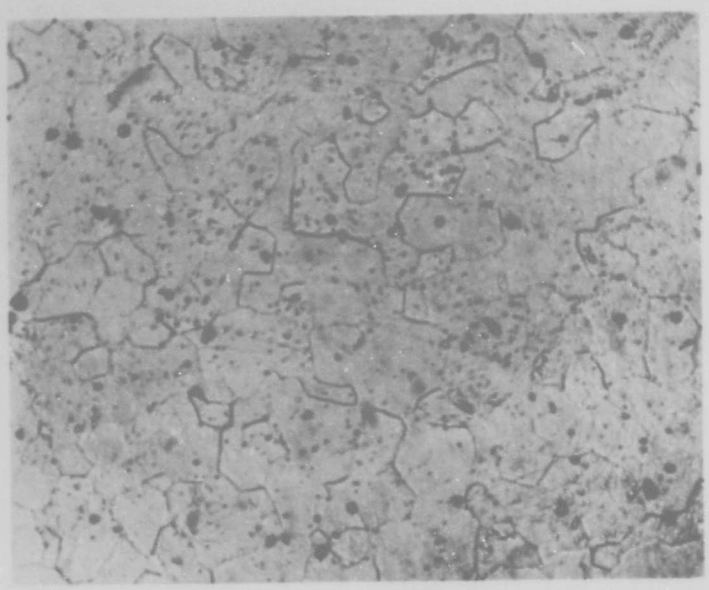
Figure 2. Microstructure of As-Received D-43 Alloy Sheet



ALLOY: D-43, EXTRUDED BAR VENDOR HEAT: NO. 423
PLANE: NORMAL TO EXTRUSION DIRECTION MAG: 500X
ETCH: 60% LACTIC ACID, 20% HYDROFLUORIC ACID, 20% NITRIC ACID

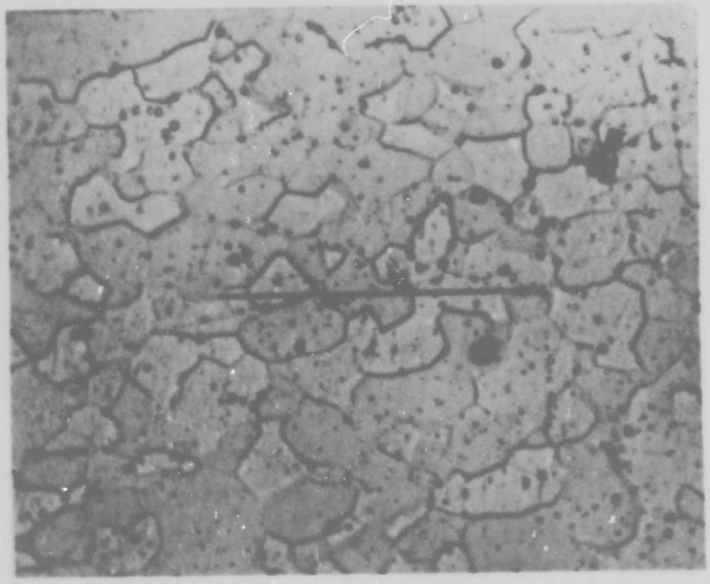
ALLOY: D-43, EXTRUDED BAR VENDOR HEAT: NO. 423
PLANE: PARALLEL TO EXTRUSION DIRECTION (ARROWS) MAG: 500X
ETCH: 60% LACTIC ACID, 20% HYDROFLUORIC ACID, 20% NITRIC ACID

Figure 3. Microstructure of As-Received D-43 Alloy Bar Stock



ALLOY: C-129Y, EXTRUDED BAR
PLANE: NORMAL TO EXTRUSION DIRECTION
ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER

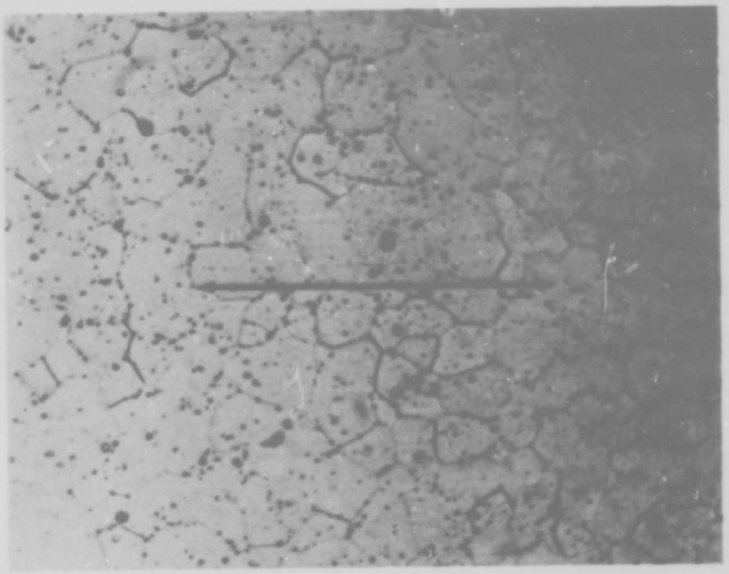
VENDOR HEAT: NO. 57259
MAG: 500X



ALLOY: C-129Y, EXTRUDED BAR
PLANE: PARALLEL TO EXTRUSION DIRECTION (ARROWS)
ETCH: 60% LACTIC ACID, 20% HYDROFLUORIC ACID, 20% NITRIC ACID

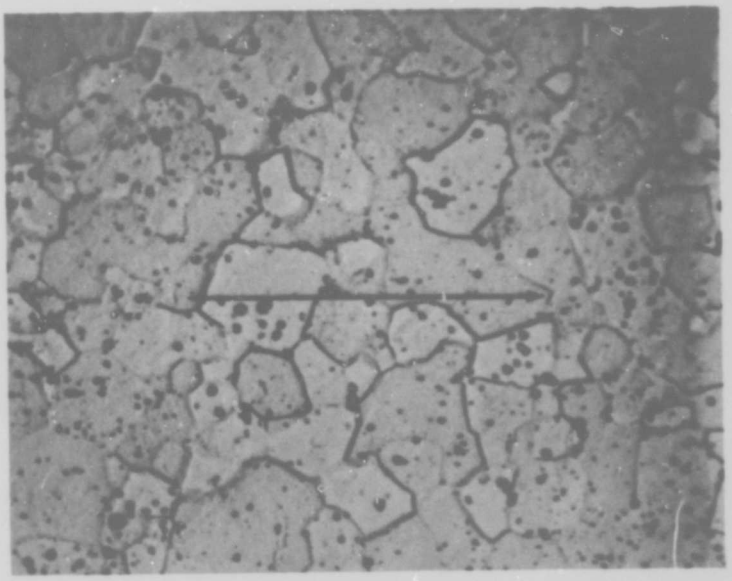
VENDOR HEAT: NO. 57259
MAG: 500X

Figure 4. Microstructure of As-Received C-129Y Alloy Bar Stock



ALLOY: C-129Y, SHEET
PLANE: PARALLEL TO ROLL DIRECTION (ARROWS)
ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER

VENDOR HEAT: NO. 57216
MAG: 500X



ALLOY: C-129Y, SHEET
PLANE: PARALLEL TO ROLL DIRECTION (ARROWS)
ETCH: 60% LACTIC ACID, 20% HYDROFLUORIC ACID, 20% NITRIC ACID

VENDOR HEAT: NO. 57230
MAG: 500X

Figure 5. Microstructure of As-Received C-129Y Alloy Sheet

TABLE II

RESULTS OF BEND TESTS ON D-43 AND C-129Y SHEET MATERIAL

Alloy	Supplier	Heat No.	Mandrel Axis Relative to Sheet Roll Direction	Test Results (No. of Specimens Tested)		
				3T Radius	2T Radius	1T Radius
D-43	Du Pont	487	Normal	Passed (1)	Passed (2)	Failed (3)
D-43	Du Pont	487	Parallel	Passed (1)	Failed (8)	---
C-129-Y	Wah Chang	57230	Normal	Passed (1)	Passed (2)	Passed (2)
C-129-Y	Wah Chang	57230	Parallel	Passed (1)	Passed (2)	Passed (1)

Note: T represents sheet thickness. Specimens passed test if bent 105° without failure.

TABLE III

CHEMICAL ANALYSES AND HARDNESS DATA FOR THE Cb-132M COLUMBIUM ALLOY EVALUATED FOR BLADE APPLICATION

Du Pont Heat No.	Composition (percent)					Interstitial (Gas) and Alloy Impurities (parts per million)				Hardness (DPH)
	Cb	Ta	W	Mo	Zr	O	H	N	C	
481	Balance	18.3	14.5	4.7	0.91	65	16	42	1060	345
489	Balance	19.2	15.4	4.9	1.1	60	19	46	945	---

Note: Hardness determined at P&WA using 50-gram load

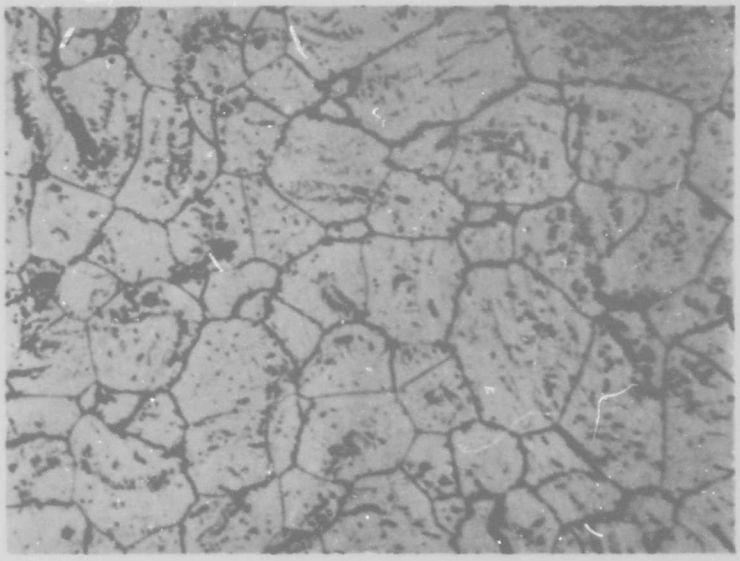
Extrusion temperature	3190° to 3600°F
Reduction ratio	20X to 25X
Canning material	Molybdenum
Extrusion die	ZrO_2 lined

The variation in extrusion parameters was due to differences in billet size. Metallography of the resultant 0.625-inch-diameter bar revealed a recrystallized structure containing Widmanstatten-type zirconium carbide precipitate (Figure 6). Hardness is shown in Table III.

The remaining billet was extruded at 3400°F with a 5-to-1 reduction ratio. This procedure was required so that nonrecrystallized material could be obtained in order that forging of thermal fatigue specimens could be accommodated. The resultant 1.25-inch-diameter material exhibited a predominantly worked microstructure (Figure 7).

The selection of the coatings for evaluation in the program was based on the results of the literature survey and data previously obtained at Pratt & Whitney Aircraft on the performance of coated columbium alloys. (Ref. 4, 6). The in-house work demonstrated that, while oxidation life of the system is considered the major problem area, embrittlement and consequent reduction of strength can be of primary importance in some operating environments. One such study determined that the TRW TiCr-Si coating performed well during high temperature (2200°-2600°F) oxidation-erosion testing. However, testing of a coated turbine blade in a turbine rig revealed that, after only 1 hour and 40 minutes, fatigue cracking appeared in the root section operating at a metal temperature of approximately 1400°F. Failure analysis revealed that the brittle nature of the coating at root temperatures (1300°-1600°F) and recrystallization of the columbium substrate during coating application gave rise to fatigue cracking at the root surface. This fatigue cracking progressed from craze cracks at the coating surface and extended into the substrate (see Figure 8). The need for crack-free ductile blade root coatings was thus established. Such a root coating could consist of a modified or partial airfoil coating on the blade root section (Ref. 24), or a coating completely different in composition from that of the airfoil. The latter application has been considered in this program; i.e., duplex coatings were evaluated.

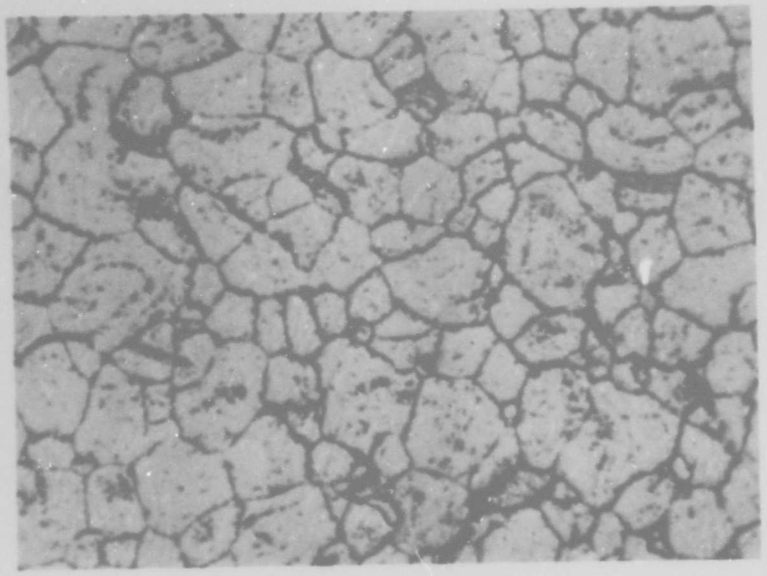
Since the vane is a stationary (nonrotating) component, lower stresses (as well as lower temperatures) are experienced in the shroud sections. For this reason, only single coatings for both airfoils and shrouds have been considered.



ALLOY: Cb-132M, EXTRUDED BAR VENDOR HEAT: NO. 481
PLANE: NORMAL TO EXTRUSION DIRECTION MAG: 500X
ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER

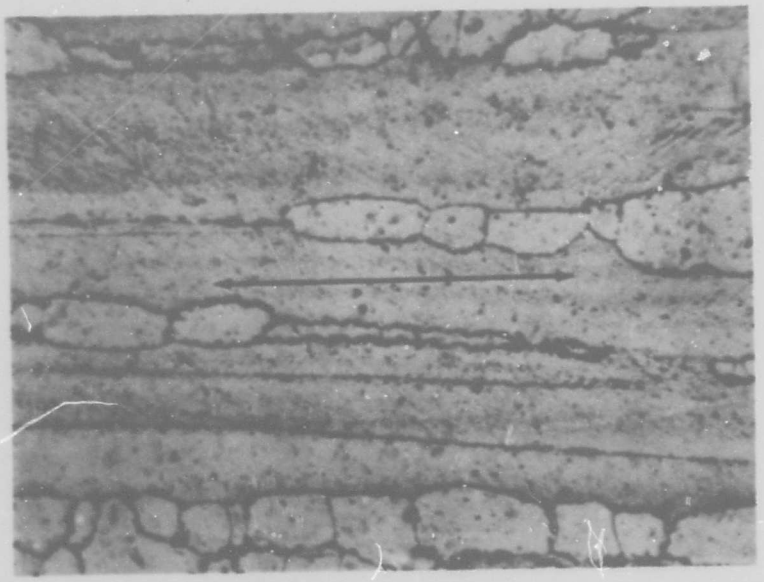
ALLOY: Cb-132M, EXTRUDED BAR VENDOR HEAT: NO. 481
PLANE: PARALLEL TO EXTRUSION DIRECTION (ARROWS) MAG: 500X
ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER

Figure 6. Microstructure of As-Received Cb-132M Alloy 5/8D Bar



ALLOY: Cb-132M, EXTRUDED BAR
PLANE: NORMAL TO EXTRUSION DIRECTION
ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER

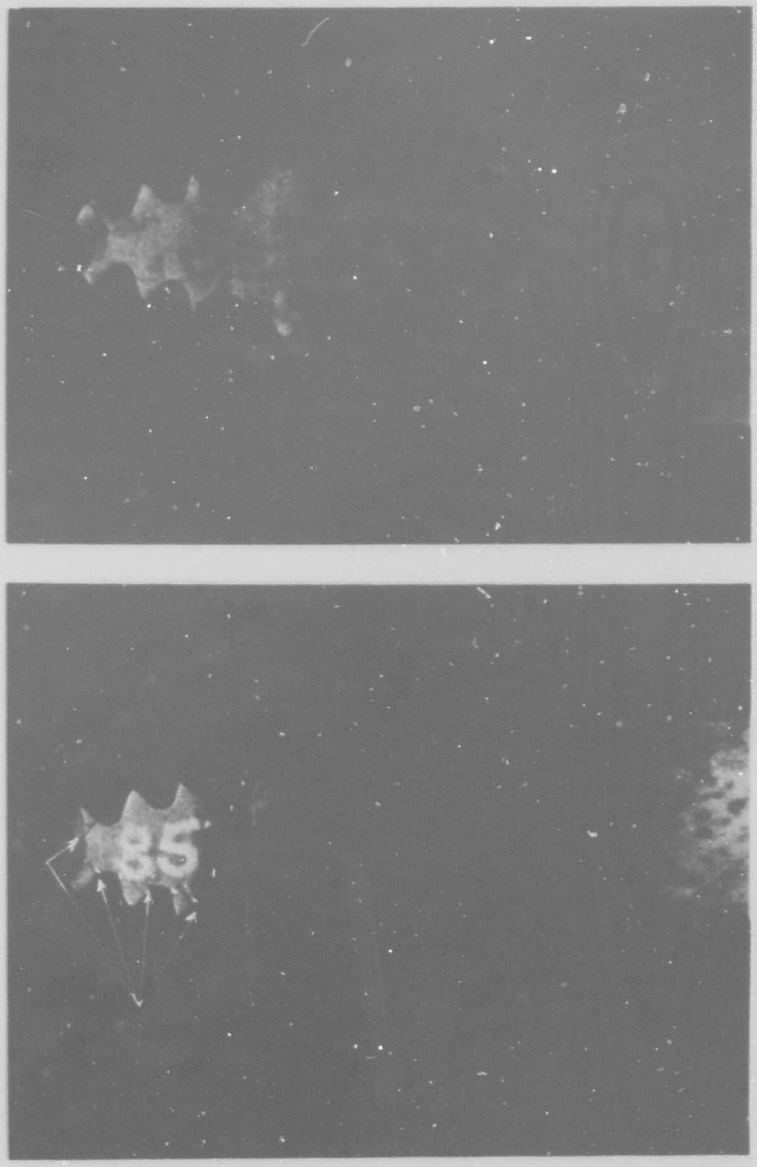
VENDOR HEAT: NO. 489
MAG: 500X



ALLOY: Cb-132M, EXTRUDED BAR
PLANE: PARALLEL TO EXTRUSION DIRECTION (ARROWS)
ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER

VENDOR HEAT: NO. 489
MAG: 500X

Figure 7. Microstructure of As-Received Cb-132M Alloy 1.25D Bar Extruded at a 5:1 Reduction Ratio



MAG: 1.5X

Figure 8 TRW TiCr-Si Coated Cb-132 Columbium Alloy Turbine Blades After 1 Hour and 40 minutes of Operation at an Airfoil Metal Temperature of 1745°F and a Root Metal Temperature of 1400°F (arrows locate fatigue cracks in blade root)

As a result of the literature survey and discussions with the AFML Project Engineer, the eight coating-substrate systems shown below were selected for the preliminary screening evaluation. As mentioned previously, the supplemental systems were not candidates for further evaluation in the Phase II and Phase III portions of the program.

System Application	Application Area	Classification	Coating/Substrate	Coating Supplier
High temperature	Vane	Original	TiCr-Si (vacuum) pack/C-129Y	TRW
		Original	Ti-CrTi-Si (triplex) pack/D-43	Sylcor
		Supplemental	SiCrTi slurry/ D-43	Sylcor
High temperature	Blade airfoil	Original	TiCr-Si (vacuum) pack/Cb-132M	TRW
		Supplemental	TiCr-Si (slip) pack/ Cb-132M	TRW
Low temperature	Blade root	Original	Sn-Al (505-F)/ Cb-132M	Sylcor
		Original	Ag-Al-Si (508-C)/ Cb-132M	Sylcor
		Original	Zn/Cb-132M	---

2.2 COATINGS APPLICATION

2.2.1 TRW TiCr-Si/C-129Y

Application of the TiCr-Si coating on the C-129Y alloy was accomplished at TRW by using a 2-cycle vacuum pack process and the parameters outlined below.

- Cycle 1: The first low-pressure cycle consisted of chromium-titanium deposition at 2200°F for 8 hours from a 60Cr-40Ti weight percent pack utilizing a potassium fluoride activator.
- Cycle 2: The second low-pressure cycle, a siliconizing process, was conducted at 2050°F for a period of 4 hours, also utilizing a potassium fluoride activator.

Metallography of the resulting coating (Figure 9) revealed a structure consisting of four distinct layers as follows: two predominantly titanium-chromium silicide outer coating layers each 1 mil thick, a 0.25-mil layer of (Cb, Ti)Cr_2 Laves phase, and a titanium-rich solid solution layer (immediately beneath the Laves phase) which visibly extended into the substrate about 0.7 mil (Ref. 25). Some Laves phase penetration into substrate grain boundaries was noted.

The metallic gray surface of the coated specimens showed fine craze cracks and randomly located "silvery" spots. The majority of the surface cracks extended only through the outermost silicide layer. However, some of these cracks penetrated through both silicide layers to the (Cb, Ti)Cr_2 Laves phase (see Figure 9). Coating coverage at the corner and edge surfaces appeared excellent.

2.2.2 TRW TiCr-Si (Vacuum) Pack/Cb-132M

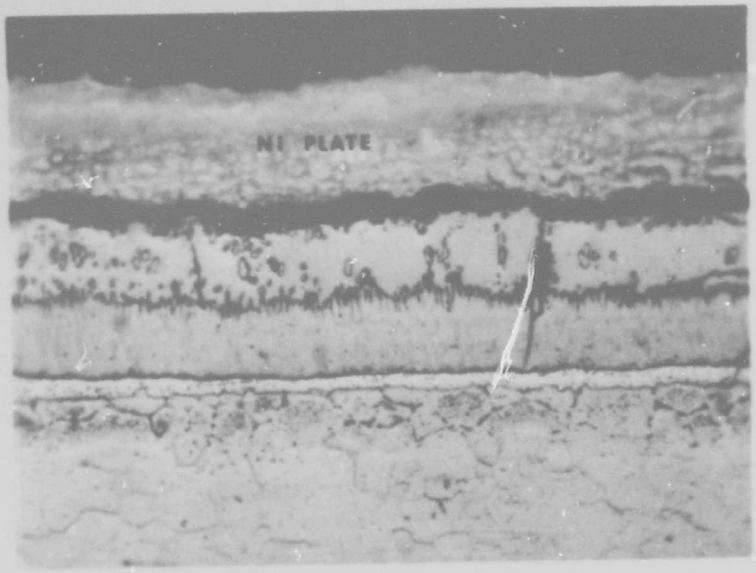
The 2-cycle vacuum pack process described for the TiCr-Si/C-129Y system was also used to apply the TiCr-Si coating to the Cb-132M alloy specimens. The coating on the Cb-132M alloy showed a structure significantly different from that observed on the C-129Y substrate. Although the coating composition can still be represented by the expression (Cb, Ti, Cr...)$_x$Si$_y$ (Ref. 25), no distinct layers of constituent-rich regions were observed (see Figure 10).

Hairline surface craze cracks, which generally penetrated through the entire coating thickness of approximately 2.5 mils, were noted. Surface coverage at specimen corners and edges was excellent.

2.2.3 TRW TiCr-Si (Slip) Pack/Cb-132M

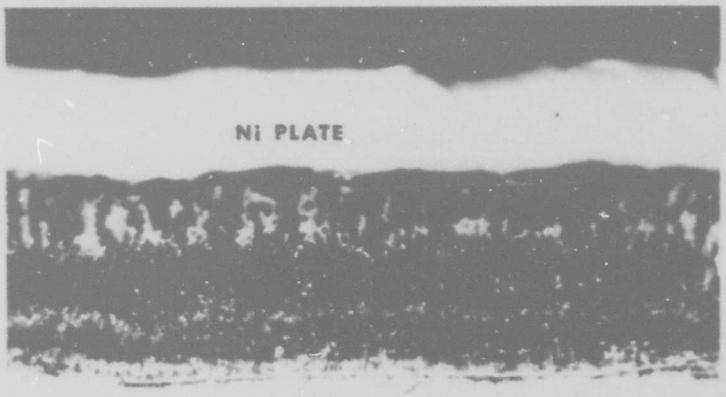
The slip pack process developed by TRW is a 2-stage slurry application of the Ti-Cr-Si coating. The first stage consisted of spray coating with a slurry of the following composition: 90 weight percent 50Cr-50Ti (prealloyed) and 10 weight percent pure titanium powder; these parts mixed with polyisobutylene binder plus 1 percent potassium fluoride in a toluol vehicle. The chromium-titanium layer was developed at 2340°F (12 hours) and 150 mm pressure of back-filled argon.

The siliconizing cycle involved application of silicon powder mixed with a binder plus 1 percent potassium fluoride in a tuluol vehicle. The silicide layer was developed at 2000°F (3 hours) and 150 mm pressure of back-filled argon.



ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER MAG: 500X

Figure 9. Typical Microstructure of the As-Applied TRW TiCr-Si (Vacuum) Pack Coating on C-129Y Alloy



ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER MAG: 500X

Figure 10. Typical Microstructure of the As-Applied TRW TiCr-Si (Vacuum) Pack Coating on Cb-132M Alloy

Two distinct coating layers were metallographically observed in addition to a 0.4-mil titanium-rich substrate diffusion layer (Figure 11). The outer coating layer appeared to have discontinuous silicon-rich regions in addition to the more continuous (Ti, Cr, Cb ...)Si_2 phase. The second layer, also predominantly (Ti, Cr, Cb...)Si_2, exhibited some penetration of the darker phase (silicon-rich) to the coating-substrate interface.

The surface appearance of the metallic-gray coating was similar to that noted for the vacuum pack process, although no macroscopic craze cracks were apparent. Coverage at edge and corner surfaces was excellent.

2.2.4 Sylcor Ti-CrTi-Si (Triplex) Pack/D-43

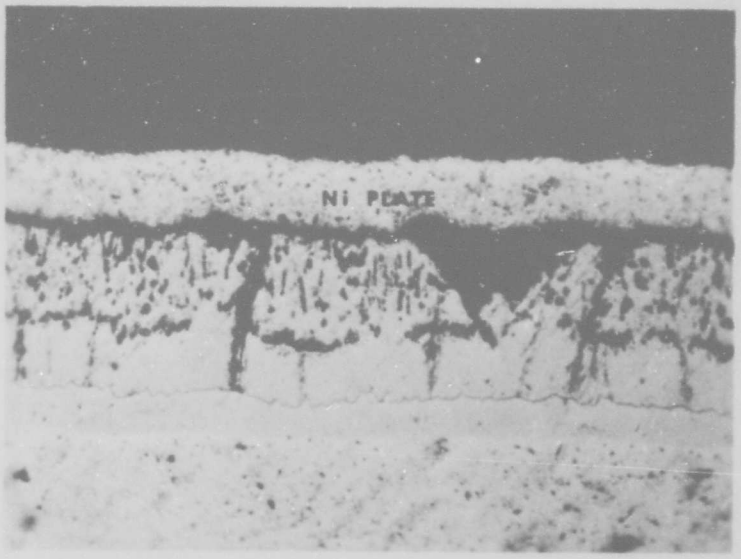
Application of the Ti-CrTi-Si (triplex) pack coating was performed at Sylcor utilizing the following parameters:

- Cycle 1: Titanizing at 2200°F for 16 hours in vacuum
- Cycle 2: Vacuum titarium-chromium deposition at 2200°F for 8 hours
- Cycle 3: Siliconizing in purified argon at 1900°F for 16 hours

Due to the complicated geometry and size of the thermal fatigue specimens, these two specimens were coated in a second batch, separate from the oxidation-erosion and ballistic impact test specimens.

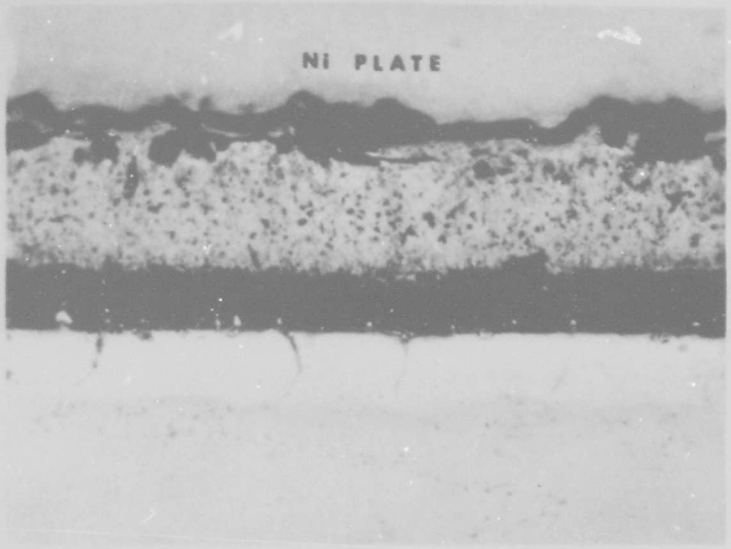
The structure of the coating deposited in the first batch (Figure 12) contained four distinct microstructural regions. The outer layer of approximately 2 mils thickness was fairly homogeneous with small dark areas of silicon-rich phase. Two darker regions totaling about 0.1 mil thickness were observed next to the coating-substrate interface. A distinct diffusion layer of titanium-rich solid solution extended into the substrate below the coating for approximately 0.9 mil.

Metallography of coupons from the batch containing the thermal fatigue specimens showed a coating consisting only of dark phase (Figure 13). Some layering of the 1.4-mil-thick coating was observed. However, these regions were not separated by sharp phase boundaries. Numerous cracks were noted in this 1.4-mil-thick layer. Beneath the coating an extensive diffusion layer of titanium-rich solid solution was observed containing some precipitate of type (Cb, Ti)Cr_2 Laves phase (Ref. 10, 25). Elevated-temperature exposure resulted in continued precipitation of type (Cb, Ti)Cr_2 Laves phase (Figure 14). Precipitation was concentrated at the coating-diffusion-zone boundary.



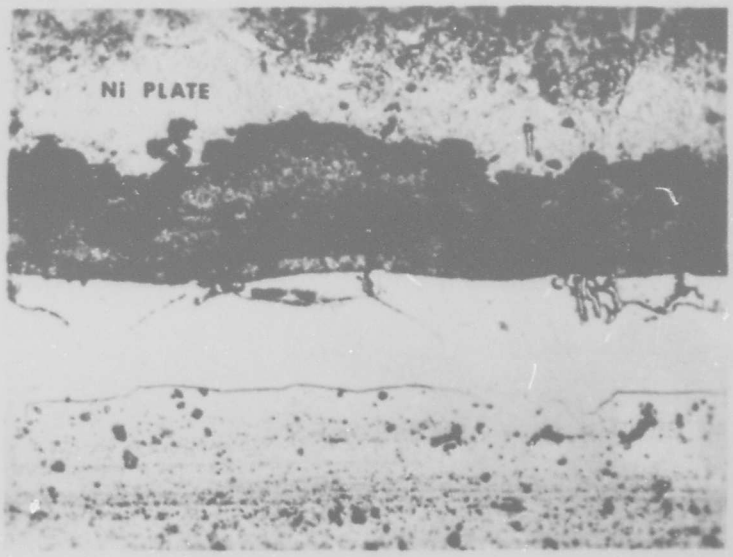
ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER MAG: 500X

Figure 11. Typical Microstructure of the As-Applied TRW TiCr-Si (Slip) Pack Coating on Cb-132M Alloy



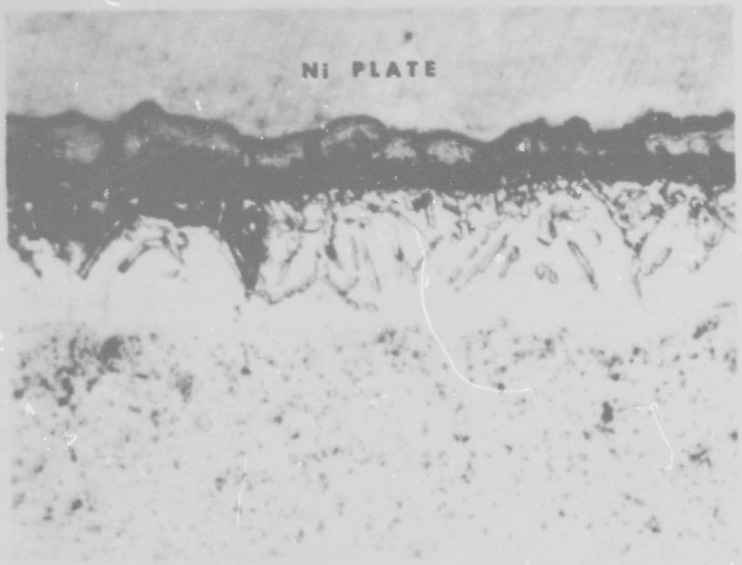
ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER MAG: 500X

Figure 12. Typical Microstructure of the As-Applied Sylcor Ti-CrTi-Si (Triplex) Pack Coating on D-43 Alloy (Batch 1)



ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER MAG: 500X

Figure 13. Typical Microstructure of the As-Applied Sylcor Ti-CrTi-Si (Triplex) Pack Coating on D-43 Columbium Alloy (Batch 2)



ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER MAG: 500X

Figure 14. Typical Microstructure of the Sylcor Ti-CrTi-Si (Triplex) Pack Coating on D-43 Alloy After Elevated Temperature Exposure (Batch 2)

Coverage by the dull-gray coating at corner and edge surfaces appeared good. The surfaces of the specimens in the first batch were very uniform. Bluish-gray discolorations were observed on those specimens coated in the second batch.

2.2.5 Sylcor SiCrTi Slurry/D-43

The single-cycle application of the Sylcor R-512-A coating was accomplished by dipping the specimens in a slurry of Si-20Cr-5Ti and vehicle. Due to the geometrical complexity of the thermal fatigue specimens, dipping of the airfoil portion was followed by local spray application in the shroud areas. The coating structure was developed in a vacuum diffusion treatment at 2550°F for a period of 1 hour.

Metallography of the resulting coating revealed a structure consisting of three distinct layers (Figure 15). The outer layer, about 1.8 mils thick, contained a discontinuous second phase. The second layer of approximately 0.6 mil thickness was columnar in structure. The discontinuous nature of the interface between these two regions suggests that the second phase in the outer coating layer is possibly of this silicide composition. The diffusion layer of 0.4 mil was very distinct and uniform in thickness. Numerous cracks were observed, some of which penetrated from the coating surface through the diffusion layer.

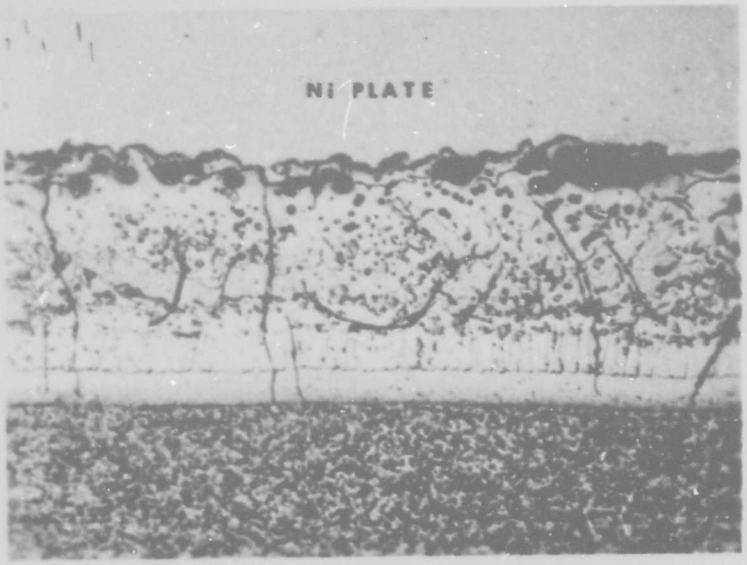
The surfaces of the coated specimens appeared very homogeneous with no visible cracks. Excellent coverage of specimen corner and edge surfaces was noted.

2.2.6 Sylcor Sn-Al (505-F)/Cb-132M

The spray application of the Sn-Al coating was followed by a vacuum diffusion treatment to develop a coating of approximately 5.5 mils thickness (Figure 16). Metallographic examination revealed four distinct coating layers: an outer layer consisting of a continuous tin-rich compound with $MoAl_3$ and $CbAl_3$ particles, a second layer predominantly $CbAl_3$, and two diffusion layers consisting of lower aluminide compounds (Ref. 10). Coverage of specimen surfaces was excellent.

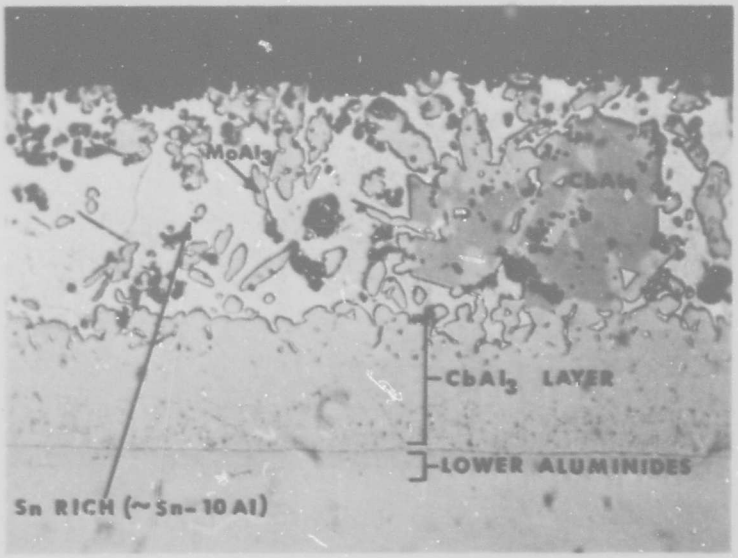
2.2.7 Sylcor Ag-Al-Si (508-C)/Cb-132M

Application of the Ag-Al-Si coating was similar to the procedure utilized for the Sn-Al coating. A coating of approximately 5.0 mils thickness was developed (Figure 17). The outer coating layer, consisting of an AgAl continuous phase, also contained particles of Si, $MoSi_3$ and Ag (Ref. 10). A



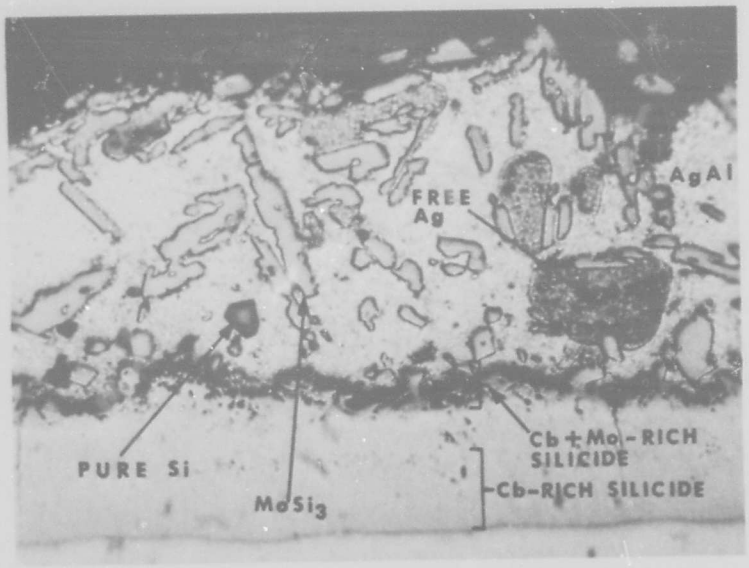
ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER MAG: 500X

Figure 15. Typical Microstructure of the As-Applied Sylcor SiCrTi Slurry Coating on D-43 Alloy



UNETCHED MAG: 500X

Figure 16. Typical Microstructure of the As-Applied Sylcor Sn-Al Coating on Cb-132M Alloy



UNETCHED MAG: 500X

Figure 17. Typical Microstructure of the As-Applied Sylcor Ag-Al-Si Coating on Cb-132M Alloy

(Cb+Mo)-rich silicide layer was observed directly beneath the outer coating layer and a columbium-rich silicide layer was noticeable adjacent to the coating-substrate interface.

As in the case of the Sylcor Sn-Al coating, coverage of the specimen corners and edges was excellent.

2.2.8 Zinc-Base Coating/Cb-132M

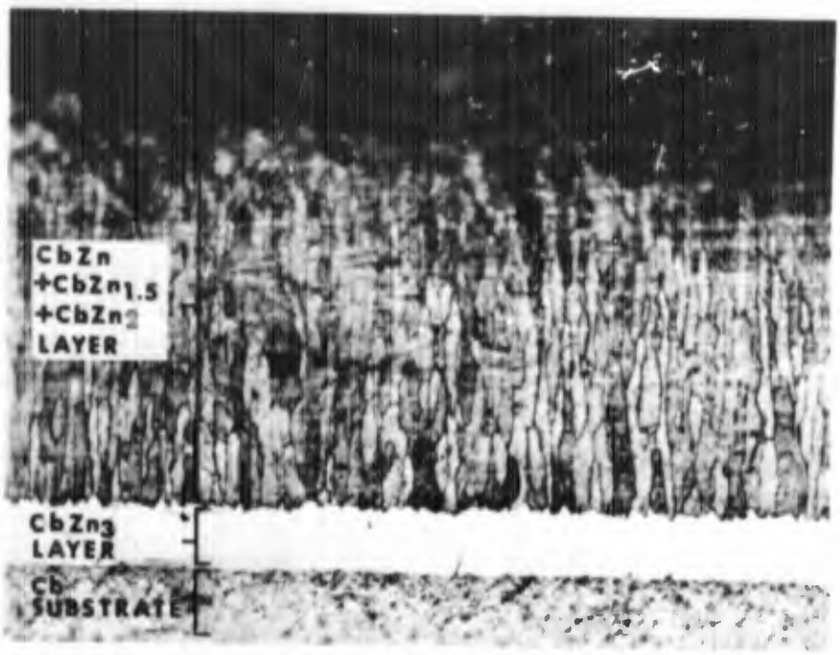
The zinc-base coating was applied to Cb-132M alloy by Pratt & Whitney Aircraft for evaluation as a system for blade root application. Preliminary trials involved application of the coating on pure columbium metal. Coating microstructure (See Figure 18) similar to those reported in the literature (Ref. 12, 13, 26) were developed. Coating thicknesses varied from 4 to 8 mils after thermal conditioning in air at 1600°F for 16 hours. Hot-dip application for 2 hours in an 1100°F zinc bath resulted in the most homogeneous coatings.

Electroplated specimens developed extremely uniform coatings, but random areas exhibited separation of the coating from the metal substrate (Figure 19). Due to this behavior, this application technique was not considered further.

A preliminary zinc hot-dipping trial conducted on Cb-132M alloy using the above established conditions resulted in extremely thin coatings (0.2 to 0.8 mil). Increasing the hot-dipping temperature from 1100°F to 1150°F and the time from 2 hours to 5 hours brought the coating thicknesses to within an acceptable 3- to 9-mil range.

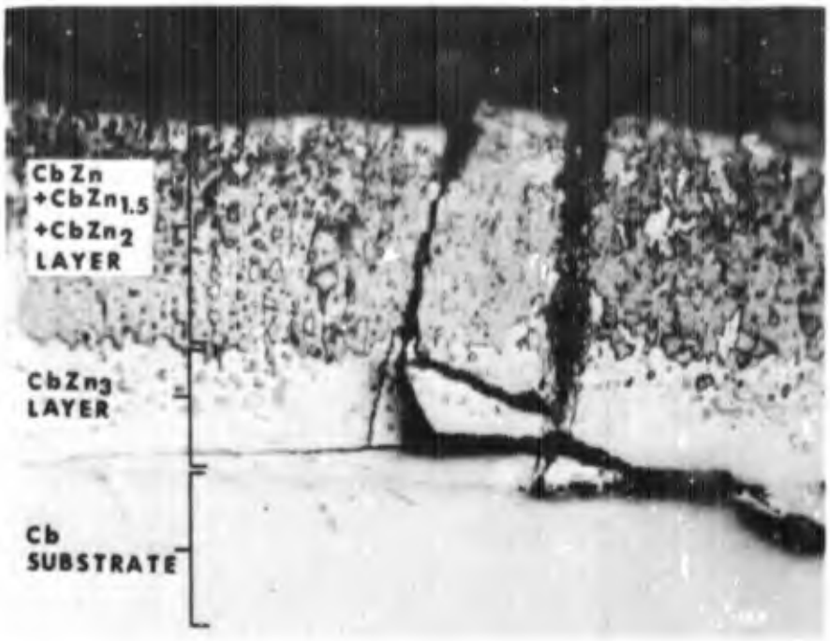
The zinc coating was subsequently applied to the Cb-132M alloy Phase I test specimens. Metallographic examination of control coupons (Figure 20) showed that although the $CbZn_3$ layer at the coating-substrate interface appeared normal, the outer coating layer of $CbZn+CbZn_{1.5}+CbZn_2$ differed significantly from the corresponding layer on the pure columbium substrate. A columnar-type development of this layer was evident on the pure columbium substrate, while on the Cb-132M alloy this layer was laminar.

Although application parameters were carefully reproduced during successive coating runs, catastrophic oxidation occurred during 1600°F conditioning of Cb-132M specimens in five of the nine specimen batches coated under this contract. Extreme inconsistency of coating application and test results led to the elimination of the Zn/Cb-132M system with approval of the Air Force Materials Laboratory Project Engineer.



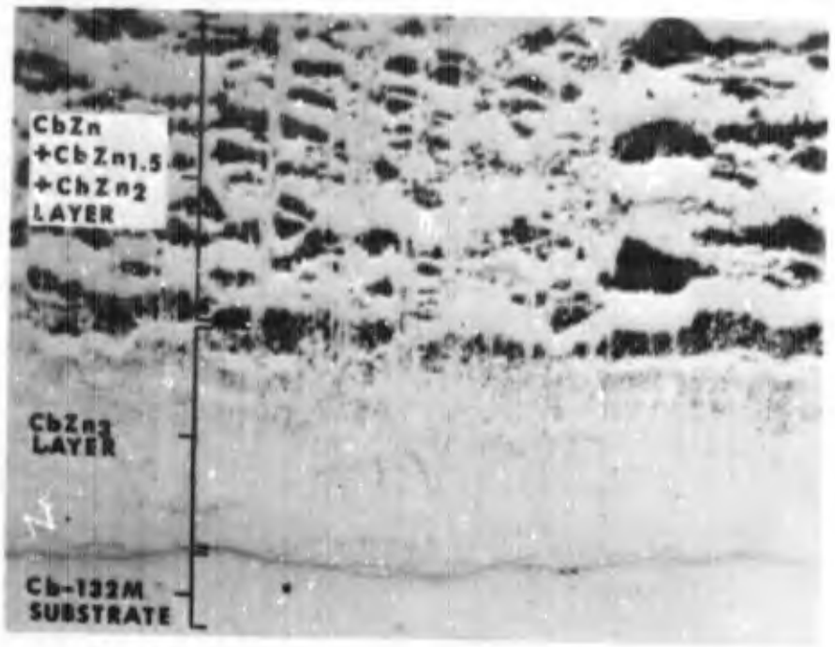
ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER MAG: 325X

Figure 18. Typical Microstructure of the Hot-Dipped Zinc Coating Developed on Pure Columbium



ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER MAG: 325X

Figure 19. Microstructure of Electroplated Zinc Coating on Pure Columbium Showing Coating-Substrate Separation



ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER MAG: 325X

Figure 20. Typical Microstructure of the Hot-Dipped Zinc Coating Developed on Cb-132M Alloy

2.3 EVALUATION TESTING, HIGH-TEMPERATURE SYSTEMS

2.3.1 Test Procedures

Performance testing of the high-temperature systems for blade airfoil and/or vane application included three complementary preliminary screening tests: oxidation-erosion, thermal fatigue, and ballistic impact. The purpose of these tests was to obtain qualitative data from which potential comparisons could be assessed and made between candidate systems for a given application. Therefore, in cases of promising but inadequate performance, modifications could be made (Phase II, Coating Improvement) or, in the case of outstanding performance, a system could be forwarded directly to Phase III, Advanced Evaluation, for the generation of quantitative data. The parameters of each test are discussed below.

2.3.1.1 Oxidation-Erosion Procedure

The erosion test specimens were machined from 0.5-inch-diameter bar stock to the required "airfoil" shape (Figure 21). Testing was conducted in a combusted JP-5 fuel and air stream at 2200° and 2400°F using the test rig shown in Figure 22. Specimens were rotated at 1750 rpm in groups of eight for temperature uniformity. Temperature during testing was determined by an optical pyrometer and checked at 30-minute intervals. Coating-substrate systems not exhibiting failure after 100 hours at 2200°F were tested further at 2400°F. Specimens were visually inspected and weighed after each 20 hours of testing. Four specimens were tested for each system evaluated.

2.3.1.2 Thermal Fatigue Procedure

Blade alloy thermal fatigue specimens were forged by TRW, Inc. from 0.5-inch-diameter bar stock (Figure 23). During testing, the specimens were rotated at 1850 rpm in groups of 12 for temperature uniformity. In cases where less than 12 specimens were being tested, dummy blanks were used to complete the set for rig-balancing purposes. The test rig is shown in Figure 24. Two specimens per system were tested.

The vane alloy thermal fatigue specimens, illustrated in Figures 25 and 26, were fabricated from sheet and bar material. The airfoils were cold-formed from 0.050-inch-thick sheet with a leading edge bend radius of two and one-half times the sheet thickness. Joining of the trailing edge was accomplished by manual TIG (tungsten inert gas) welding with parent metal filler rod in an argon atmosphere dry box. Atmosphere dew point was maintained at -40°F or lower. The resultant joint was blended to the

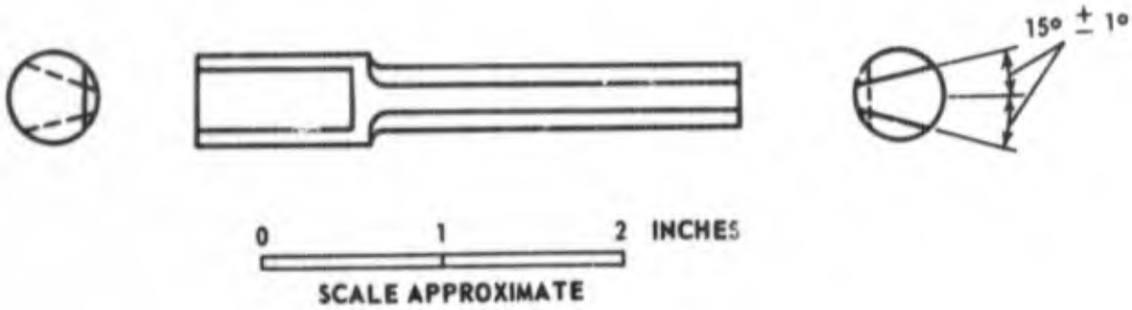


Figure 21. Erosion Bar Specimen Configuration for Vane and Blade Alloy Oxidation-Erosion Tests

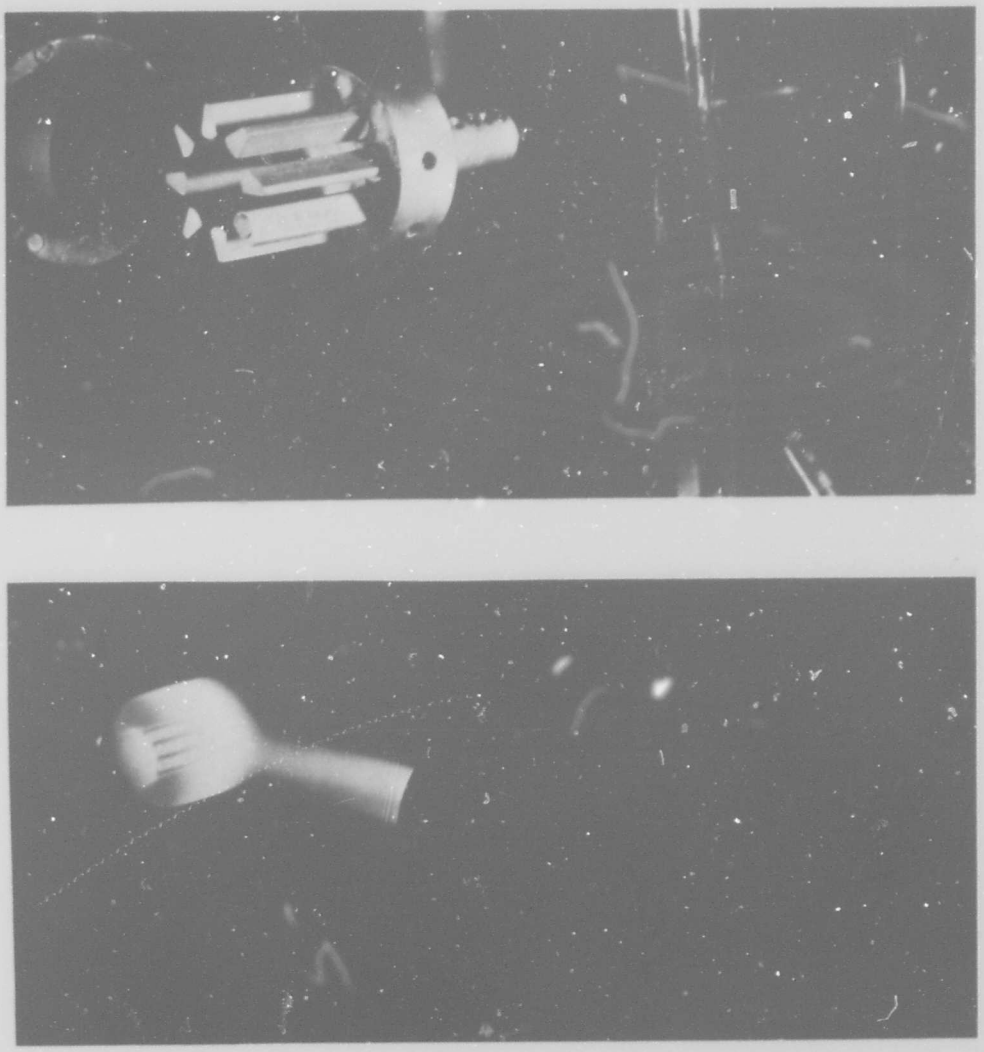


Figure 22. Burner Test Rig for Oxidation-Erosion Studies

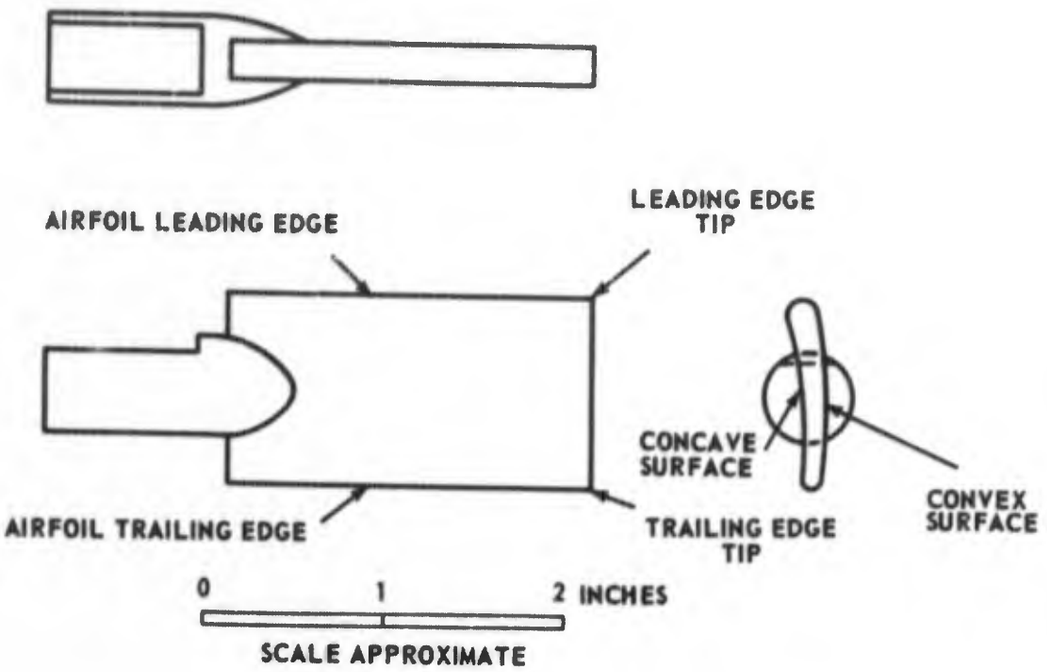


Figure 23. Forged Paddle Specimen Configuration for Thermal Cycling Tests

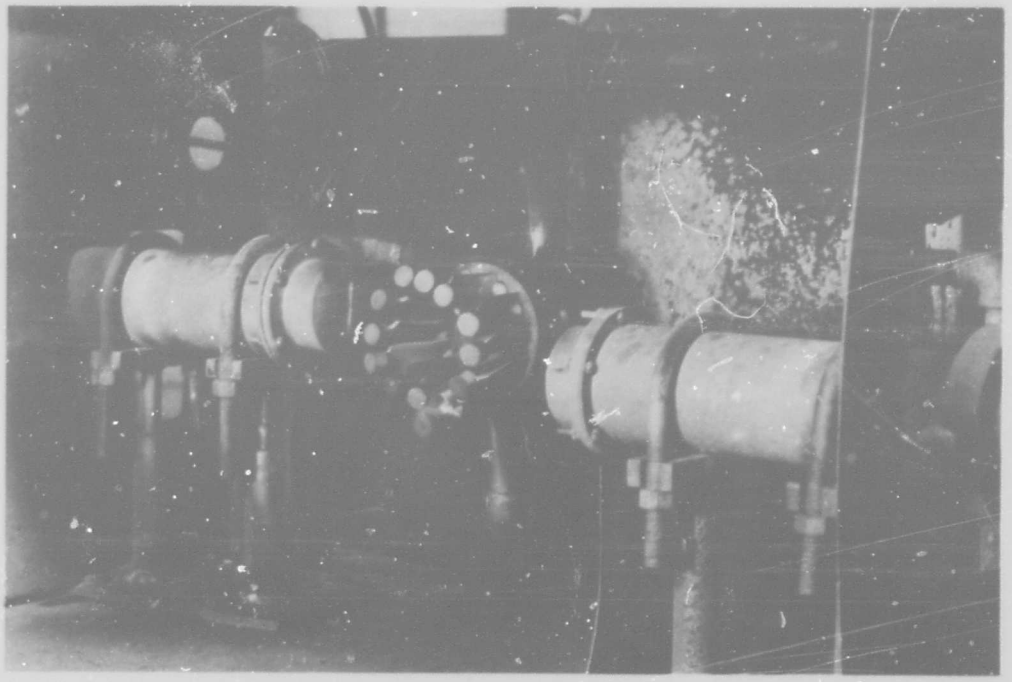


Figure 24. Thermal Fatigue Bow Rig Showing Position of Specimens in Relation to Burner Nozzles

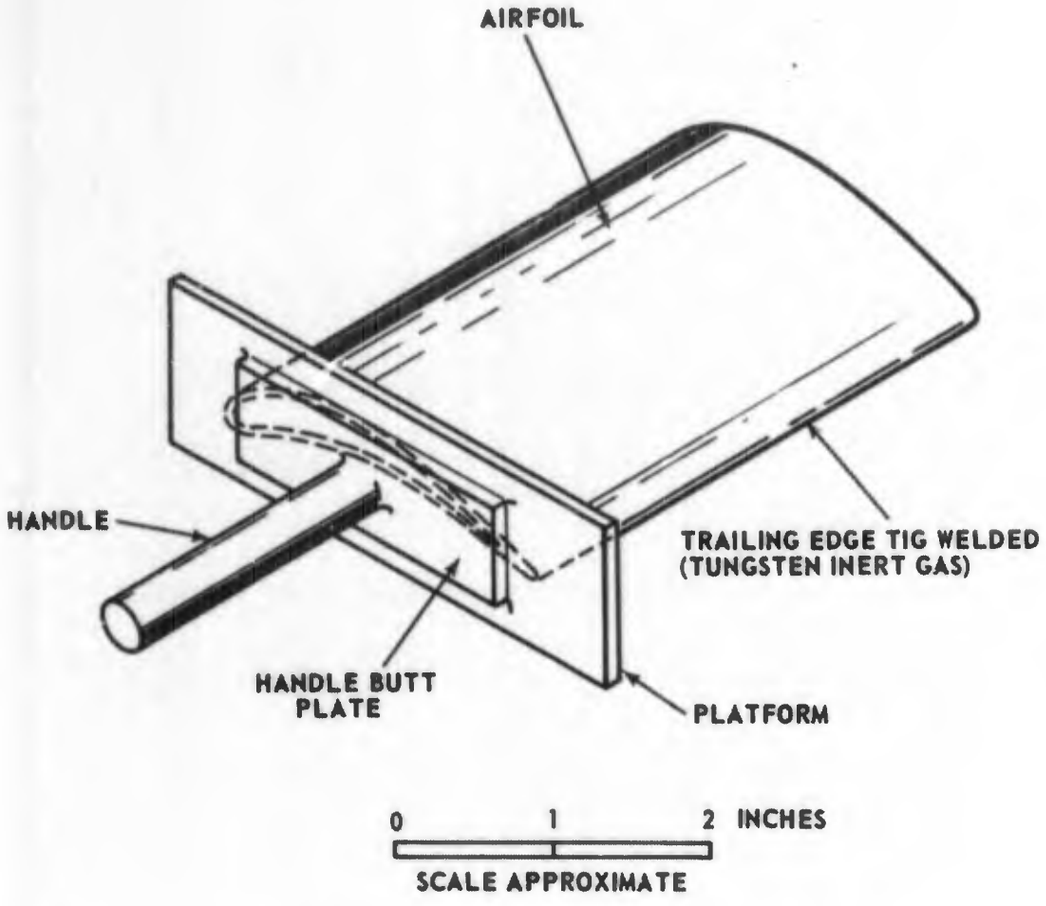
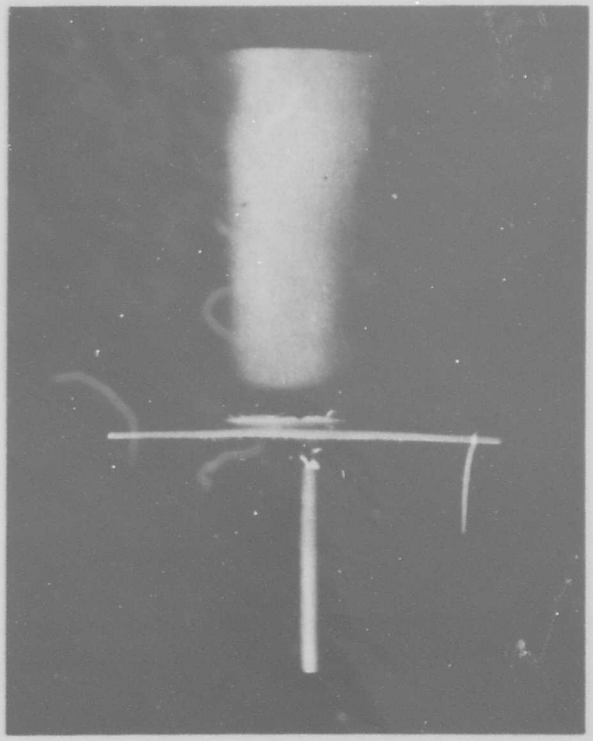


Figure 25. Vane Airfoil Specimen Configuration for Thermal Cycling Tests



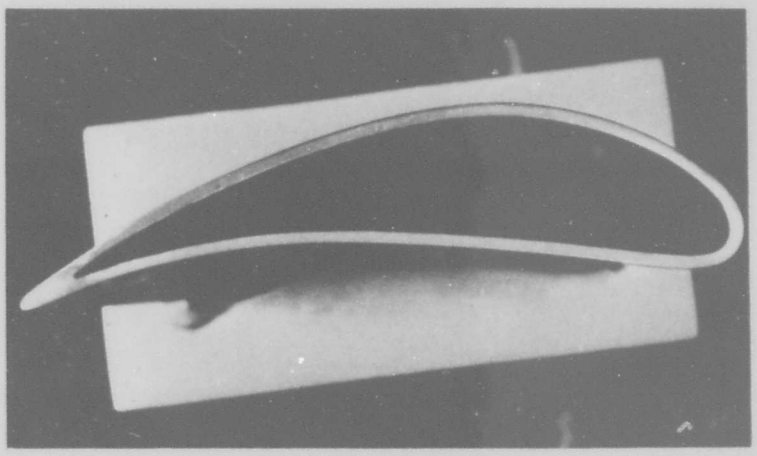


Figure 26. Typical Uncoated Vane Thermal Fatigue Specimen

desired trailing edge contour. Remaining details of the completed assembly were similarly joined by welding. The specimens were tested utilizing the single-vane test rig shown in Figure 27. Duplicate specimens were tested.

Test parameters for both the blade and vane alloy specimens consisted of heating to the desired peak temperature in combusted JP-5 fuel and air stream, stabilization at this temperature for 30 minutes, and cycling by alternate heating to the peak temperature and cooling to approximately 200°F with an ambient temperature air blast. Parts were visually inspected at the end of each 100 or 200 cycles. On resumption of testing at the initial elevated temperature, a 5-minute stabilization was used for the blade airfoil specimens and a 30-minute stabilization for the vane alloy specimens. Each cycle consisted of 1 minute at the elevated temperature followed by a 30-second ambient temperature air blast of 60-65 psi. Temperatures were determined with an optical pyrometer.

2.3.1.3 Ballistic Impact Procedure

The ballistic impact tests were conducted on 0.050x1x2-inch coupons for the vane systems and 0.125x0.5x2-inch coupons for the blade airfoil system. During testing, the specimens were mounted in grips utilizing transite coverings to avoid local coating damage. A 0.75-gram steel pellet was fired at velocities of 200, 500, and 900 feet per second at a point one-half inch above the grips to avoid bending moment variations between tests. Heating was accomplished with an acetylene torch with impact occurring at the end of a 10-minute stabilization period. Specimens were impacted at room temperature, 2200°F, and 2400°F. Temperature measurements were made with an optical pyrometer. To determine the amount of resulting coating damage, the specimens were subjected to 2200°F static-air oxidation after impact.

2.3.2 Test Results

2.3.2.1 Oxidation-Erosion Results

As previously discussed in the Literature Survey section of this report, the TRW TiCr-Si coating on D-43 alloy was evaluated by Pratt & Whitney Aircraft prior to this program. Since in-house data on the oxidation-erosion performance of this system was available, additional TRW TiCr-Si/D-43 test specimens were included during some of the Phase I (and Phase II) tests in this program as test standards.

TRW TiCr-Si (Vacuum) Pack/C-129Y. The maximum protective life of the TRW TiCr-Si coating on the C-129Y substrate was 100 hours at 2200°F plus 40 hours at 2400°F. Failures by local oxidation first appeared at the end of 100 hours at 2200°F. Prior to failure, the specimens developed a coarse surface texture resulting from oxide formation within surface craze

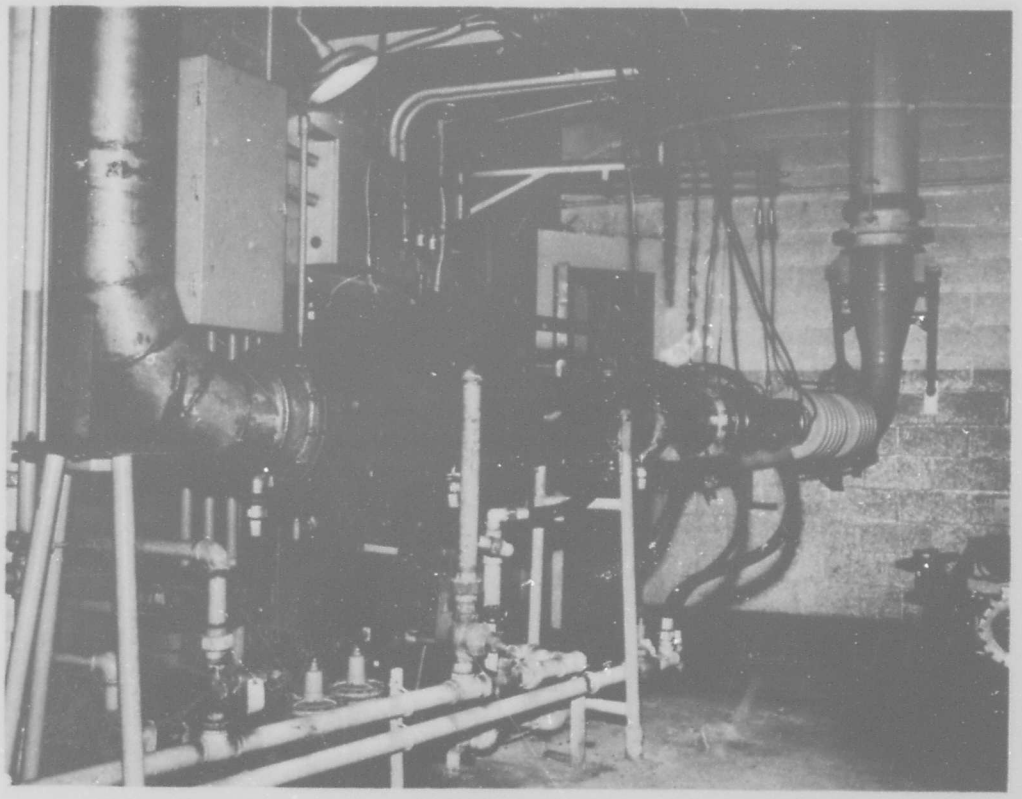


Figure 27. Single-Vane Thermal Fatigue Test Rig

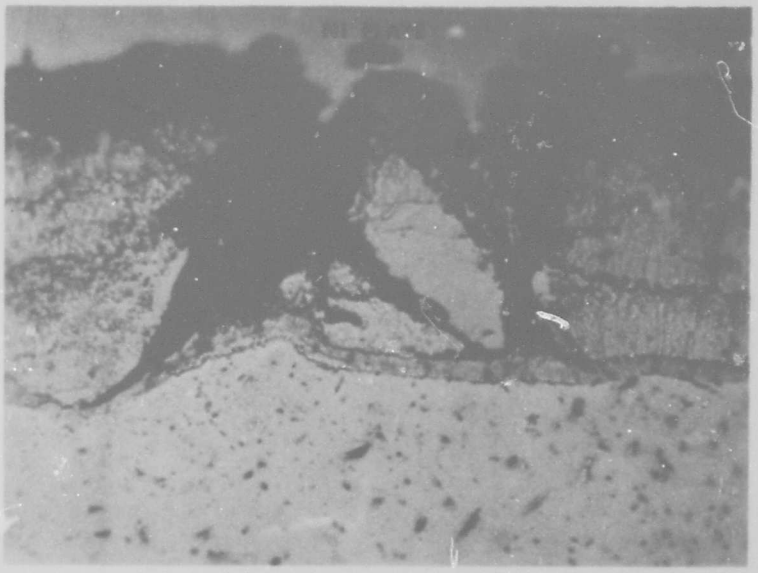
cracks (Figure 28), which eventually led to localized spalling of the coating. The progressive failure of these specimens as observed metallographically was similar to that described in paragraph 2.3.2.2, Thermal Fatigue Results, TRW/C-129Y system.

Catastrophic low-temperature (1500°-2000°F) oxidation was experienced on TRW coated C-129Y alloy. During testing, the specimens were secured in a circular holder (Figure 22). The holder or grip portions of the specimens generally operate 200° to 500°F cooler than the airfoils, the temperature difference depending on the airfoil temperature. After 40 test hours at an airfoil temperature of 2200°F, considerable oxidation occurred in this area and increased during continued testing (Figure 29). It was felt that this behavior may have resulted from a detrimental reaction between the coating and the nickel-base-alloy specimen holder. However, this attack was not noted on TRW coated Cb-132M and the D-43 alloy specimens. Wurst and Cherry (Ref. 2) reported similar results for the TRW TiCr-Si coating on C-129Y alloy during cyclic oxidation testing at 1600°F.

TRW TiCr-Si (Vacuum) Pack/Cb-132M. This coating-substrate system demonstrated an oxidation-erosion life of 100 hours at 2200°F plus 40 hours at 2400°F. No premature failures were observed. During testing, the surface of the coating developed yellowish-white discolorations on flat airfoil surfaces and gray-to-black discolorations on the leading and trailing edges. Failure occurred by localized oxidation on the leading edge, trailing edge, and flat airfoil surfaces. These oxide formations extended out of surface craze cracks (Figure 30). The progressive deterioration of the coating at these craze cracks was similar to that mentioned previously for the TRW TiCr-Si/C-129Y system. General specimen appearance after failure was good in that failure of the entire coated airfoil surface had not occurred.

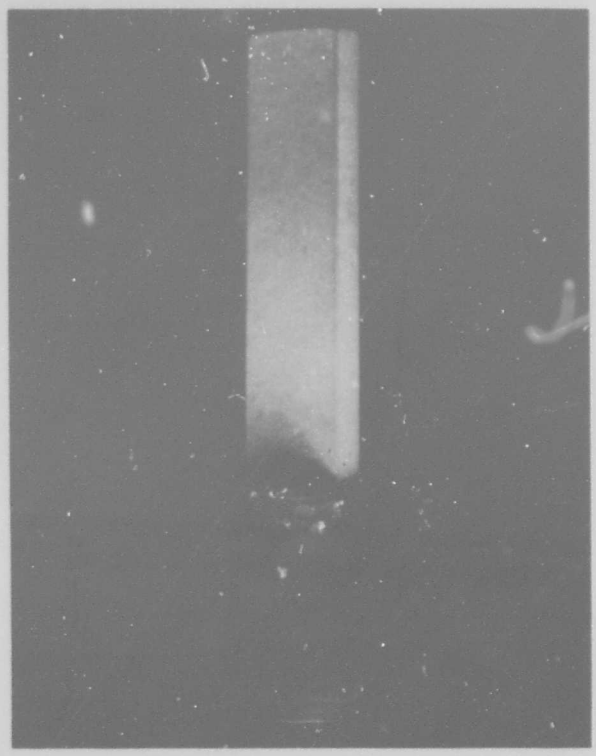
TRW TiCr-Si (Slip) Pack/Cb-132M. During oxidation-erosion testing, failures were observed for the TRW slip pack coating from 60 hours at 2200°F to 100 hours at 2200°F plus 40 hours at 2400°F. These failures were localized on the trailing edge surface and the airfoil tip (Figure 31). Microexamination of these spot oxide formations revealed sufficient substrate oxidation at these locations (Figure 32). Continued testing resulted in the formation of additional oxidation progressing from the lower-temperature tip toward the center of the airfoil (operating at a slightly higher temperature). Metallographic examination revealed oxide penetration (Figure 33).

Sylcor Ti-CrTi-Si (Triplex) Pack/D-43. This coating-substrate system exhibited an oxidation-erosion life of from 80 hours at 2200°F to 100 hours at 2200°F plus 20 hours at 2400°F. During testing, yellowish-brown discoloration of the gray coating was observed on the airfoil surfaces. Failure occurred by local oxidation at the leading and trailing edge surfaces. In



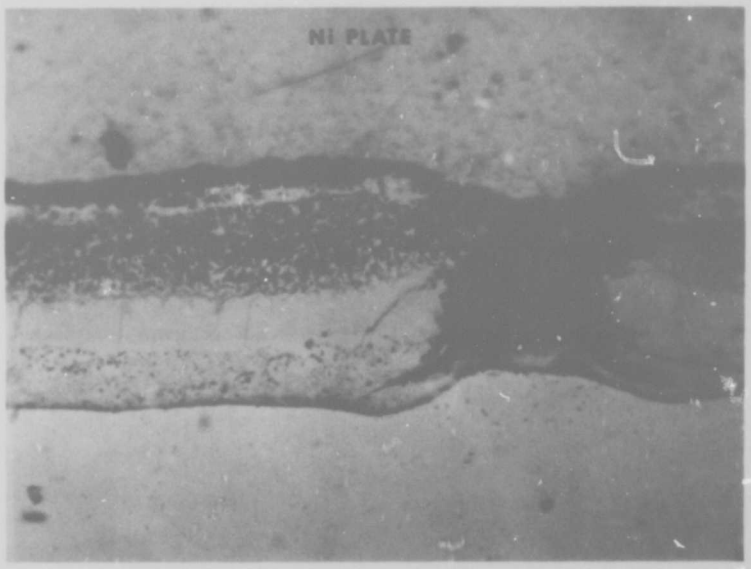
ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER MAG: 500X

Figure 28. Microstructure of TRW TiCr-Si (Vacuum) Pack Coating on C-129Y Columbium Alloy After 100 Hours of Oxidation-Erosion Testing at 2200°F



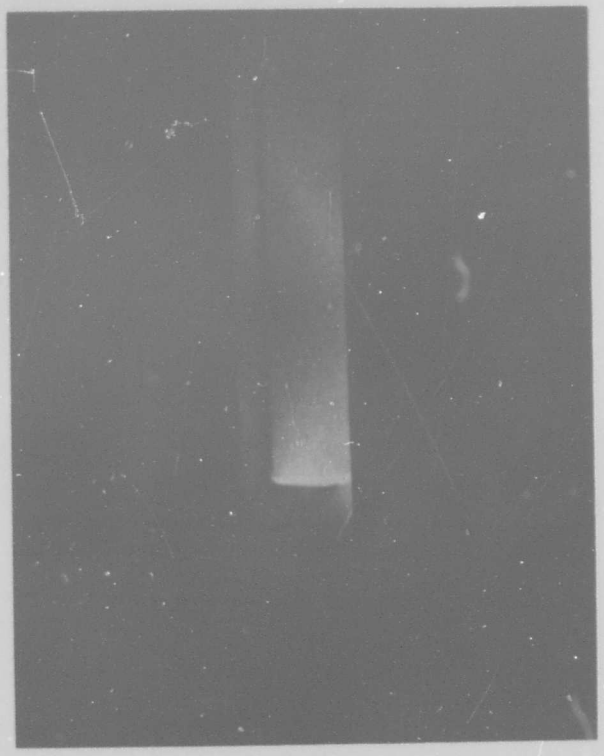
MAG: 1.25X

Figure 29. Typical Surface Appearance of the TRW TiCr-Si (Vacuum) Pack Coating on C-129Y Alloy After 100 Hours Oxidation-Erosion Testing at 2200°F



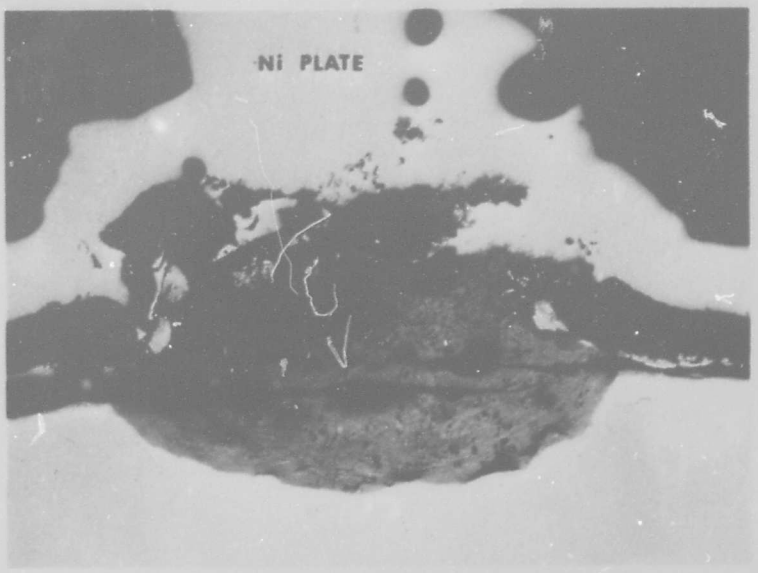
UNETCHED MAG: 500X

Figure 30. Microstructure of TRW TiCr-Si (Vacuum) Pack Coating on Cb-132M Columbium Alloy After Oxidation-Erosion Testing for 100 Hours at 2200°F Plus 40 Hours at 2400°F



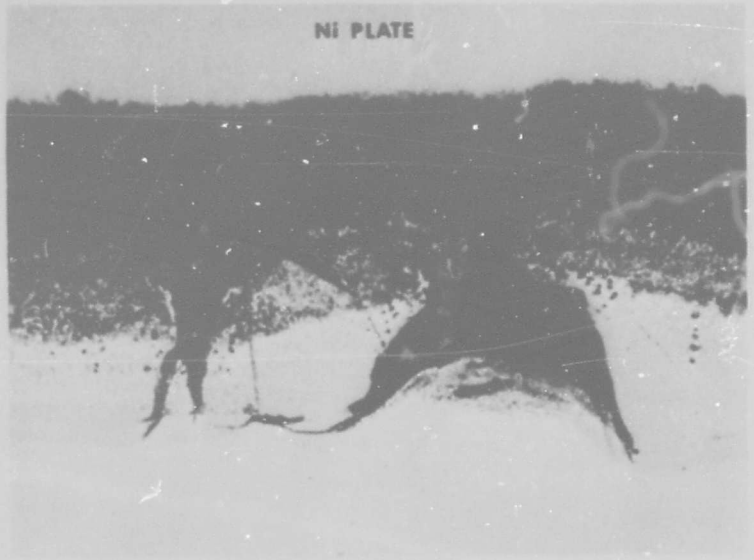
MAG: 1.25X

Figure 31. Typical Surface Appearance of the TRW TiCr-Si (Slip) Pack Coating on Cb-132M Alloy After 40 Hours Oxidation-Erosion Testing at 2200°F (note spot failures developing at airfoil tip)



ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER MAG: 100X

Figure 32. Microstructure of TRW TiCr-Si (Slip) Pack Coated Cb-132M Columbium Alloy in Area of Typical Failure During Oxidation-Erosion Testing.



ETCH: 60% LACTIC ACID, 20% HYDROFLUORIC ACID, 20% NITRIC ACID MAG: 500X

Figure 33. Typical Microstructure of the TRW TiCr-Si (Slip) Pack Coating on Cb-132M Alloy After Oxidation-Erosion Testing for 100 Hours at 2200°F

areas away from these point failures, the coating appeared completely intact and in satisfactory condition; i.e., failure of the airfoil surfaces was more confined for Sylcor/D-43 system than for the TRW/C-129Y and the TRW/Cb-132M systems. Microexamination (Figure 34) showed craze crack propagation extending through the coating. Oxidation progressed along the coating-diffusion-zone interface during continued testing.

Sylcor SiCrTi Slurry/D-43. Oxidation-erosion performance of the Sylcor SiCrTi slurry coating was markedly different from that of the other coatings. After 20 hours at 2200°F, the coating appeared to be eroding along the airfoil corners and edges (Figure 35). This behavior continued for approximately 60 test hours, resulting in increasing exposure of a dark underlayer. Although this behavior apparently resulted from coating erosion, the protective qualities of the coating did not appear to be detrimentally affected (Figure 36).

The life of the coating was from 100 hours at 2200°F plus 20 hours at 2400°F to 100 hours at 2200°F plus 40 hours at 2400°F. Failure was by extensive oxidation of the trailing edge surface. Typical structure of the coating after testing is presented in Figure 37.

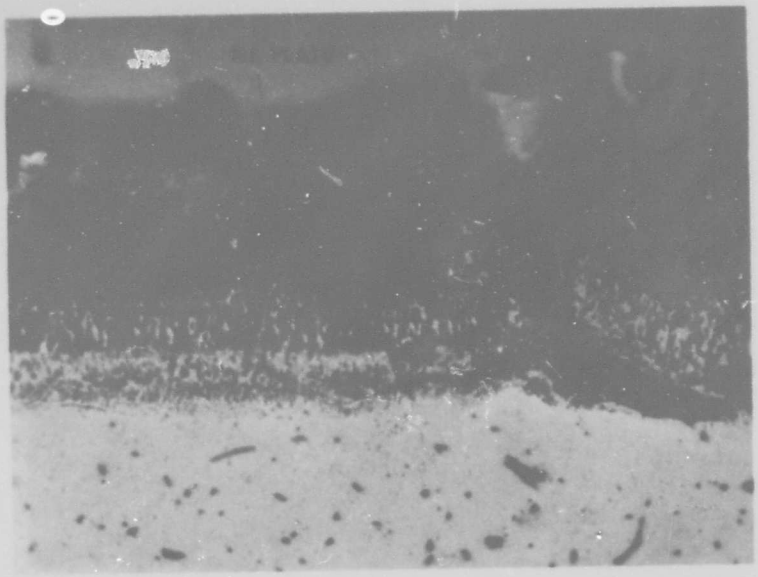
2.3.2.2 Thermal Fatigue Results

TRW TiCr-Si (Vacuum) Pack/C-129Y. The first of two TRW TiCr-Si (vacuum) pack coated C-129Y alloy specimens was tested for 600 thermal fatigue cycles at 2200°F. Although no failure was noted (see Figure 38) the specimen was examined metallographically to obtain information on the coating structure at this stage of testing. Surface craze cracks resulting from coating-substrate thermal expansion differences were visible. These cracks, however, had not penetrated the $(Cb,Ti)Cr_2$ Laves phase into the C-129Y alloy substrate (Figure 39).

The second TRW TiCr-Si coated C-129Y alloy specimen was thermal fatigue tested as follows:

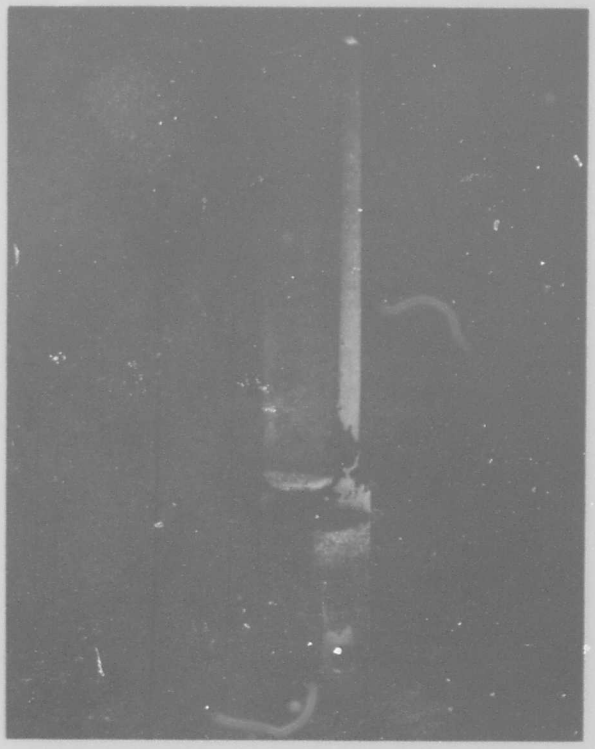
- 600 cycles at 2200°F, followed by
- 400 cycles at 2400°F, followed by
- 400 cycles at 2500°F

The additional 800 cycles at the elevated temperatures extended flow of a dark surface phase at the leading and trailing edges (Figure 40). The airfoil surface was analyzed by X-ray diffraction to determine whether any sulfur or sulfur-rich combustion products from the JP-5 fuel were reacting with the silicides in the coating to form the dark surface phase. No evidence of sulfur compounds was detected. The dark, glassy phase was



ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER MAG: 500X

Figure 34. Microstructure of Sylcor Ti-CrTi-Si (Triplex) Pack Coating on D-43 Columbium Alloy After 100 Hours of Oxidation-Erosion Testing at 2200°F

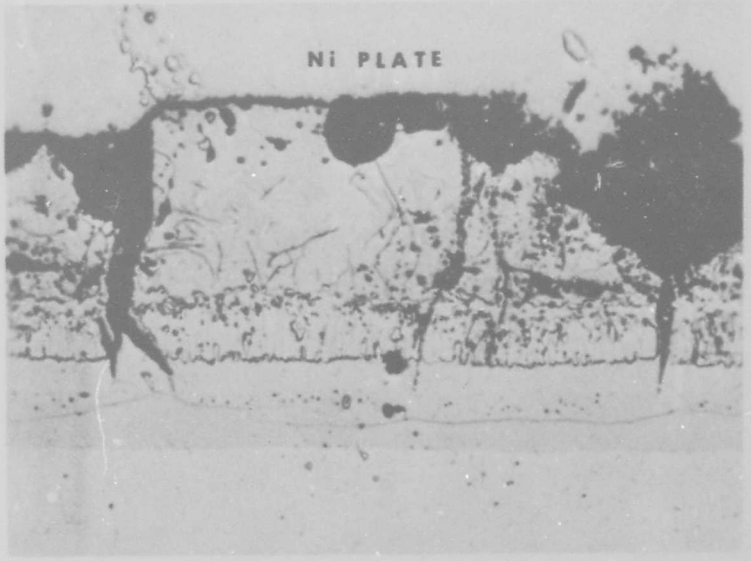


MAG: 1.2X

Figure 35. Typical Surface Appearance of the Sylcor SiCrTi Slurry Coating on D-43 Alloy After 20 Hours of Oxidation-Erosion Testing at 2200°F

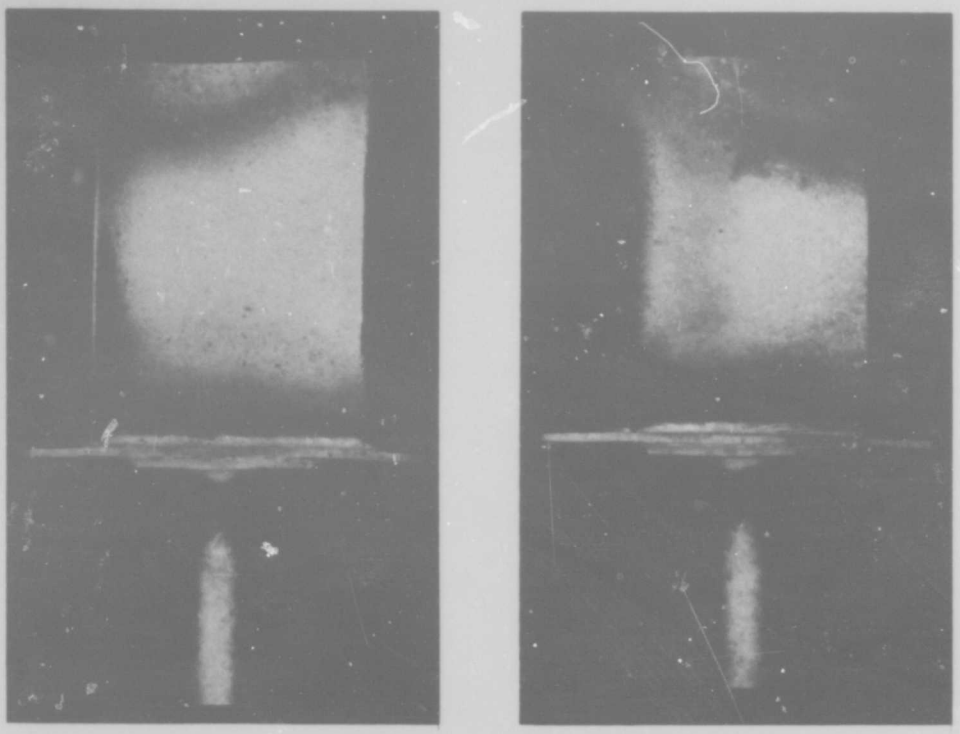
MAG: 1.2X

Figure 36. Typical Surface Appearance of the Sylcor SiCrTi Slurry Coating on D-43 Alloy After 100 Hours of Oxidation-Erosion Testing at 2200°F



ETCH: 60% LACTIC ACID, 20% HYDROFLUORIC ACID, 20% NITRIC ACID MAG: 500X

Figure 37. Microstructure of the Sylcor SiCrTi Slurry Coating on D-43 Alloy After Oxidation-Erosion Testing for 100 Hours at 2200°F Plus 40 Hours at 2400°F

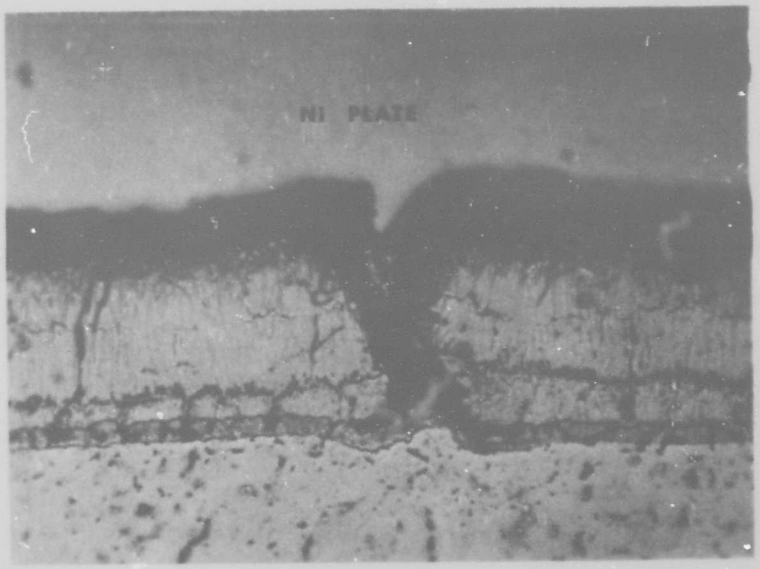


CONCAVE AIRFOIL SURFACE

CONVEX AIRFOIL SURFACE

MAG: 0.7X

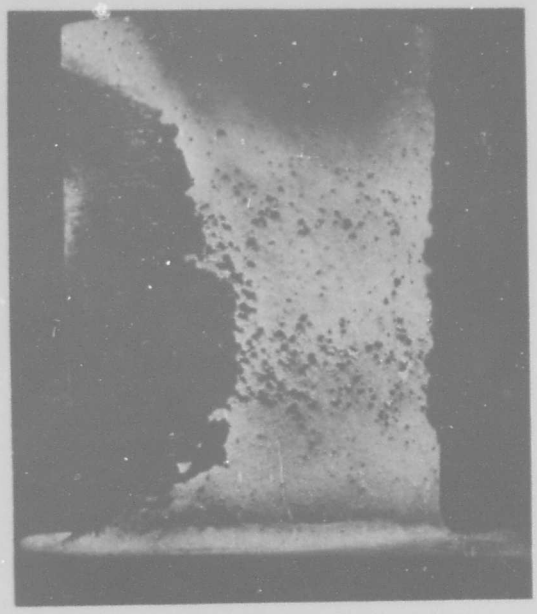
Figure 38. TRW TiCr-Si (Vacuum) Pack Coated C-129Y Columbium Alloy Vane Specimen After 600 Thermal Cycles at 2200°F



ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER MAG: 500X

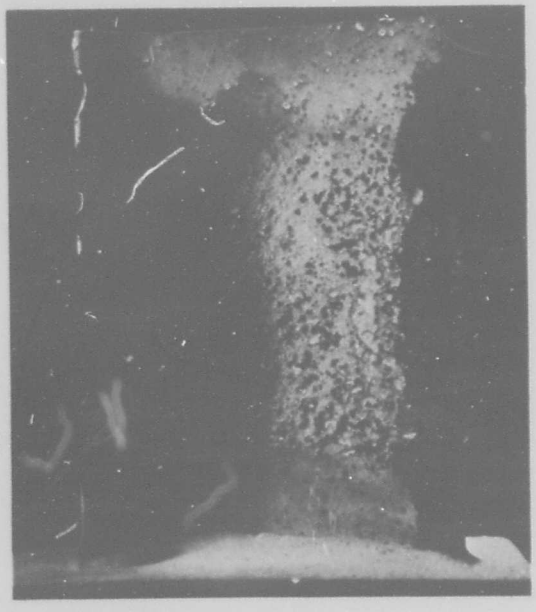
Figure 39. Microstructure of TRW TiCr-Si (Vacuum) Pack Coated C-129Y Columbium Alloy Vane Specimen After 600 Thermal Cycles at 2200°F

LEADING EDGE SURFACE



CONVEX AIRFOIL SURFACE
(NOTE FLOW OF SiO_2-RICH SURFACE PHASE)

TRAILING EDGE SURFACE



CONCAVE AIRFOIL SURFACE

MAG: 1X

Figure 40. TRW TiCr-Si (Vacuum) Pack Coated C-129Y Columbium Alloy Vane Specimen After 600 Thermal Cycles at 2200°F, 400 Cycles at 2400°F, and 400 Cycles at 2500°F

apparently an SiO_2-rich amorphous phase resulting from reaction with oxygen. Some erosion of the SiO_2-rich phase occurred on the specimen leading edge surface as illustrated in Figure 40.

Localized oxidation and spalling were observed on the internal airfoil surfaces and the external surfaces away from the leading and trailing edges. Substrate oxidation had occurred at three locations on the airfoil trailing edge (see Figure 40). No airfoil cracking was apparent, and, in most cases, oxidation at the base of coating craze cracks had not penetrated the (Cb, Ti)Cr_2 Laves phase. Increases in substrate microhardness just below the coating interface, however, suggest that embrittlement had occurred in these areas (Figure 41).

In comparing the coating structure of the latter specimen with the one tested for only 600 cycles at 2200°F, two failure mechanisms were observed to be operative. First, repeated cycling (or longer times in the case of oxidation-erosion) at the higher temperatures led to increased craze crack oxidation extending laterally between the (Cb, Ti)Cr_2 Laves phase layer and the outer silicide layers of the coating (Figure 42). This continued oxidation caused greater separation of the coating layers and a widening of the craze cracks. Second, oxygen apparently penetrated the (Cb, Ti)Cr_2 Laves phase during this period, causing a volume increase in the contaminated substrate region. The Laves phase exhibited sufficient ductility at the elevated temperatures to plastically deform during the volume change. However, the phase eventually either mechanically ruptured or was depleted to the extent that attack of the columbium substrate occurred (see Figure 42).

TRW TiCr-Si (Vacuum) Pack/Cb-132M. After only 400 cycles at 2200°F, the specimens exhibited localized oxidation and spalling on the airfoil surfaces (Figure 43). These spot failures resulted from the presence of surface craze cracks (observed on the as-coated specimens) and led to progressive deterioration of the coating during continued testing. This poor performance was in contrast to quite favorable results obtained during the oxidation-erosion tests. However, as previously discussed in paragraph 2.2, Coatings Application, no distinct (Cb, Ti)Cr_2-type Laves phase was observed for the TiCr-Si coating on the Cb-132M alloy and these results therefore indicate that a continuous protective phase at the coating-substrate interface is necessary for good thermal fatigue performance. In the absence of a protective layer adjacent to the substrate, surface craze cracks extended from the surface directly to the substrate where oxidation occurred (Figure 44). Further testing (beyond the first 400 cycles) led to progressive specimen deterioration due to substrate oxidation and embrittlement.

TRW TiCr-Si (Slip) Pack/Cb-132M. After 200 thermal fatigue cycles at 2200°F, localized oxidation in spalled areas was observed on the

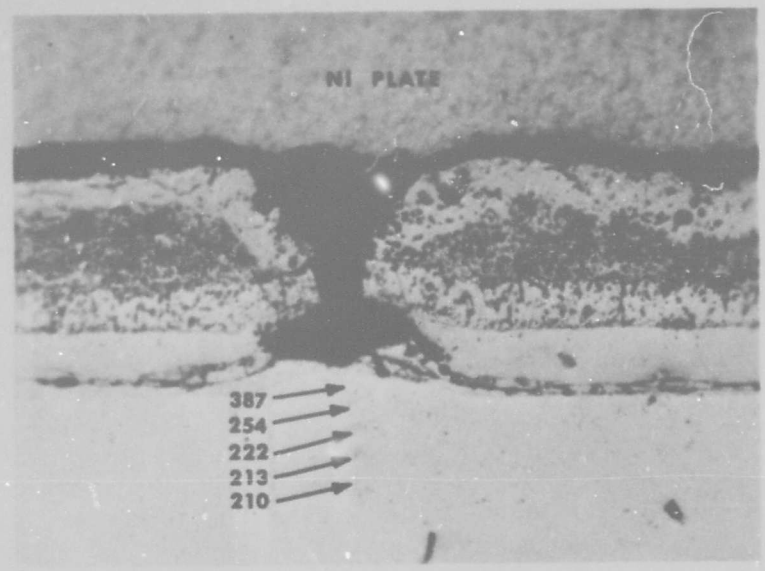
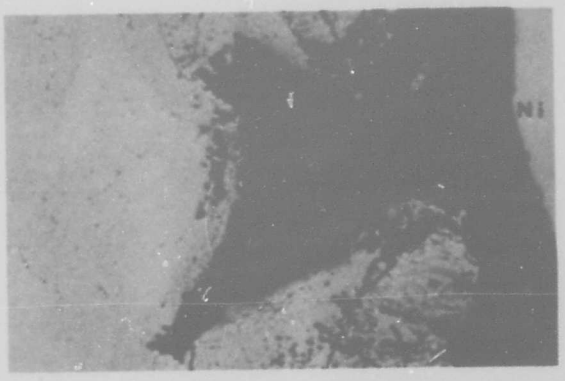
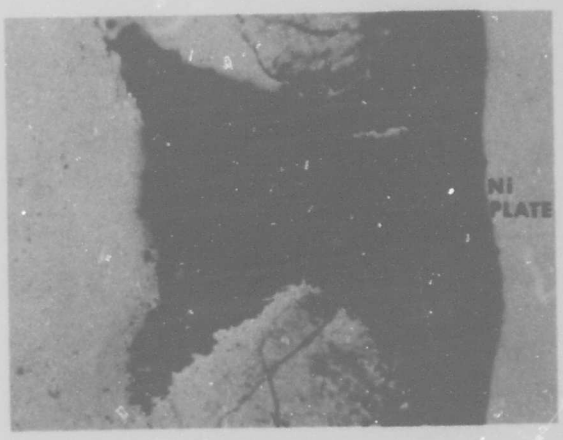


Figure 41. Microstructure of TRW TiCr-Si (Vacuum) Pack Coated C-129Y Alloy Vane Specimen After 600 Thermal Cycles at 2200°F, 400 Cycles at 2400°F, and 400 Cycles at 2500°F, Showing Representative Diamond Pyramid Hardness Values (gm/mm^2; 50-gram load) at Locations in Substrate

600 CYCLES AT 2200°F



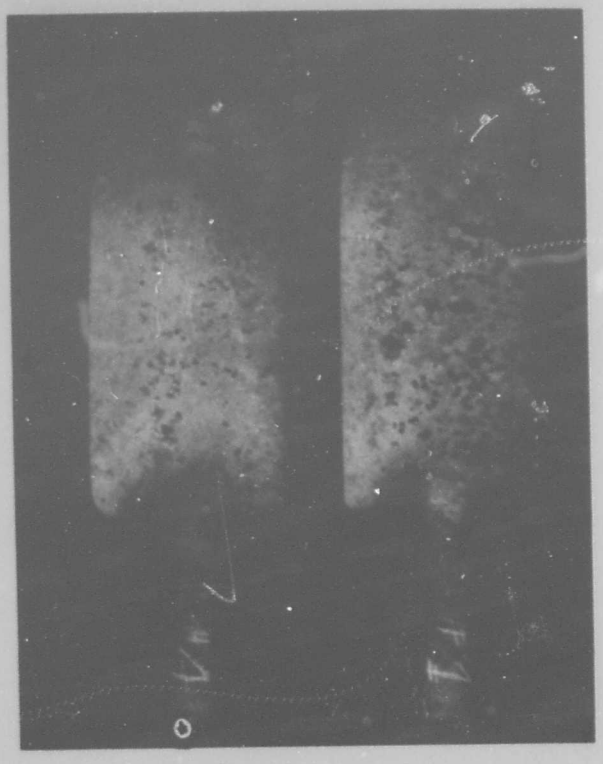
600 CYCLES AT 2200°F
+400 CYCLES AT 2400°F
+400 CYCLES AT 2500°F



600 CYCLES AT 2200°F
+400 CYCLES AT 2400°F
+400 CYCLES AT 2500°F

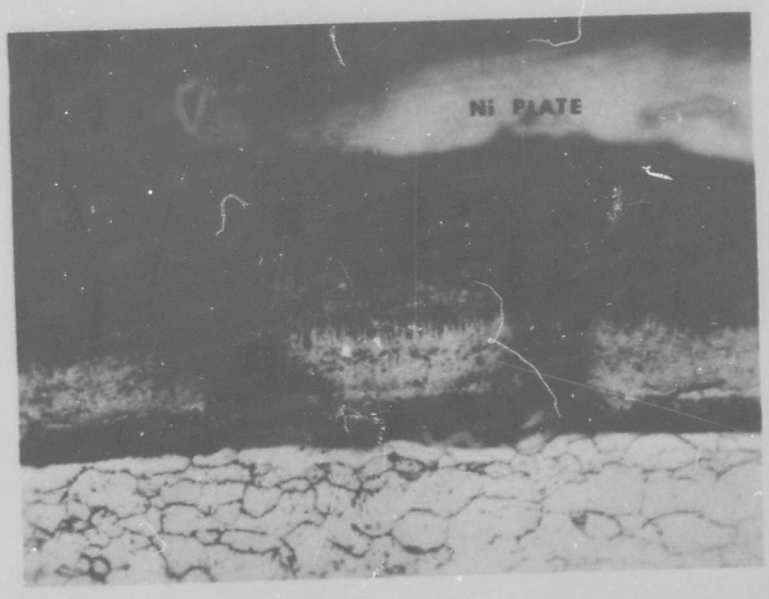
ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER MAG: 500X

Figure 42. Microstructure of the TRW TiCr-Si (Vacuum) Pack Coating on C-129Y Alloy Showing Extension of Craze Crack and Penetration of the $(Cb, Ti)Cr_2$ Laves Phase as a Result of Continued Testing



MAG: 1.25X

Figure 43. Surface Appearance of the TRW TiCr-Si (Vacuum) Pack Coating on Cb-132M Alloy After 400 Thermal Cycles at 2200°F



ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER MAG: 500X

Figure 44. Microstructure of the TRW TiCr-Si (Vacuum) Pack Coating on Cb-132M Alloy After 600 Thermal Cycles at 2200°F, 400 Cycles at 2400°F, and 400 Cycles at 2500°F

specimen airfoil surfaces (Figure 45). An additional 200 cycles at 2200°F resulted in further degradation of these areas as well as the generation of new spalled areas.

Metallographic examination after 400 thermal fatigue cycles revealed oxide penetration through the coating and oxidation of the substrate (Figure 46).

Sylcor Ti-CrTi-Si (Triplex) Pack/D-43. Two specimens failed during 2200°F testing, one specimen after 400 cycles (Figure 47) and the other after 600 cycles (Figure 48). In both cases, failure was by airfoil cracking. The coating on the external airfoil surfaces after failure appeared to be in good condition with no areas of substrate oxidation. Metallographic examination revealed that the inadequate performance was due to the following factors.

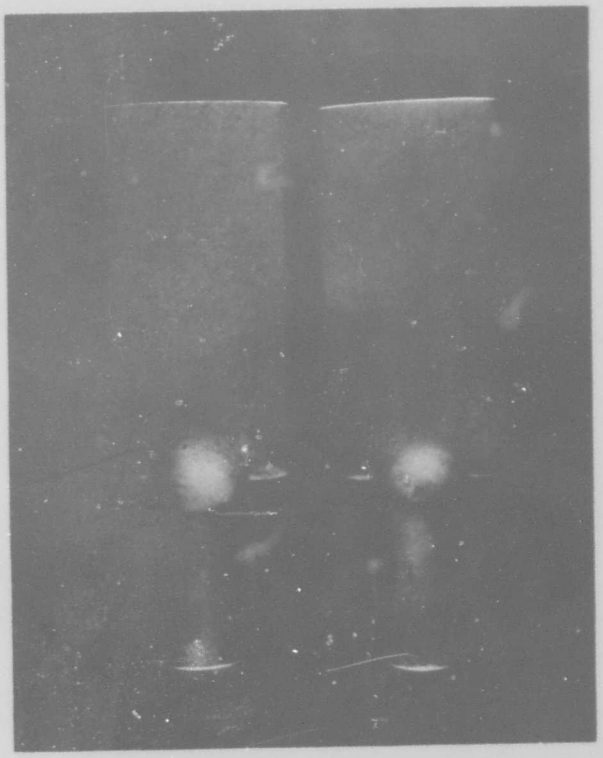
- The absence of a continuous coating protective phase at the coating-substrate interface resulting in craze crack propagation from the coating surface to the substrate (Figure 49)

- Inadequate coating coverage on internal surfaces of the airfoil, particularly at the trailing edge (Figure 50)

Sylcor SiCrTi Slurry/D-43. After 600 thermal fatigue cycles at 2200°F, both test specimens were in good condition. The general surface appearance of the coating was very good, although a reaction had occurred at the leading edge surface (Figure 51). Additional testing at 2400°F for 400 cycles resulted in continued reaction and liquid phase flow back along the airfoil surface (Figure 52).

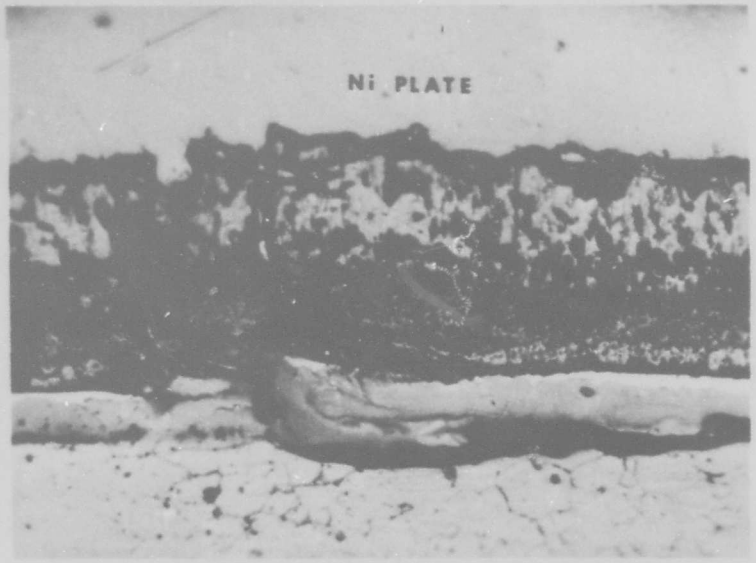
After an additional 500 cycles at 2500°F, the first specimen exhibited severe degradation at the leading edge, an area of substrate oxidation at the trailing edge, and substrate oxidation at the open end of the airfoil at the airfoil trailing edge weld joint (Figure 53). The previously described coating flow was witnessed during 2500°F testing. The flow was extensive and proceeded in a pulsating fashion.

An additional 400 thermal cycles at 2500°F on the second test specimen did not produce the severe degradation exhibited by the first specimen, but a small area of coating failure was observed on the leading edge surface (Figure 54). After testing, that portion of the leading edge from where the majority of the outer coating layers had flowed back along the airfoil was very smooth and appeared to be "fire polished". In addition to coating loss, metallographic examination of the leading edge revealed formation of discrete particles (probably oxide) in the outer layers of reacted coating. Penetration of oxide through the coating was evident (Figure 55), but no distinct oxidation of the substrate was noticeable. Metallographic



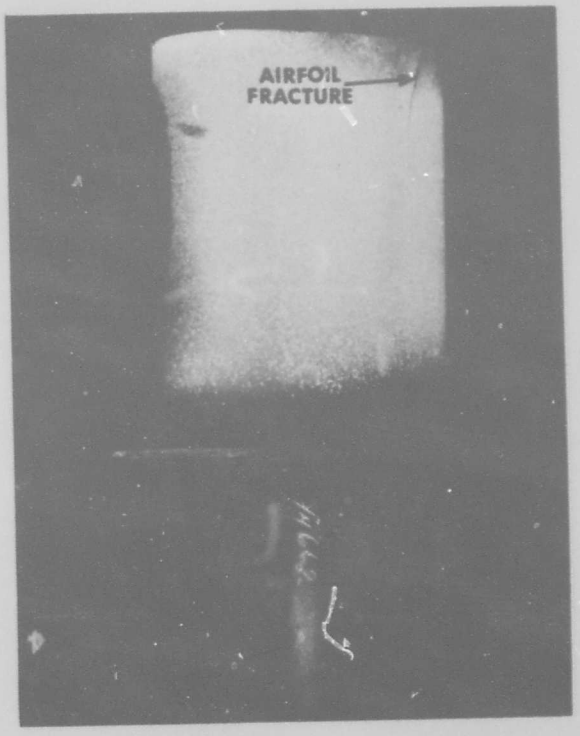
MAG: 1.2X

Figure 45. Surface Appearance of the TRW TiCr-Si (Slip) Pack Coating on Cb-132M Alloy After 200 Thermal Cycles at 2200°F



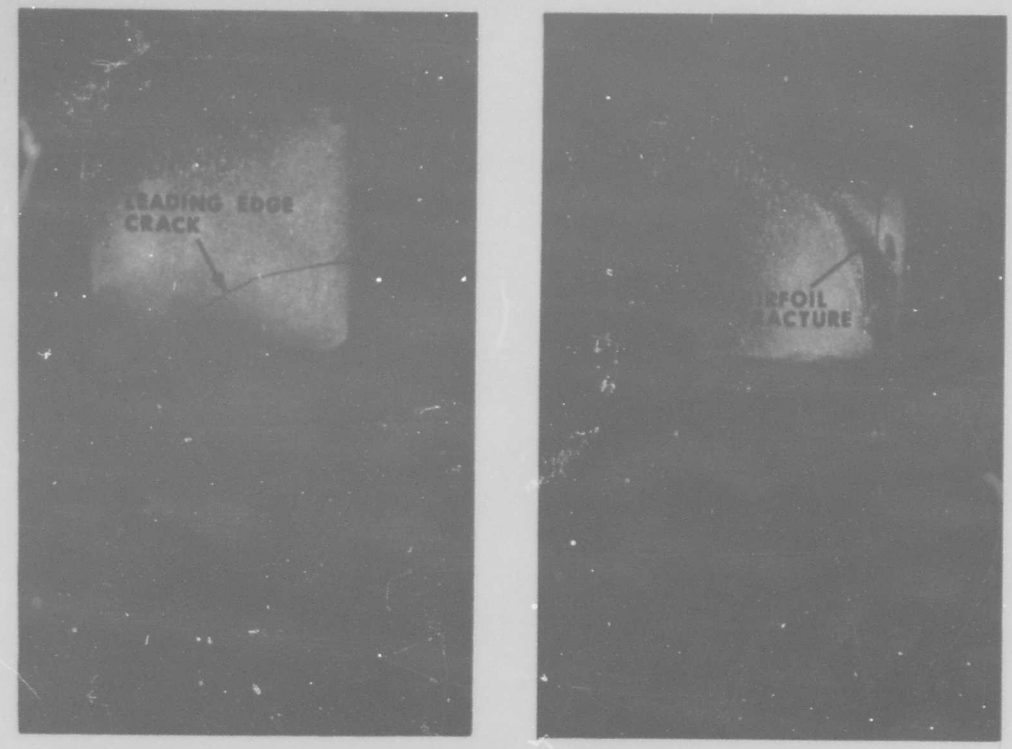
ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER MAG: 500X

Figure 46. Microstructure of the TRW TiCr-Si (Slip) Pack Coating on Cb-132M Alloy After 400 Thermal Cycles at 2200°F



MAG: 0.75X

Figure 47. Sylcor Ti-CrTi-Si (Triplex) Pack Coated D-43 Columbium Alloy Vane Specimen After 400 Thermal Cycles at 2200°F



CONCAVE AIRFOIL SURFACE

CONVEX AIRFOIL SURFACE

MAG: 0.7X

Figure 48. Sylcor Ti-CrTi-Si (Triplex) Pack Coated D-43 Columbium Alloy Vane Specimen After 600 Thermal Cycles at 2200°F

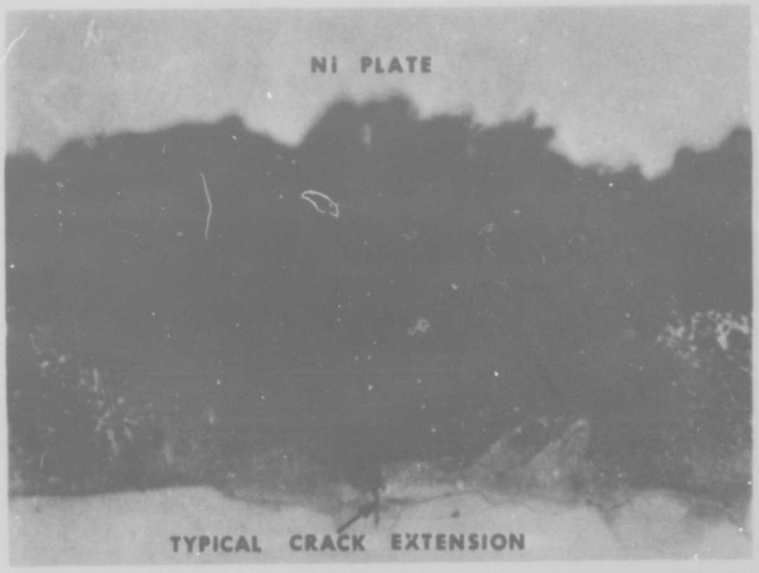


Figure 49. Microstructure at External Leading Edge Surface of Sylcor Ti-CrTi-Si (Triplex) Pack Coated D-43 Columbium Alloy Vane Specimen After 400 Thermal Cycles at 2200°F

ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER MAG: 500X

Figure 50. Microstructure at Internal Trailing Edge Surface of Sylcor Ti-CrTi-Si (Triplex) Pack Coated D-43 Columbium Alloy Vane Specimen After 400 Thermal Cycles at 2200°F (note absence of coating)

CONCAVE AIRFOIL SURFACE

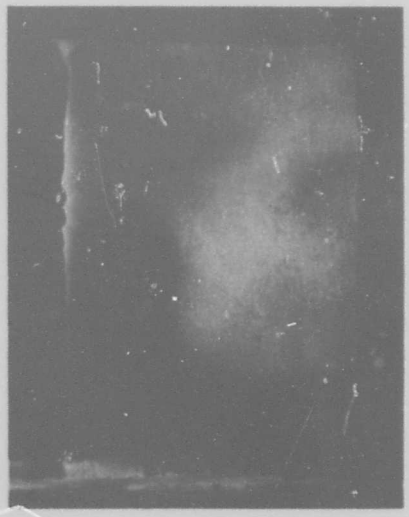
CONVEX AIRFOIL SURFACE

MAG: 0.9X

Figure 51. Surface Appearance of the Sylcor SiCrTi Slurry Coating on D-43 Alloy After 600 Thermal Cycles at 2200°F

CONVEX AIRFOIL SURFACE

LEADING EDGE SURFACE



CONCAVE AIRFOIL SURFACE

MAG: 0.8X

Figure 52. Surface Appearance of the Sylcor SiCrTi Slurry Coating on D-43 Columbium Alloy After 600 Thermal Cycles at 2200°F and 400 Cycles at 2400°F

CONVEX AIRFOIL SURFACE

LEADING EDGE SURFACE

CONCAVE AIRFOIL SURFACE

TRAILING EDGE SURFACE

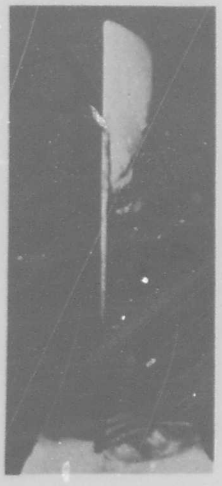
MAG: 0.8X

Figure 53. Surface Appearance of the Sylcor SiCrTi Slurry Coating on D-43 Columbium Alloy After 600 Thermal Cycles at 2200°F, 400 Cycles at 2400°F, and 500 Cycles at 2500°F (Specimen No. 1)

CONVEX AIRFOIL SURFACE

LEADING EDGE SURFACE

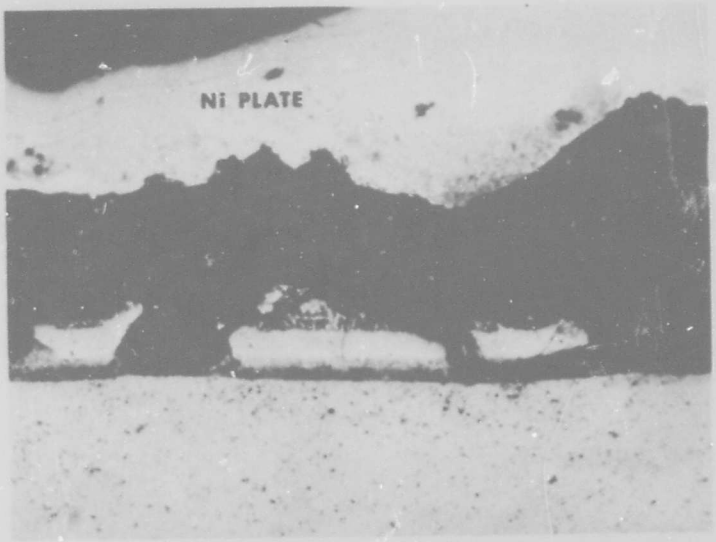
CONCAVE AIRFOIL SURFACE



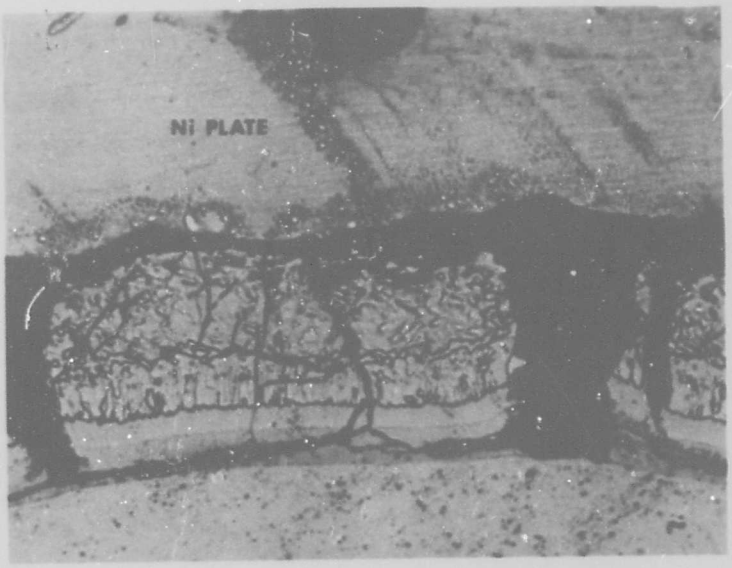
TRAILING EDGE SURFACE

MAG: 0.8X

Figure 54. Surface Appearance of the Sylcor SiCrTi Slurry Coating on D-43 Columbium Alloy After 600 Thermal Cycles at 2200°F, 400 Cycles at 2400°F, and 400 Cycles at 2500°F (Specimen No. 2)



COATING STRUCTURE AT LEADING EDGE SURFACE



COATING STRUCTURE AT TRAILING EDGE SURFACE

ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER MAG: 500X

Figure 55. Microstructure of Sylcor SiCrTi Slurry Coated D-43 Columbium Alloy Vane Specimen After 600 Cycles at 2200°F Plus 400 Cycles at 2400°F Plus 400 Cycles at 2500°F

examination also showed excellent coating coverage on internal airfoil surfaces (Figure 56).

2.3.2.3 Ballistic Impact Results

The impact characteristics for both the original and supplemental systems did not differ appreciably during ballistic impact testing. The physical characteristics of the specimens were characterized by five zones, depending on the condition of the outer silicide layers after impact and before oxidation exposure (Figure 57). These zones were as follows:

- Zone I, impact depression: A region consisting of crushed and compressed coating. The outer silicide layers exhibit complete structure disregistry after impact.

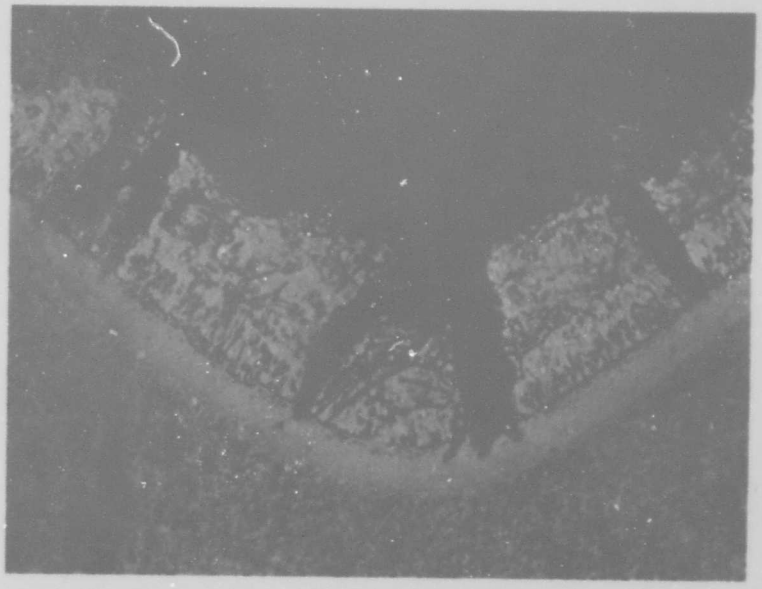
- Zone II, scab area surrounding impact depression: This area is characterized by complete removal of the outer silicide coating layers. An increase in impact velocity results in extension of this zone; whereas scabbing is reduced as impact temperature is increased beyond approximately 2200°F.

- Zone III, unaffected coating: The areas of the specimen lying outside of the impact-affected zones.

- Zone IV: This zone is characterized by coating adherence with perpendicular cracking and separation of the outer silicide layers in the area of specimen directly behind the impact point.

- Zone V, scab area surrounding the protrusion of Zone IV: The scabbing surrounding Zone IV is similar in nature but less severe than that observed in Zone II.

Due to the thickness of the blade airfoil test specimens (0.125 inches compared to 0.050 inches for the vane alloy test specimens), Zones IV and V did not appear on these specimens. The as-impacted surface appearance of representative specimens of each system are illustrated in Figures 58 through 65. A summary of the ballistic impact test results on all five coating-substrate systems is presented in Table IV, with the observed condition after oxidation exposure for various periods at 2200°F. In all cases, failures were first noted in the scabbed areas (either Zone II or Zone V). Oxidation was generally more prevalent on the impact sides of the specimens.



ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER MAG: 500X

Figure 56. Microstructure at Internal Trailing Edge Surface of Sylcor SiCrTi Slurry Coated D-43 Alloy Vane Specimen After 600 Cycles at 2200°F, 400 Cycles at 2400°F, and 500 Cycles at 2500°F (airfoil center location)

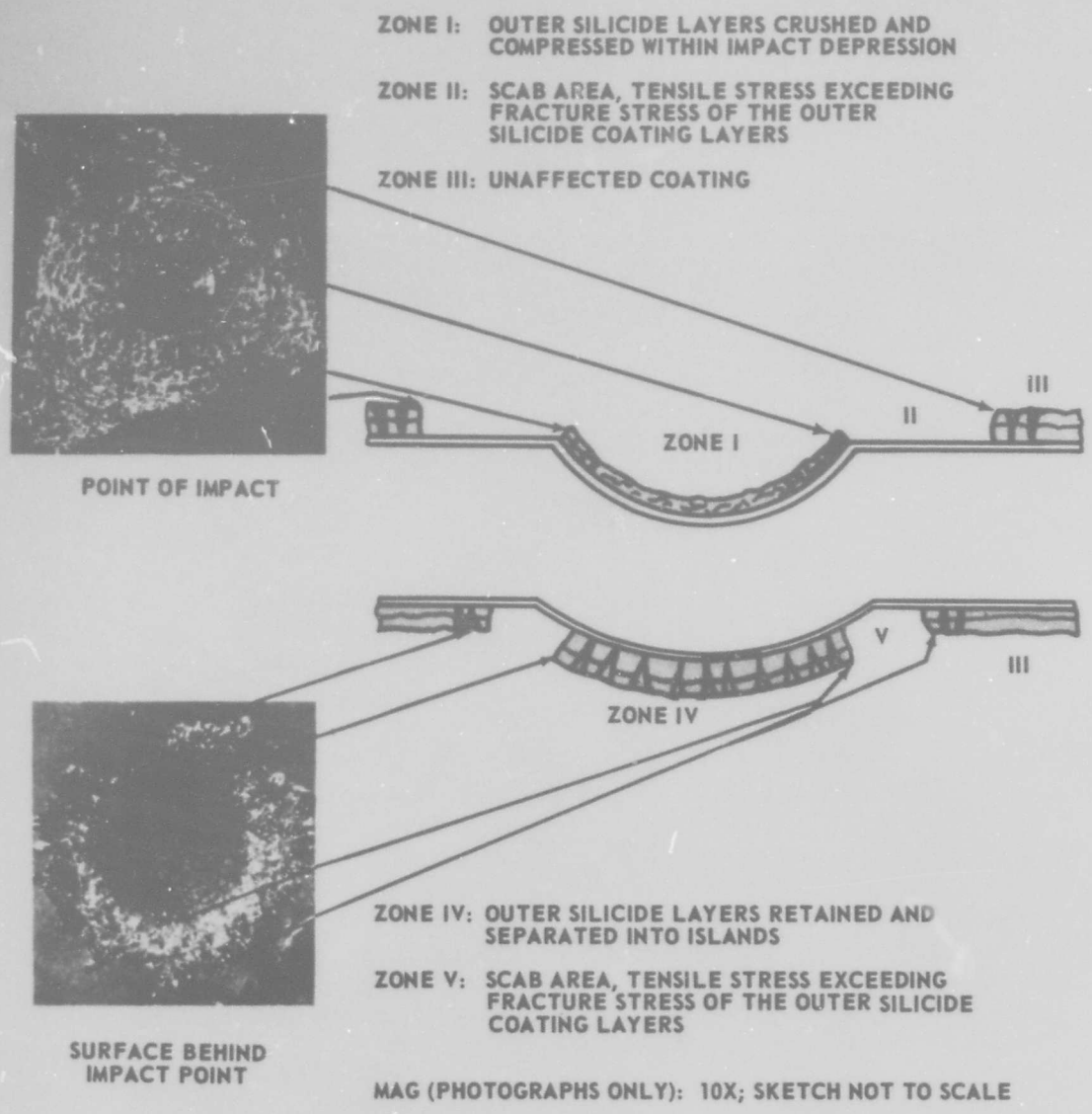


Figure 57. Schematic Cross Section of a Typical Coated Specimen After Ballistic Impact Test

POINT OF IMPACT

SURFACE BEHIND IMPACT POINT

MAG: 10X

Figure 58. Surfaces of TRW TiCr-Si (Vacuum) Pack Coated C-129Y Columbium Alloy After Room Temperature Impact at 500 Feet Per Second (no oxidation exposure)

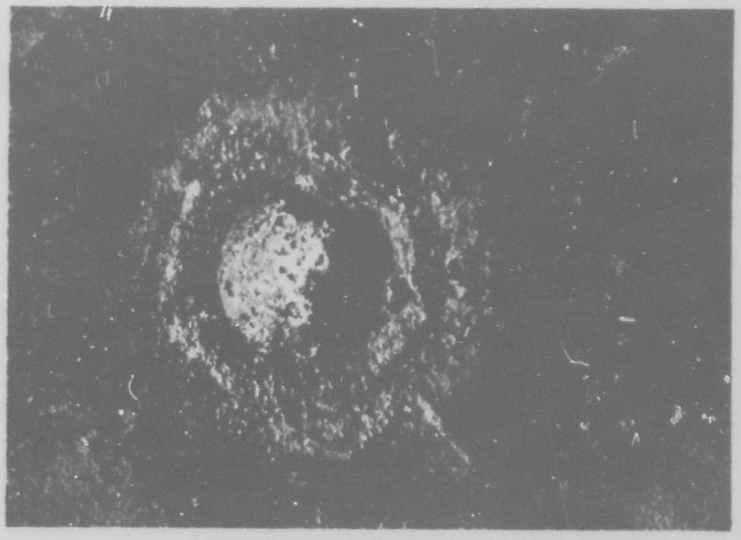
POINT OF IMPACT

SURFACE BEHIND IMPACT POINT

MAG: 10X

Figure 59. Surfaces of TRW TiCr-Si (Vacuum) Pack Coated C-129Y Columbium Alloy After Impact at 500 Feet Per Second at 2200°F (no oxidation exposure)

POINT OF IMPACT

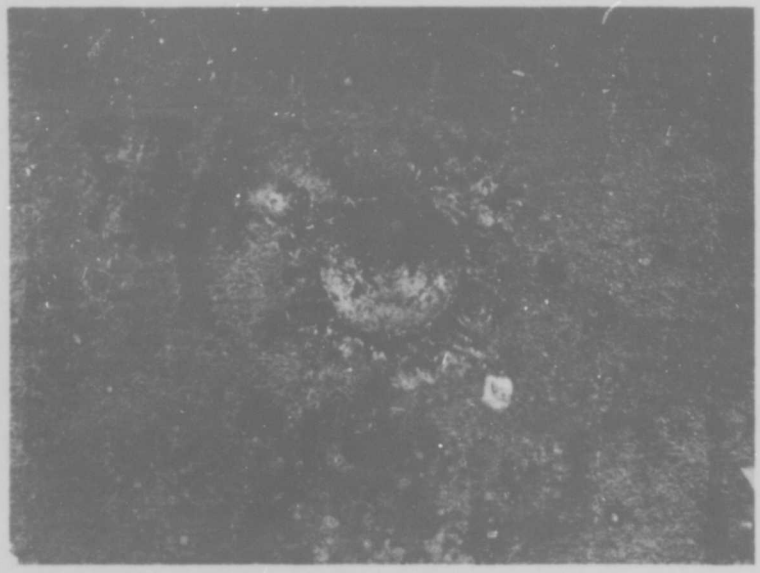


POINT OF IMPACT

MAG: 10X

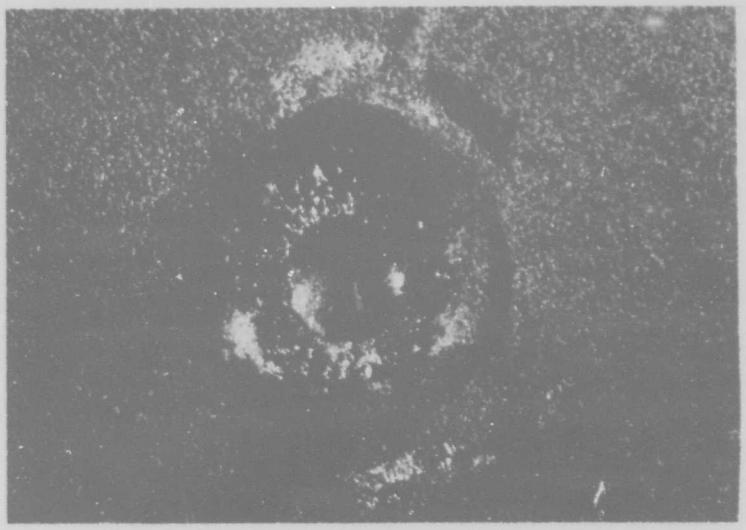
Figure 60. Surface of TRW TiCr-Si (Vacuum) Pack Coated Cb-132M Alloy After 2200°F Impact at 500 (top) and 900 (bottom) Feet Per Second (no oxidation exposure)

POINT OF IMPACT

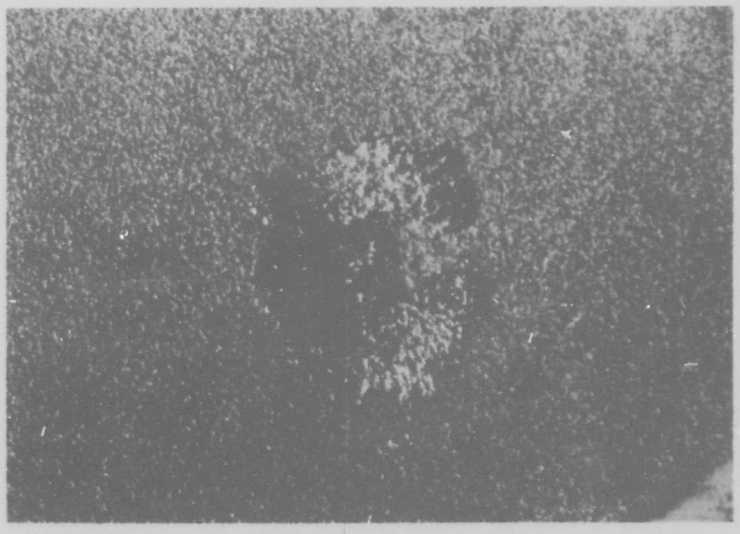


POINT OF IMPACT MAG: 10X

Figure 61. Surface of TRW TiCr-Si (Slip) Pack Coated Cb-132M Alloy After Room Temperature Impact at 500 Feet Per Second (top) and 2200°F Impact at 500 Feet Per Second (bottom) (no oxidation exposure)



POINT OF IMPACT



SURFACE BEHIND IMPACT POINT

MAG: 10X

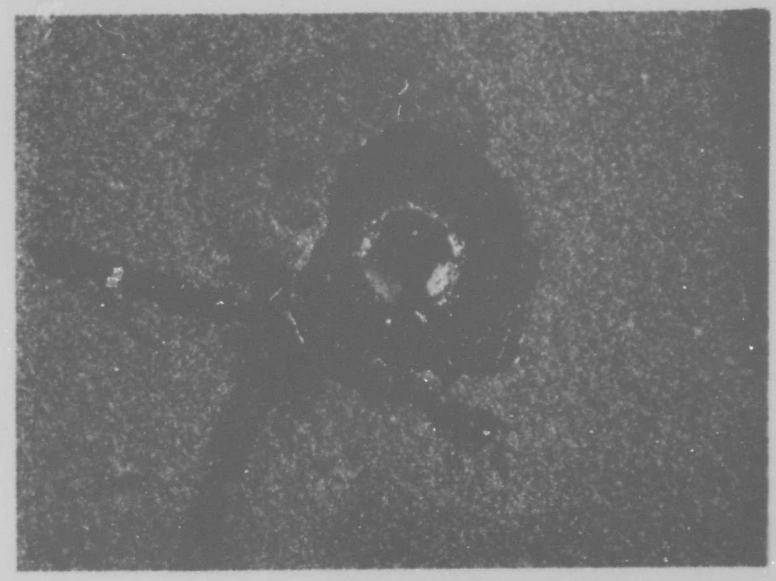
Figure 62. Surfaces of Sylcor Ti-CrTi-Si (Triplex) Pack Coated D-43 Columbium Alloy After Room Temperature Impact Testing at 500 Feet Per Second (no oxidation exposure)

POINT OF IMPACT

SURFACE BEHIND IMPACT POINT

MAG: 10X

Figure 63. Surfaces of Sylcor Ti-CrTi-Si (Triplex) Pack Coated D-43 Columbium Alloy After Impact at 500 Feet Per Second at 2200°F (no oxidation exposure)



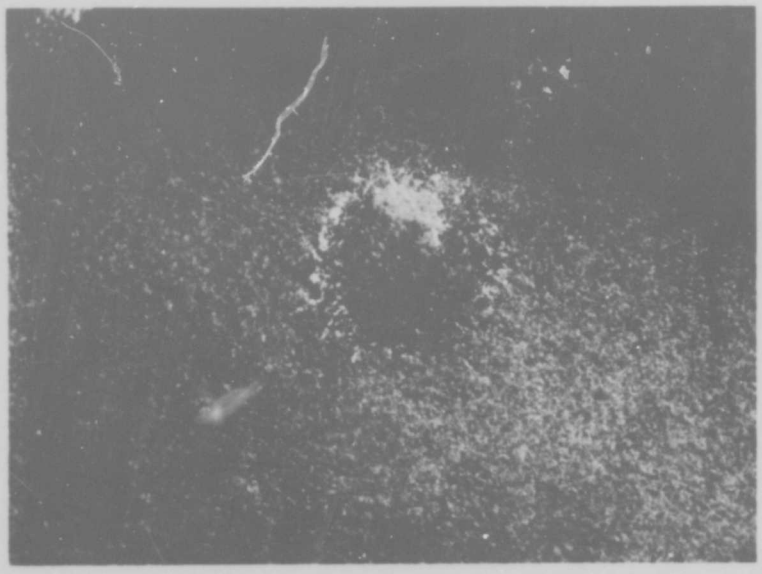
POINT OF IMPACT

SURFACE BEHIND IMPACT POINT

MAG: 10X

Figure 64. Surfaces of Sylcor SiCrTi Slurry Coated D-43 Columbium Alloy After Room Temperature Impact at 200 Feet Per Second (no oxidation exposure)

POINT OF IMPACT



SURFACE BEHIND IMPACT POINT

MAG: 10X

Figure 65. Surfaces of Sylcor SiCrTi Slurry Coated D-43 Columbium Alloy After 2200°F Impact at 500 Feet Per Second (no oxidation exposure)

TABLE IV

RESULTS OF PHASE I BALLISTIC IMPACT TESTING OF THE VANE AND BLADE AIRFOIL COATING-SUBSTRATE SYSTEMS

Application	Test No.	Specimen No.	Coating	Substrate Alloy	Specimen Temp. at Impact (°F)	Impact Velocity (ft/sec)	Oxidation Life After Impact		Observed Condition After Oxidation Exposure	
							Temp. (°F)	Time (hrs.)	Surface at Impact Point	Surface Directly Behind Impact Point
Vane	1	DBI-1-1	Sylcor Ti-CrTi-Si (triplex)	D-43	70	200	2200	8	Oxide surrounding impact depression	Oxidation of surface
	2	DBI-1-2	Sylcor Ti-CrTi-Si (triplex)	D-43	2200	200	2200	8	Oxide surrounding impact depression	No oxide noted
	3	DBI-1-3	Sylcor Ti-CrTi-Si (triplex)	D-43	70	500	2200	5	Oxide surrounding impact depression	Oxidation of surface
	4	DBI-1-4	Sylcor Ti-CrTi-Si (triplex)	D-43	2200	500	2200	8	No oxide noted	No oxide noted
	5	DBI-1-3	Sylcor Ti-CrTi-Si (triplex)	D-43	2200	900	2200	3	Oxide surrounding impact depression	No oxide noted
	6	DBI-1-4	Sylcor Ti-CrTi-Si (triplex)	D-43	2400	200	2200	9	No oxide noted	No oxide noted
	7	YBI-1-1	TRW TiCr-Si (vacuum)	C-129Y	70	200	2200	8	Oxide surrounding impact depression	Oxidation of surface
	8	YBI-1-2	TRW TiCr-Si (vacuum)	C-129Y	2200	200	2200	8	Oxide surrounding impact depression	No oxide noted
	9	YBI-1-3	TRW TiCr-Si (vacuum)	C-129Y	70	500	2200	5	Oxide surrounding impact depression	Oxidation of surface
	10	YBI-1-4	TRW TiCr-Si (vacuum)	C-129Y	2200	500	2200	8	No oxide noted	Oxidation of surface
	11	YBI-1-3	TRW TiCr-Si (vacuum)	C-129Y	2200	900	2200	6	No oxide noted	No oxide noted
	12	YBI-1-4	TRW TiCr-Si (vacuum)	C-129Y	2400	200	2200	9	No oxide noted	No oxide noted
	13	DBI-B-1	Sylcor SiCrTi slurry	D-43	70	200	2200	1	Oxide surrounding impact depression	No oxide noted
	14	DBI-B-1	Sylcor SiCrTi slurry	D-43	70	200	2200	1	Oxide surrounding impact depression	No oxide noted
	15	DBI-B-2	Sylcor SiCrTi slurry	D-43	70	500	2200	1	Oxide surrounding impact depression	No oxide noted
	16	DBI-B-2	Sylcor SiCrTi slurry	D-43	70	500	2200	1	Oxide surrounding impact depression	No oxide noted
	17	DBI-B-3	Sylcor SiCrTi slurry	D-43	2200	200	2200	5	Oxide surrounding impact depression	No oxide noted
	18	DBI-B-3	Sylcor SiCrTi slurry	D-43	2200	200	2200	5	Oxide surrounding impact depression	No oxide noted
	19	DBI-B-4	Sylcor SiCrTi slurry	D-43	2200	500	2200	2	Oxide surrounding impact depression	No oxide noted
	20	DBI-B-4	Sylcor SiCrTi slurry	D-43	2200	500	2200	3	Oxide surrounding impact depression	No oxide noted
Blade Airfoil	21	MBI-1-1	TRW TiCr-Si (vacuum)	Cb-132M	70	200	--	-	Specimen fractured at impact point & at grip	
	22	MBI-1-2	TRW TiCr-Si (vacuum)	Cb-132M	2200	200	--	-	Specimen fractured at grip	
	23	MBI-1-3	TRW TiCr-Si (vacuum)	Cb-132M	70	500	2200	8	No oxide noted	See Note 3
	24	MBI-1-4	TRW TiCr-Si (vacuum)	Cb-132M	2200	500	2200	8	No oxide noted	
	25	MBI-1-3	TRW TiCr-Si (vacuum)	Cb-132M	2200	900	2200	5.5	Oxide surrounding impact depression	
	26	MBI-1-4	TRW TiCr-Si (vacuum)	Cb-132M	2400	200	2200	9	No oxide noted	
	27	MBI-B-1	TRW TiCr-Si (slip) pack	Cb-132M	70	200	2200	5	Oxide beneath an adherent brown skin surrounding impact depression	See Note 3
	28	MBI-B-2	TRW TiCr-Si (slip) pack	Cb-132M	70	500	2200	5		
	29	MBI-B-3	TRW TiCr-Si (slip) pack	Cb-132M	2200	200	2200	5		
	30	MBI-B-4	TRW TiCr-Si (slip) pack	Cb-132M	2200	500	2200	5		

Notes:

1. Weight of steel projectiles: 0.75 gram each
2. Thickness of substrate: D-43 and C-129Y: 0.050 inch; Cb-132M: 0.125 inch
3. No effects were noted on the surface directly behind the impact point on the coated Cb-132M due to the greater thickness of these specimens (0.125 inch).

2.4 EVALUATION TESTING, LOW-TEMPERATURE SYSTEMS

Coating of the root section of a columbium alloy turbine blade requires a different approach than coating of the airfoil. The blade root is not exposed to a high-velocity gas stream and therefore does not require a coating with good erosive properties. However, the coating must exhibit sufficient ductility to deform without cracking in the highly stressed root section and sufficient wear resistance to tolerate the blade-disk bearing loads. Root strains are sufficient to fracture brittle coatings and lead to subsequent failure through oxidation and/or mechanical fatigue. A result of this type is illustrated in Figure 8, where coating cracks led to substrate fatigue failure in a turbine blade root section during engine operation.

Two complementary tests were utilized to evaluate the performance of the candidate blade root coating systems: prestrain/oxidation and wear-galling. These tests were performed to measure coating ductility and resistance to bearing and shear loads.

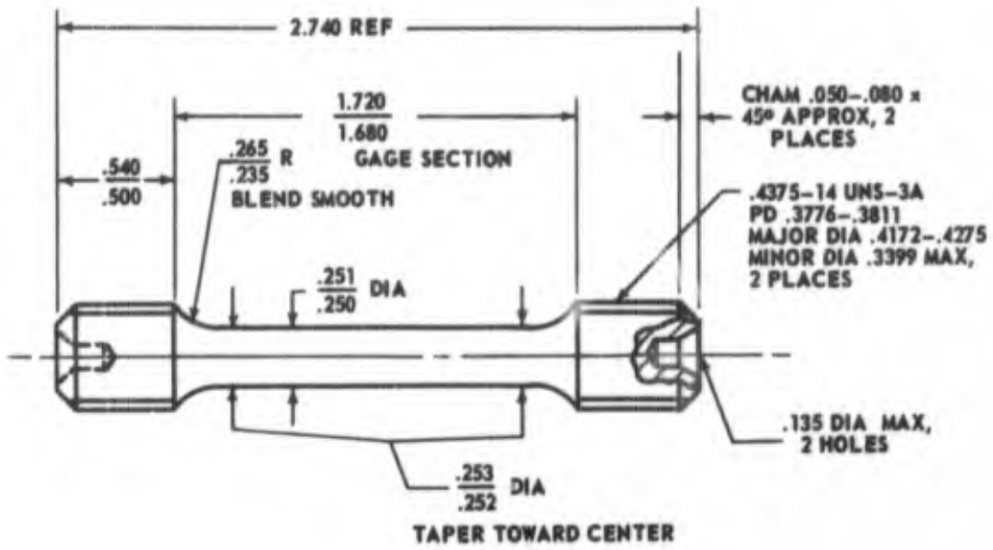
2.4.1 Test Procedures

2.4.1.1 Prestrain/Oxidation Procedure

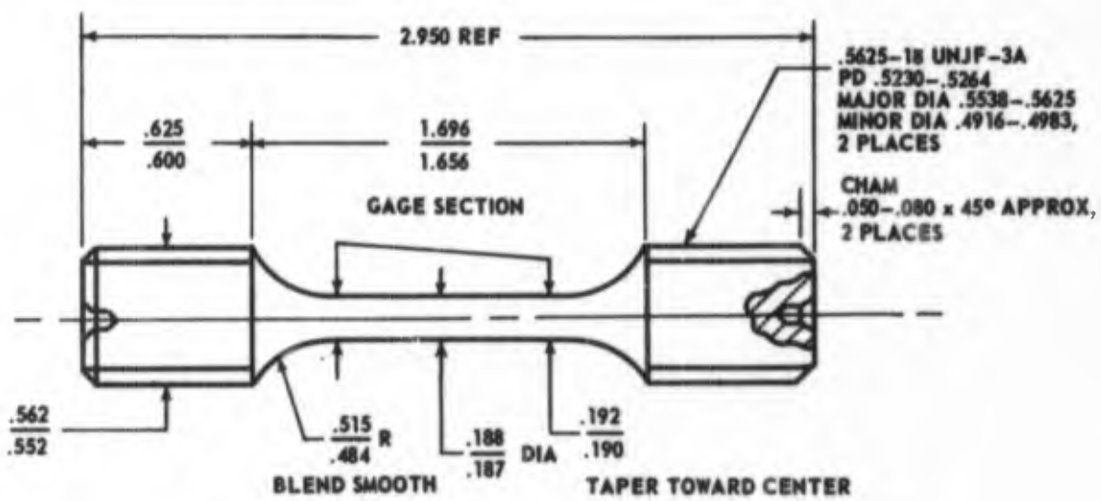
Blade alloy prestrain/oxidation specimens were of a round-bar tensile configuration (Figure 66, drawing A). Specimens were initially strained to a predetermined level of elastic-plus-plastic deformation at room temperature followed by static oxidation exposure at 1300°F.

Uncoated Cb-132M alloy specimens were tested at room temperature to establish a representative tensile stress-strain curve. All specimens failed prematurely in the threaded areas at stress levels below the proportional limit due to the notch sensitivity of the recrystallized Cb-132M alloy. To accommodate evaluation of the Sn-Al and the Ag-Al-Si coated test bars of this geometry, specimens were subsequently strained at 1300°F in an effort to minimize the brittle behavior of the alloy.

The geometry of the specimen was then modified to decrease the cross-sectional area of the gage section relative to that of the threads and also to increase thread root radii (Figure 63, drawing B). An uncoated specimen of this configuration was strained at room temperature and served as a standard for subsequent tests. Prestraining of the coated specimens was conducted at both 70°F and 1300°F. After prestraining, the specimens were subjected to oxidation exposure in still air at 1300°F until failure or for a maximum of 8 hours.



DRAWING A



DRAWING B

Figure 66. Tensile Specimen Configuration Used for Prestrain/Oxidation Tests Before (Drawing A) and After (Drawing B) Modification

2.4.1.2 Wear-Galling Procedure

Two tests which simulate the bearing and shear loads experienced by blade root areas were utilized. One specimen configuration (Figure 67) permitted two tests to be run on each piece (one test at each end of the specimen). Coated specimens were loaded against a mating surface of PWA 1003 (Incoloy 901) nickel-base turbine disk alloy, and subjected at various face pressures to translational motion imposed parallel to the mating surfaces at 1725 cycles per minute with a total deflection of 0.050 inch per one-half cycle. The tests were performed at 1300°F. The deflection and face pressure for succeeding tests were varied between the limits of 0.010 to 0.050 inch and 200 to 5000 psi, respectively. All tests were performed at 1300°F.

To supplement these data, the wear-galling test described below was performed to simulate conditions found in the turbine blade root sections during steady state operation. Rather than a traversing motion between two parallel surfaces, very small shear distances were utilized on a dovetail root configuration (Figure 68). Specimens were held at 1300°F, loaded against a mating surface of PWA 687 (Waspaloy) nickel-base turbine disk alloy, subjected to a tensile load of 2000 psi and a vibratory load of ±200 psi. The vibration frequency was 3600 cycles per minute.

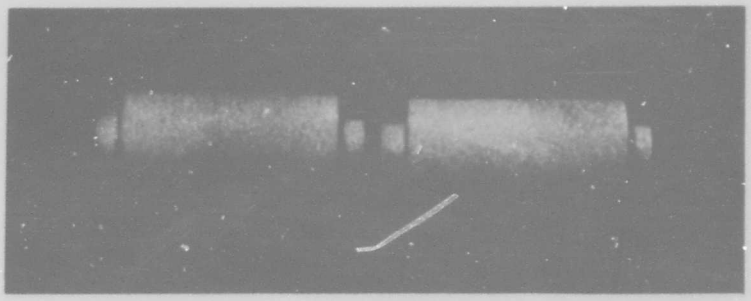
After testing, the specimens were exposed to still air at 1300°F to establish the extent of coating damage. The maximum exposure was 24 hours.

2.4.2 Test Results

2.4.2.1 Prestrain/Oxidation Results

Initially, 1.5 to 3.0 percent elastic-plus-plastic prestrain at room temperature was desired so that direct comparisons of relative coating performance could be made with published data (Ref. 27, 28). Although the modified tensile specimen configuration eliminated the problem of failure within the specimen threads, brittle fracture often occurred within the gage section after approximately 0.2 percent elastic and 0.5 percent plastic strain at 1300°F.

Sylcor Sn-Al (505-F)/Cb-132M. The two original specimens were prestrained a total of 1.45 and 1.52 percent plastic strain, respectively, at 1300°F prior to failure in the specimen threads (Table V, Tests Nos. 1 and 2). For the modified specimens (Table V, Tests Nos. 3 and 4) oxidation exposure at 1300°F after prestraining resulted in almost immediate oxidation at ruptured areas in the coating (see Figure 69). Metallographic



TYPICAL AS-COATED TEST SPECIMENS; MAG: 1.3X

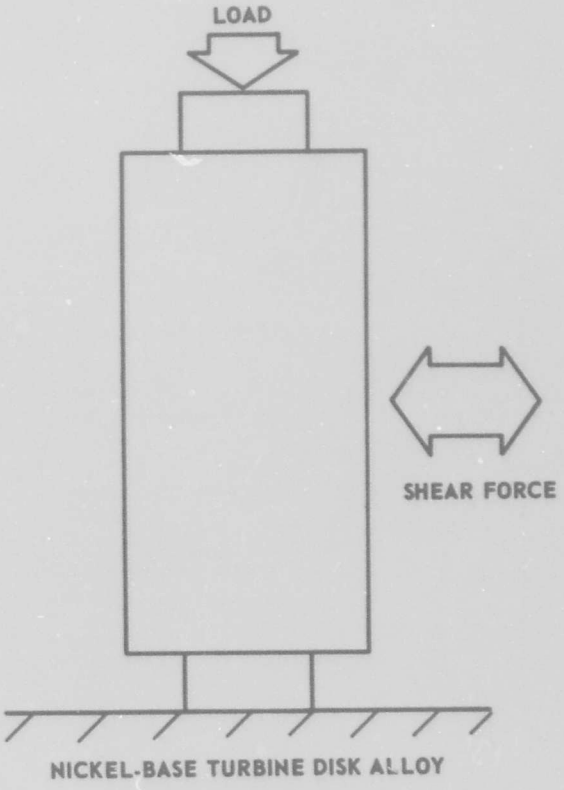
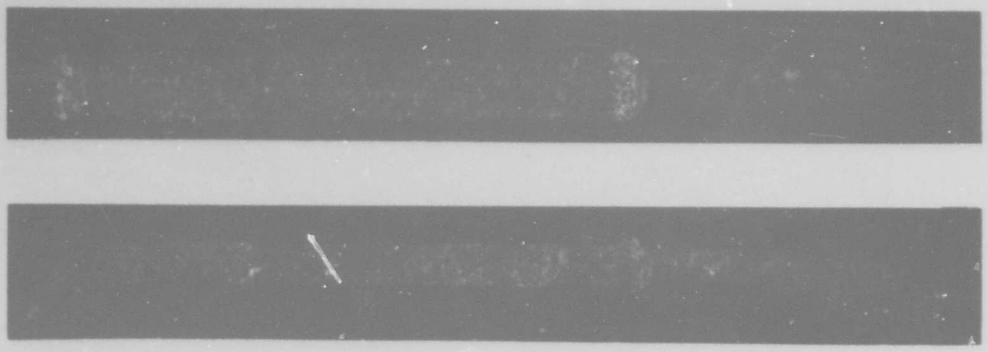


Figure 67. Drum-Type Specimen Configuration Used for Wear-Galling Test



TYPICAL AS-COATED TEST SPECIMEN; MAG: APPROX. 1.2X

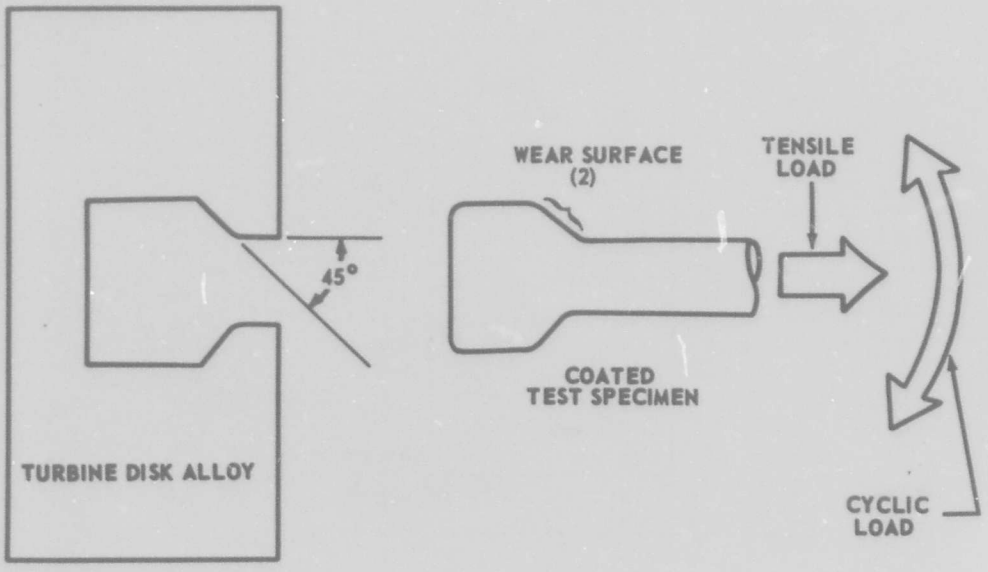


Figure 68. Dovetail Specimen Configuration Used for Wear-Galling Test

TABLE V

RESULTS OF PRESTRAIN/OXIDATION TESTING OF THE PHASE I COATED Cb-132M ALLOY SPECIMENS FOR BLADE ROOT APPLICATION

Test No.	Specimen	Coating	Prestrain Temp. (°F)	Proportional Limit (psi)	Stress at Termination of Load (psi)	Ultimate Strength (psi)	Total Plastic Strain (percent)	Remarks
	Uncoated Standard	---	70	118,500	---	128,000	2.20	Brittle fracture in gage area
1	MTSO-1-1 original	Sn-Al (505-F)	1300	47,550	---	54,300	1.45	Failed in specimen threads
2	MTSO-1-2 original	Sn-Al (505-F)	1300	46,900	---	69,200	1.52	Failed in specimen threads
3	MTSO-1-1 modified	Sn-Al (505-F)	70	90,400	---	99,700	0.15	Brittle fracture in gage area
4	MTSO-1-2 modified	Sn-Al (505-F)	1300	51,300	66,900	---	0.72	Coating ruptured in gage area after prestrain and oxidation at 1300°F
5	MTSO-1-3 modified	Sn-Al (505-F)	1300	55,800	66,400	---	0.54	No visual coating failure after prestraining and subsequent 8-hour oxidation exposure at 1300°F
6	MTSO-1-4 modified	Sn-Al (505-F)	1300	53,900	66,200	---	0.50	Same as MTSO-1-3 modified
7	MAASO-1-1 original	Ag-Al-Si (508-C)	1300	47,300	---	54,900	1.14	Brittle fracture in gage area
8	MAASO-1-2 original	Ag-Al-Si (508-C)	1300	46,900	---	65,300	1.01	Brittle fracture in gage area
9	MAASO-1-1 modified	Ag-Al-Si (508-C)	70	98,200	---	110,500	0.55	Brittle fracture in gage area
10	MAASO-1-2 modified	Ag-Al-Si (508-C)	1300	46,300	54,900	---	0.43	No visual coating failure after prestraining and subsequent 8-hour oxidation exposure at 1300°F
11	MZSO-1-1 modified	Zn	70	---	---	30,000	---	Brittle fracture in elastic range
12	MZSO-1-2 modified	Zn	1300	---	---	43,100	---	Brittle fracture in elastic range

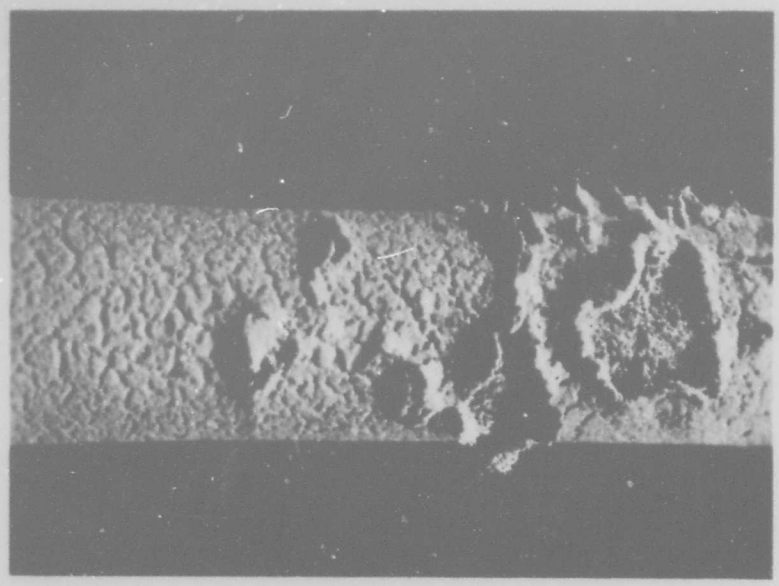


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Figure 69. Sylcor Sn-Al Coating on Cb-132M Test Specimen Gage Section After 0.72-Percent Plastic Prestrain at 1300°F and Oxidation Exposure at 1300°F

examination revealed that rupture, and in some cases separation of the $CbAl_3$ and the lower aluminide layer, had occurred near the coating-substrate interface (Figure 70). Discussions with the coating supplier disclosed that this behavior was not typical. Two additional specimens were therefore coated at Sylcor and subsequently prestrained at 1300°F (Table V, Tests Nos. 5 and 6). No coating failures were observed after 8 hours oxidation exposure at 1300°F, and metallographic examination after testing revealed that although some coating cracking had occurred, the integrity of the coating had been maintained (Figure 71).

Sylcor Ag-Al-Si (508-C)/Cb-132M. The Ag-Al-Si/Cb-132M modified specimens performed well after 1300°F prestraining (Table V, Tests Nos. 7-10). After 1300°F oxidation exposure of the specimen in Test No. 10 (Table V), some coating cracking at the coating-substrate interface was observed (Figure 72), but no oxidation had occurred during the 8-hour exposure after prestraining.

Zinc-Base Coating/Cb-132M. The zinc coating and/or the zinc coating process resulted in severe embrittlement of the substrate (see Table V, Tests Nos. 11 and 12). Both specimens failed below the proportional limit, thereby yielding no plastic strain data. As discussed previously in paragraph 2.2.8, Coatings Application, Zinc-Base Coating, the poor reproducibility of the coating application process led to elimination of the zinc coating from the program.

2.4.2.2 Wear-Galling Results

Sylcor Sn-Al (505-F)/Cb-132M and Ag-Al-Si (508-C)/Cb-132M. For the drum-type specimen configuration, tests showed that both the Sn-Al and the Ag-Al-Si coatings had almost no resistance to wear at face pressures from 200 to 5000 psi (Table VI). In each test, complete coating destruction resulted almost immediately, although the total test times ranged from 15 to 60 minutes (approximately 26,000 to 104,000 cycles). The poor performance was attributed to two factors:

- At 1300°F, some phases of each coating exist as high-viscosity liquids, and the presence of molybdenum compounds in outer coating layers does not sufficiently improve the wear characteristics.
- The traversing distance of 0.050 inch in combination with the pure shear stresses was too severe for the extremely "ductile" coating phases (the tin-rich phase and the silver- or aluminum-rich phases).

The additional test data generated, using the dovetail-configuration specimens, are summarized in Table VII. Results of these tests were

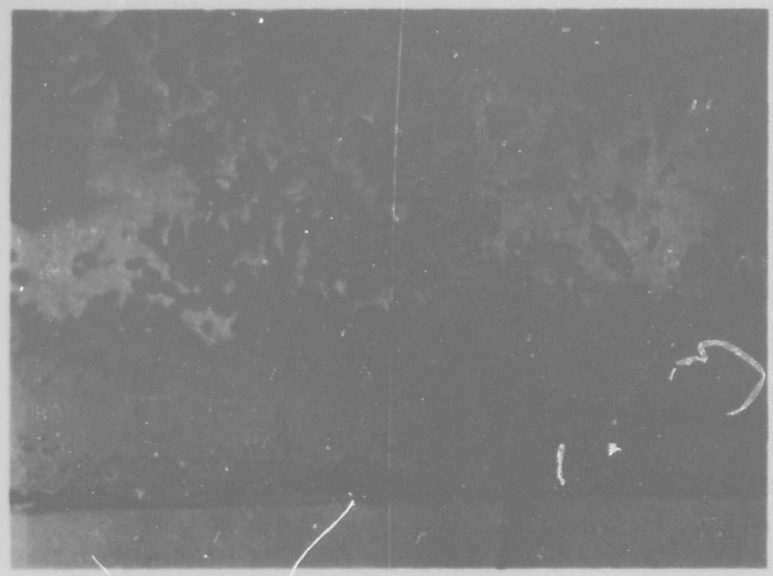

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Figure 70. Microstructure of Sylcor Sn-Al Coating on Cb-132M Alloy After 1300°F Prestrain Showing Cracks At and Near Coating-Substrate Interface

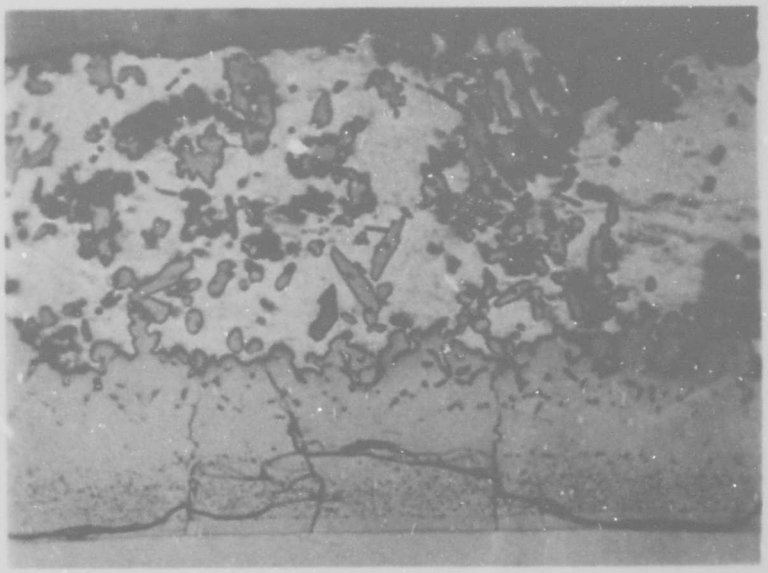

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Figure 71. Microstructure of Sylcor Sn-Al Coating on Cb-132M Alloy After 1300°F Prestrain

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Figure 72. Microstructure of Sylcor Ag-Al-Si Coating on Cb-132M Alloy After 1300°F Prestrain Showing Cracks Near Coating-Substrate Interface

TABLE VI

WEAR-GALLING TEST RESULTS (1300°F) FOR THE
PHASE I COATED Cb-132M ALLOY DRUM-TYPE SPECIMENS

Rubbing surface: PWA 1003 (Incoloy 901) nickel-base alloy
Specimen traversing distance: 0.050 inch per half-cycle
Specimen vibration frequency: 1725 cycles per minute

Test No.	Coating	Specimen Number	Specimen End	Face Pressure (psi)	Wear Check Interval (minutes)	Coating Condition at End of First Check Interval
1	Sn-Al	MTWG-1-1	1	5000	60	Completely removed
2	Sn-Al	MTWG-1-1	2	5000	15	Completely removed
3	Sn-Al	MTWG-1-2	1	1000	15	Completely removed
4	Ag-Al-Si	MAAWG-1-1	1	1000	15	Completely removed
5	Ag-Al-Si	MAAWG-1-1	2	1000	50	Completely removed
6	Ag-Al-Si	MAAWG-1-2	1	200	50	Completely removed

TABLE VII

WEAR-GALLING TEST RESULTS (1300°F) FOR THE PHASE I COATED Cb-132M ALLOY DOVETAIL SPECIMENS

Rubbing surface: PWA 687 (Waspaloy) nickel-base turbine disk alloy
Tensile load: 2000 psi
Vibratory load: ±200 psi
Specimen vibration frequency: 3600 cycles per minute

Coating	Number of Tests	Total Test Cycles	Specimen Condition After Test	Specimen Condition After 1300°F Oxidation Exposure
Sn-Al (505-F)	2	4.5×10^5	Specimens brazed to rubbing surface in the wear-galling areas; coating smeared and partially removed at load-bearing surfaces; no oxidation at wear-galling surface	Local oxidation on load-bearing surface areas after 4 hours; after 8 hours, general oxidation along most of the wear-galling surfaces
Ag-Al-Si (508-C)	2	4.5×10^5	Minor braze bonding in wear-galling areas; significant flow of coating off load-bearing surfaces; dark discolorations but no oxidation on wear-galling surfaces	Bearing surface areas unaffected after 4 hours; after 16 hours, yellowish discolorations but no failures noted; no further degradation of the wear-galling surfaces after 24 hours

somewhat better than those obtained using the drum-type specimens. After 4.5×10^5 cycles at 1300°F, both Sn-Al coated test specimens were brazed to the nickel-base turbine disk alloy in the contact areas. The coating was smeared and partially removed, but no areas of substrate oxidation were visible after testing (Figure 73). Subsequent exposure at 1300°F in still air revealed local oxidation on load-bearing surfaces after 4 hours. After 8 hours, general oxidation occurred along most of the wear-galling surfaces. The Ag-Al-Si coating suffered only minor braze bonding after 4.5×10^5 cycles at 1300°F. However, significant flow of the coating out of the load-bearing areas was noted (Figure 74). No oxidation occurred after 24 hours of subsequent exposure at 1300°F.

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Figure 73. Typical Appearance of Sylcor Sn-Al (505-F) Coating on Cb-132M Columbium Alloy After Wear-Galling Testing for 4.5×10^5 Cycles at 1300°F With a Tensile Load of 2000 psi

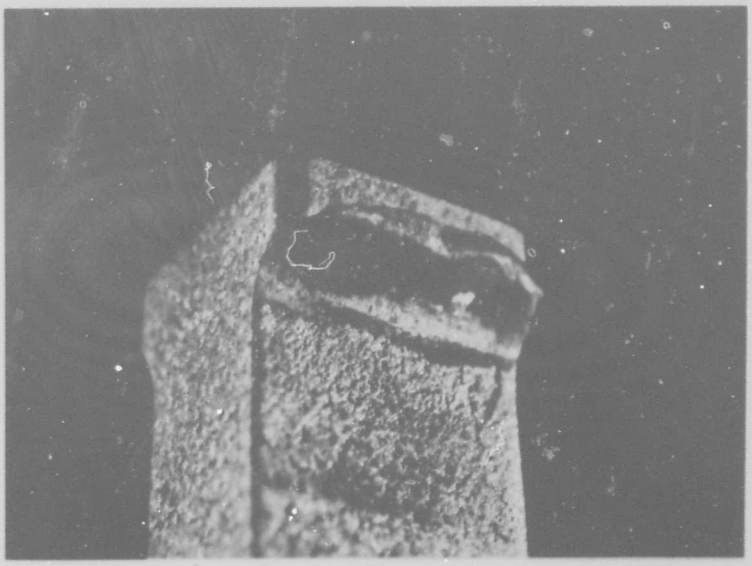

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Figure 74. Typical Appearance of Sylcor Ag-Al-Si (508-C) Coating on Cb-132M Columbium Alloy After Wear-Galling Testing for 4.5×10^5 Cycles at 1300°F With a Tensile Load of 2000 psi (note flow of coating in test area)

3. ITEM 3

COATING IMPROVEMENT

The work required in Item 3 of the contract is Phase II of the coated columbium alloy evaluation program. In this phase the contractor must provide appropriate compositional and/or coating application processing modifications for any of the six original systems demonstrating substandard performance in Phase I (Item 2) tests. Appropriate Phase I tests for each of the modified systems must be conducted to verify optimization and justify promotion to Phase III (Items 4 and 5). A technical flow chart showing the progression of tests through Phase II of the program is presented in Figure 75.

3.1 MATERIALS

The substrate materials used in Phase II of the program were the same as those used in Phase I. The substrate properties are described in Item 2, paragraph 2.1, Materials. As mentioned previously, the two Phase I supplemental systems, TRW TiCr-Si (slip) pack/Cb-132M and Sylcor SiCrTi slurry/D-43, were not candidates for Phase II. The following candidate systems were evaluated in the Phase II portion of the program.

System Application	Application Area	Coating/Substrate	Coating Supplier
High temperature	Vane	TiCr-Si (vacuum) pack/C-129Y	TRW
		Ti-CrTi-Si (triplex) pack/D-43	Sylcor
High temperature	Blade airfoil	TiCr-Si (vacuum) pack/Cb-132M	TRW
Low temperature	Blade root	Sn-Al (505-C)/ Cb-132M	Sylcor
		Ag-Al-Si (508-F)/Cb-132M	Sylcor

3.2 COATINGS APPLICATION

3.2.1 TRW TiCr-Si/C-129Y

Although The TRW TiCr-Si (vacuum) pack/C-129Y system performed reasonably well in the Phase I evaluation tests, discussions with TRW representatives indicated that the performance of the coating was probably not optimized, based on examination of the Phase I coating microstructure (Figure 9). It was agreed that more titanium would therefore be incorporated

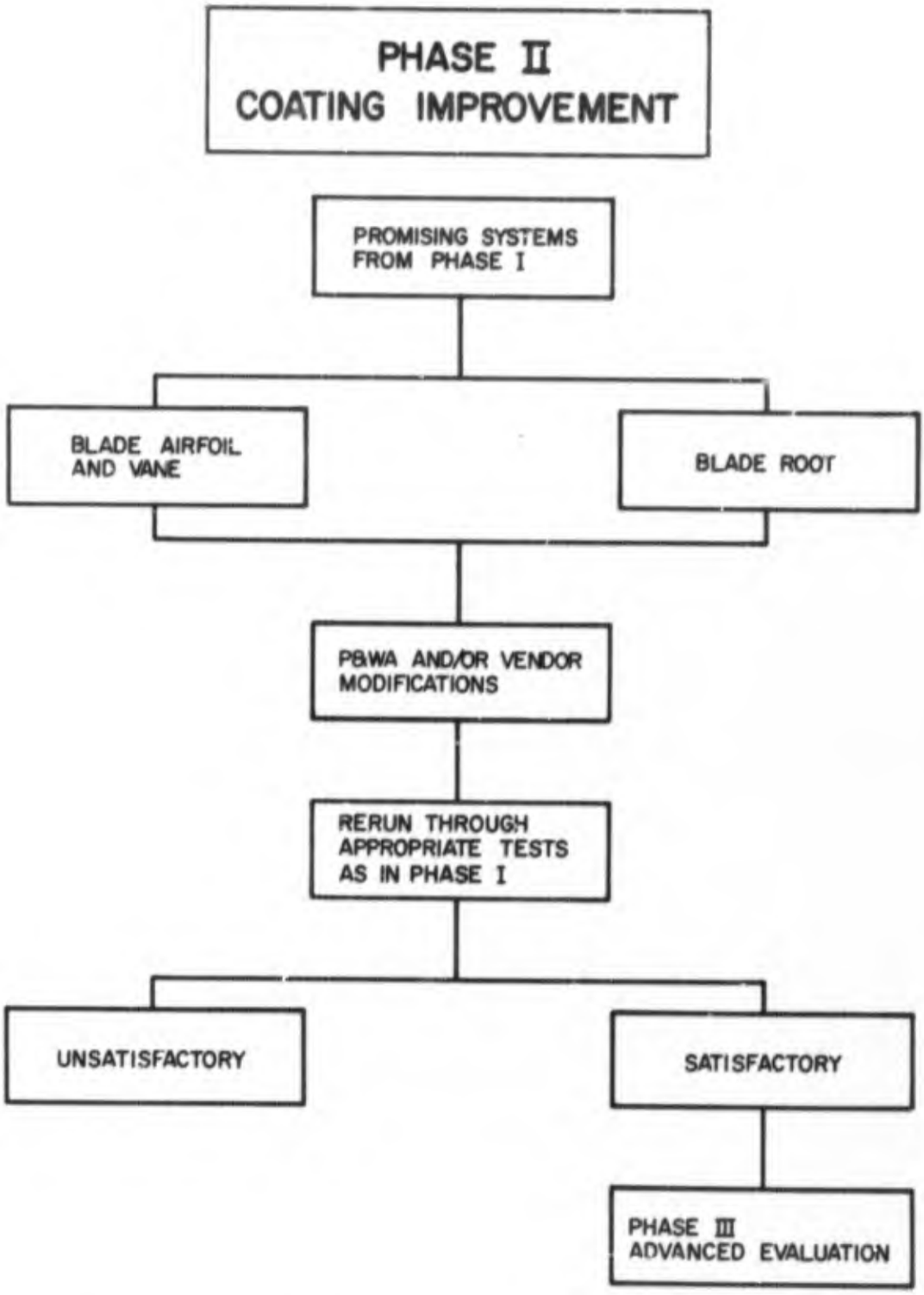


Figure 75. Flow Chart for Phase II of Program (Item 3)

into the coating composition to increase the thickness of the (Cb, Ti)Cr_2 Laves phase on the Phase II test specimens.

Application of the modified TiCr-Si coating on the C-129Y alloy was accomplished at TRW by using a 2-cycle vacuum pack process and the parameters outlined below.

- Cycle 1: In a manner similar to the application of the Phase I coating, the first low-pressure cycle consisted of chromium-titanium deposition at 2200°F for 8 hours from a 60Cr-40Ti weight percent pack utilizing a potassium fluoride activator. Unlike the Phase I coating, however, flow of the gaseous constituents was restricted at the system exhaust, thereby increasing the pressure of the pack. This procedure reportedly produces more thermodynamically favorable conditions for the deposition of titanium.

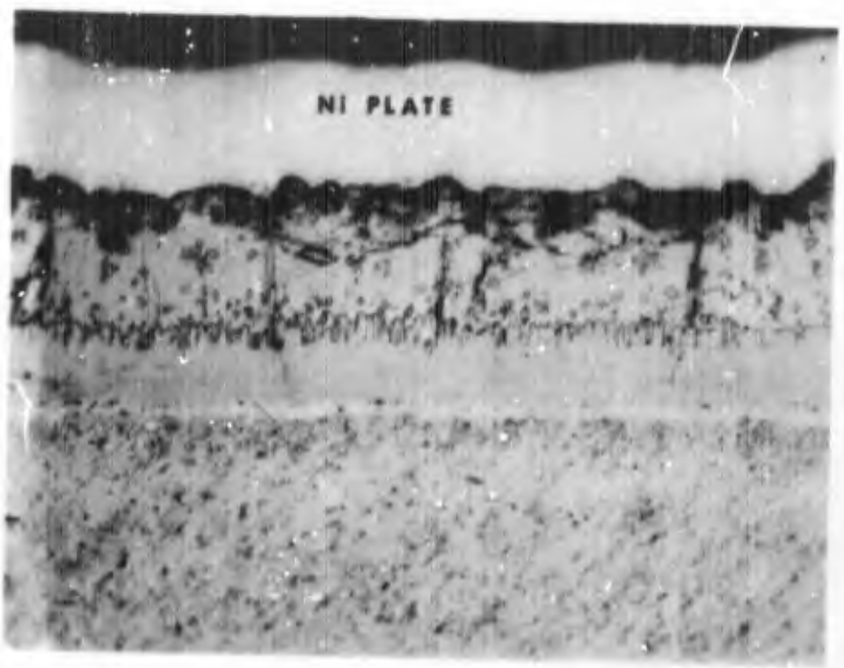
- Cycle 2: In a manner identical to the Phase I coating application, the second low-pressure cycle consisted of a siliconizing cycle at 2050°F for a period of 4 hours, also utilizing a potassium fluoride activator.

The microstructure of the resulting coating (Figure 76) demonstrated that the increased titanium content increased the thickness of the (Cb, Ti) Cr_2 Laves phase from approximately 0.25 mil to approximately 0.44 mil. The overall coating thickness of 2.4 mils was approximately the same as that of the Phase I coating (2.3 mils). The coating contained four distinct layers, two outer silicide layers, the (Cb, Ti)Cr_2 Laves phase layer, and a titanium-rich solid solution zone.

Coating surface appearance was excellent, with no visible surface craze cracks. Coverage appeared excellent at specimen corners and edges.

3.2.2 TRW TiCr-Si (Vacuum) Pack/Cb-132M

As discussed previously in paragraph 2.2, Coatings Application (Phase I), the structure of the TRW TiCr-Si coating on Cb-132M alloy showed no distinct microstructural layers and therefore no apparent continuous protective layer adjacent to the substrate. Since the increased titanium content in the TiCr-Si system was beneficial in increasing the thickness and continuity of the (Cb, Ti)Cr_2-type Laves phase on the C-129Y alloy, it was concluded that additional titanium in the TiCr-Si coating composition on the Cb-132M alloy would be similarly beneficial.



ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER MAG: 500X

Figure 76. Typical Microstructure of the As-Applied Phase II TRW TiCr-Si (Vacuum) Pack Coating on C-129Y Columbium Alloy

Two approaches were considered as summarized below.

- Alteration of the coating application parameters to provide conditions more thermodynamically favorable for the deposition of higher titanium concentrations (the method successfully employed for modifying the coating on the C-129Y alloy)

- Adjustment of the pack composition used during the first vacuum cycle from a 60:40 chromium-titanium ratio to a 50:50 ratio

The first approach involved the same application parameters as discussed for the TRW TiCr-Si/C-129Y system modification. Examination of the resulting as-coated microstructure (Figure 77) revealed no appreciable difference between the Phase I and Phase II coatings. Test results (summarized in later sections) subsequently supported this microstructural similarity between the two coatings.

A second modification was therefore performed following the second approach outlined above; i.e., coating application parameters were the same as discussed previously for application of the Phase I coating, except that a 50:50 chromium-titanium pack ratio was utilized in the first vacuum cycle rather than the 60:40 ratio previously used.

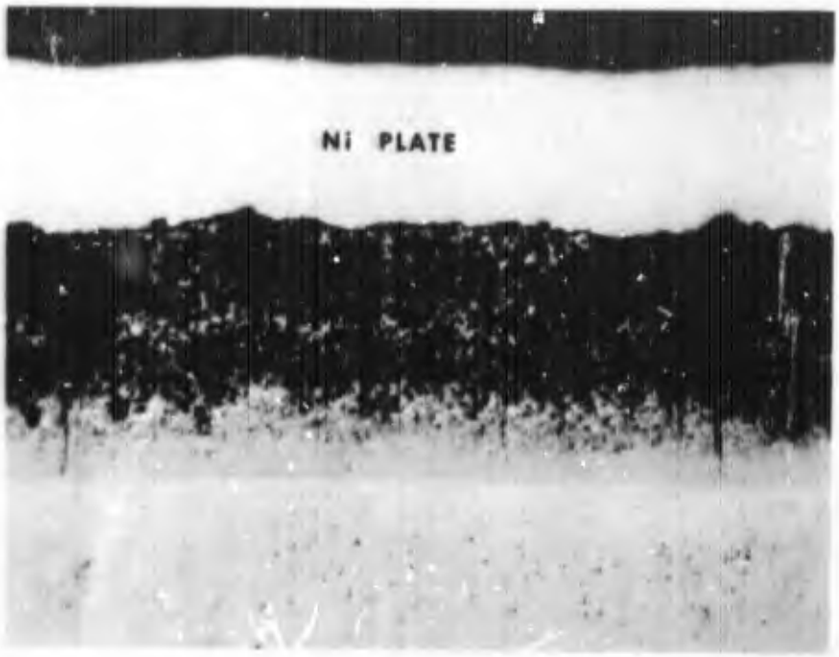
The structure of the resulting coating was similar to both the Phase I coating and Phase II first-modification coating in that no distinct regions were observed in the outer silicide layers (Figure 78). A titanium-rich solid solution zone extended approximately 0.9 mil into the substrate and contained precipitate of type $(Cb,Ti)Cr_2$ Laves phase (Ref. 25). Coating coverage at corners and edges of the 2.2-mil coating was excellent.

3.2.3 Sylcor Ti-CrTi-Si (Triplex) Pack/D-43

Early failure of the Phase I Sylcor Ti-CrTi-Si (triplex) pack coating on D-43 alloy in thermal fatigue was not due to poor coating protectiveness, but, rather, lack of coating coverage on the internal surfaces of the thermal fatigue specimens (configuration schematically illustrated in Figure 25). The following modifications were considered to offer the greatest potential for overall improvement of the coating's performance.

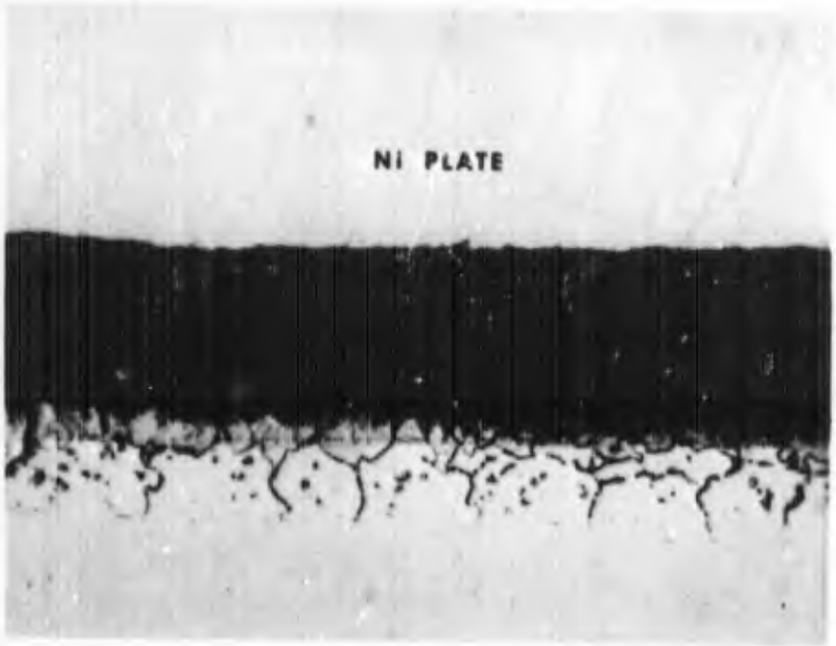
- Development of a greater coating thickness on the airfoil internal surfaces

- Incorporation of more chromium into the coating composition to better develop and increase the continuity of the type $(Cb,Ti)Cr_2$ Laves phase



ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER MAG: 500X

Figure 77. Typical Microstructure of the As-Applied Phase II Modified TRW TiCr-Si (Vacuum) Pack Coating on Cb-132M Columbium Alloy



ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER MAG: 500X

Figure 78. Typical Microstructure of the As-Applied Phase II Remodified TRW TiCr-Si (Vacuum) Pack Coating on Cb-132M Columbium Alloy

- Application of a heavier silicide layer

The modified triplex pack coating was applied utilizing the following parameters:

- Cycle 1: Titanizing at 2200°F for 16 hours in vacuum
- Cycle 2: Vacuum titanium-chromium deposition at 2300°F for 8 hours
- Cycle 3: Siliconizing in purified argon at 1900°F for 16 hours

A 1.1-mil titanium-rich diffusion zone containing a discontinuous precipitate was observed metallographically (Figure 79). The precipitate was presumed to be a type $(Cb,Ti)Cr_2$ Laves phase. Four coating regions were noted: a semicontinuous 0.09-mil layer at the coating-substrate interface, a 0.6-mil dark-etching layer, a light 1.5-mil layer containing a uniformly dispersed dark phase, and a light-colored phase of about 0.9 mils thickness at the coating surface. This light-etching surface layer contained a dark second phase (Figure 79). In some areas of the outer coating layer, this dark phase (presumed to be silicon rich) was continuous (Figure 80).

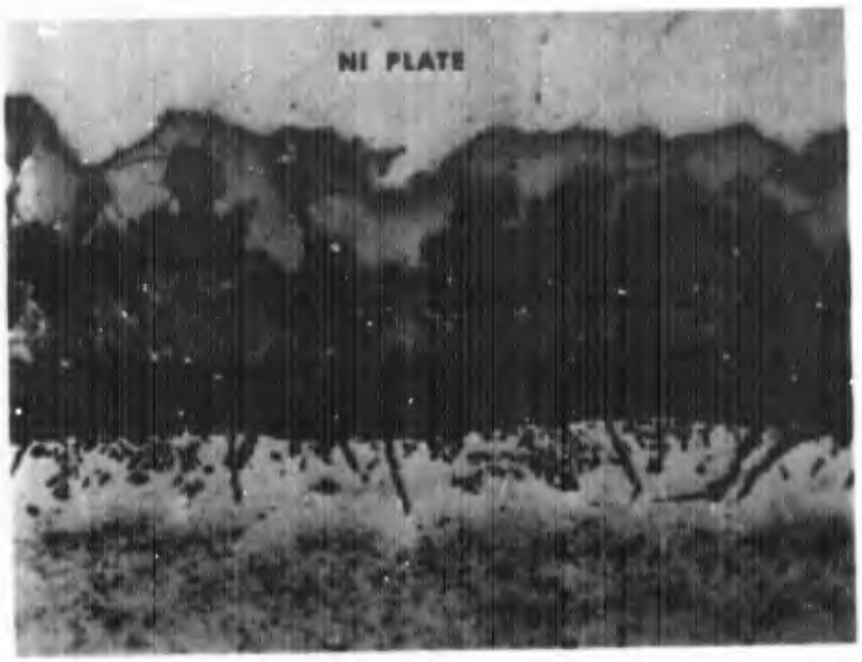
Microcracks penetrated the outer coating layers to the coating-substrate interface layer. Coverage of the 3.1-mil-thick coating was excellent.

3.2.4 Sylcor Sn-Al (505-C)/Cb-132M

The Phase I Sylcor 505-F Sn-Al coating which performed poorly in wear-galling tests contained the maximum molybdenum content commensurate with adequate oxidation resistance. Rather than alter the primary constituents, that is, the tin- and/or aluminum-base compounds, the molybdenum content was therefore decreased. Spray application was followed by a vacuum diffusion treatment to develop the 5.9-mil Phase II coating. Although the coating microstructure of the 505-C coating appeared similar to the microstructure of the Phase I coating, the discontinuous phase in the outer coating layers appeared as twisted stringers (Figure 81) rather than discretely elongated particles as noted previously. Coating coverage was excellent including specimen corners and edges.

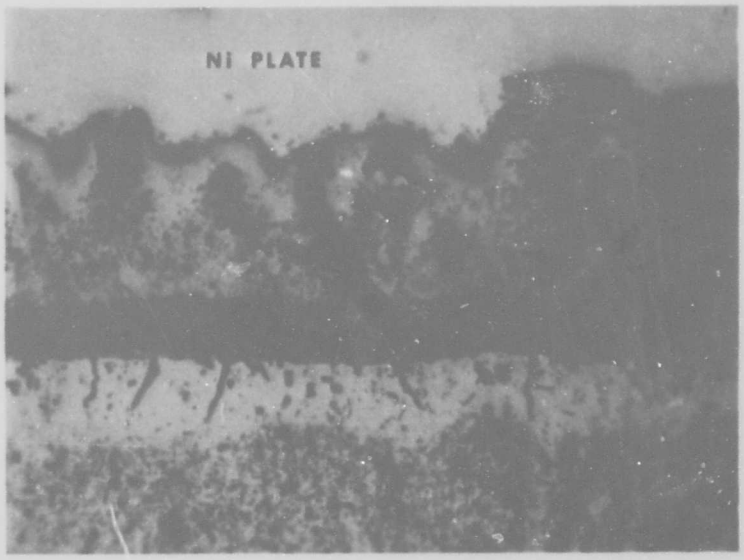
3.2.5 Sylcor Ag-Al-Si (508-F)/Cb-132M

For the Ag-Al-Si coating, it was also decided to modify the molybdenum content to improve wear-galling performance. In this instance the Phase I composition reportedly did not contain the maximum permissible molybdenum, and the concentration was therefore increased for Phase II testing.



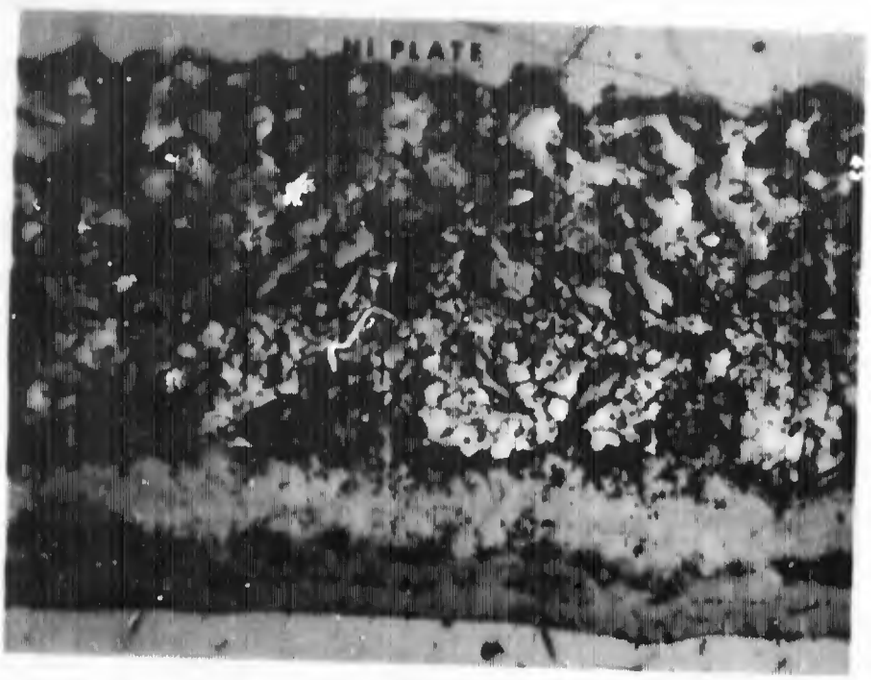
ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER MAG: 500X

Figure 79. Typical Microstructure of the As-Applied Phase II Sylcor Ti-CrTi-Si (Triplex) Pack Coating on D-43 Columbium Alloy



ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER MAG: 500X

Figure 80. Microstructure of the Phase II Sylcor Ti-CrTi-Si (Triplex) Pack Coating on D-43 Columbium Alloy Showing Continuous Dark Outer Surface Layer



ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER MAG: 500X

Figure 81. Typical Microstructure of the As-Applied Phase II Sylcor Sn-Al (505-C) Coating on Cb-132M Columbium Alloy

Four regions were observed metallographically in the Phase II coating (Figure 82). These areas consisted of two 0.3-mil layers at the coating-substrate interface, a homogeneous 3.4-mil layer above the coating-substrate layers, and a 2.7-mil surface layer. The surface layer contained discrete dark etching areas and particles similar in appearance to the 3.4-mil intermediate layer. The 6.5- to 7.0-mil coating was slurry applied and developed in a manner similar to the procedure described previously for the Sn-Al coating. Coating coverage was excellent.

3.3 EVALUATION TESTING, HIGH-TEMPERATURE SYSTEMS

3.3.1 Test Procedures

As mentioned at the beginning of Item 3, appropriate Phase I tests were performed to verify optimization or improvement for each of the modified coating-substrate systems. In fulfilling this requirement, the evaluation procedures for the oxidation-erosion, thermal fatigue, and ballistic impact tests were necessarily identical to those described in paragraph 2.3.1, Phase I test procedures.

3.3.2 Test Results

3.3.2.1 Oxidation-Erosion Results

As in the case of the Phase I oxidation-erosion tests, TRW TiCr-Si coated D-43 erosion specimens were included during some of the Phase II erosion tests as standards.

Modified TRW TiCr-Si (Vacuum) Pack/C-129Y. The modified TRW coating on C-129Y alloy demonstrated significant improvement in oxidation-erosion performance over that of the Phase I unmodified coating. The test period in which no airfoil coating failures were observed was increased from 80 hours at 2200°F to 100 hours at 2200°F plus 40 hours at 2400°F. The maximum protective life of the coating increased from 40 to 60 test hours at 2400°F after 100 hours at 2200°F.

Surface characteristics of the Phase II coating during testing were similar to those of the Phase I coating in that the specimens developed a coarse texture as a result of oxide formation within surface craze cracks prior to failure (Figure 83). Craze crack oxidation led to eventual localized spalling of the outer coating layers and subsequent substrate oxidation.

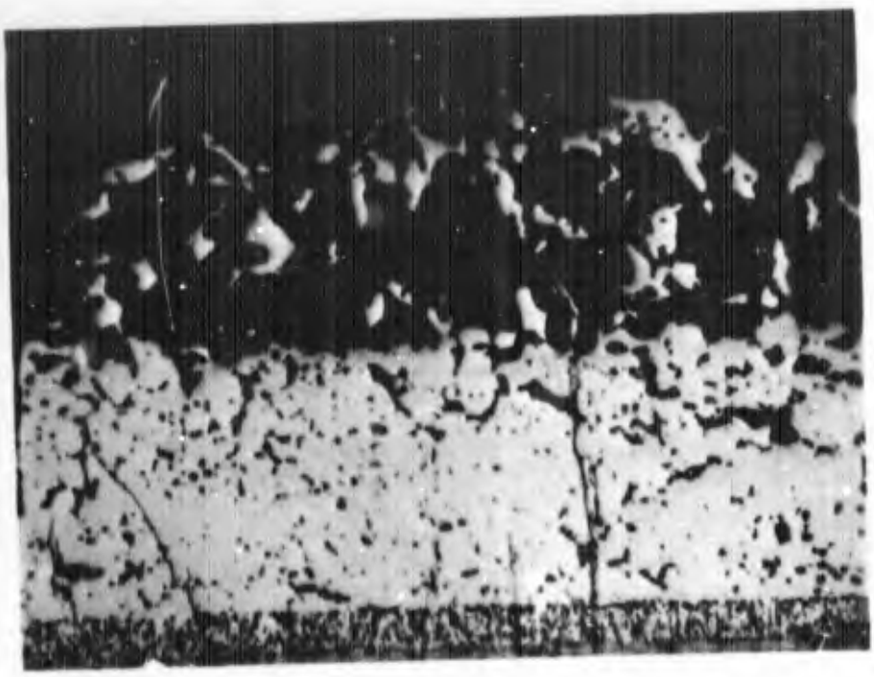


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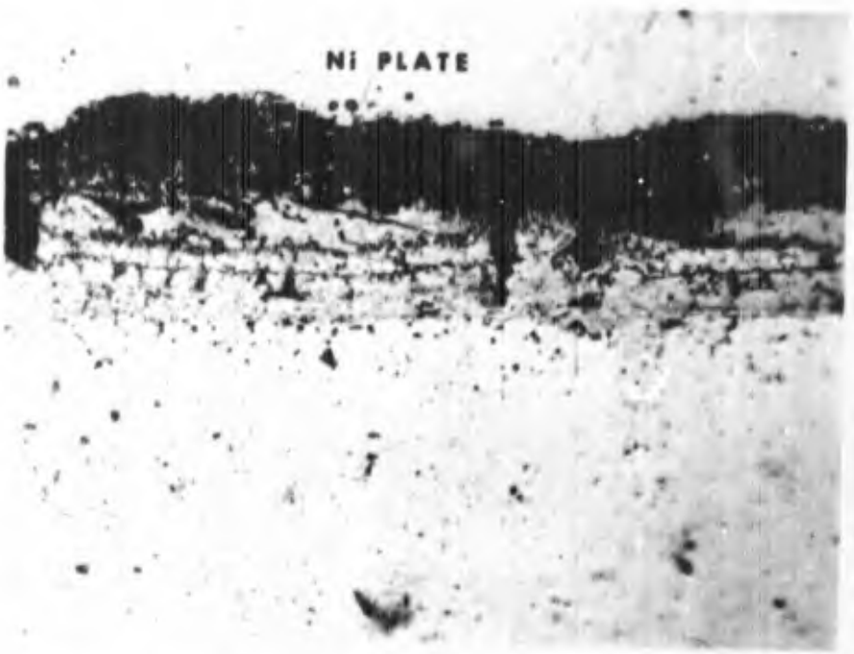
Figure 82. Typical Microstructure of the As-Applied Phase II Sylcor Ag-Al-Si (508-F) Coating on Cb-132M Columbium Alloy



ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER MAG: 500X

Figure 83. Microstructure of the Modified TRW TiCr-Si (Vacuum) Pack Coating on C-129Y Columbium Alloy After 100 Hours at 2200°F Plus 60 Hours at 2400°F in Oxidation-Erosion

A low-temperature oxidation problem was observed for the Phase II TRW coating on C-129Y alloy similiar to that previously noted during Phase I testing of this system. Premature oxidation occurred after about 40 hours in the holder or grip portions of the oxidation-erosion specimens (see Figure 29).

Modified TRW TiCr-Si (Vacuum) Pack/Cb-132M. The initial modification of the TRW coating on Cb-132M alloy produced only a slight improvement in the oxidation-erosion performance. As mentioned previously, the protective life of the Phase I coating was 100 hours at 2200°F plus 40 hours at 2400°F. Although three of four Phase II coated specimens failed at temperatures and times identical to the Phase I specimens, a fourth specimen exhibited a life of 100 hours at 2200°F plus 60 hours at 2400°F, i.e., an increase of 20 hours at the 2400°F temperature.

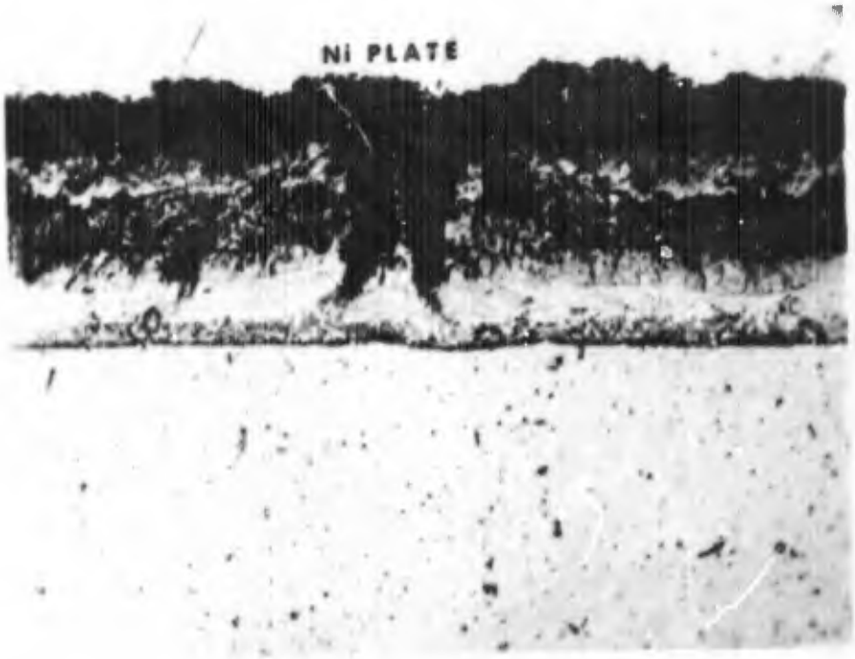
During testing, the coating developed yellowish-white discolorations on flat airfoil surfaces and gray-to-black discolorations on the leading and trailing edge surfaces. Failure occurred by localized oxidation at the leading edge, trailing edge, and/or flat airfoil surfaces. These oxide formations extended out of surface craze cracks (Figure 84) in a manner similar to that described previously for the Phase I unmodified coating.

Remodified TRW TiCr-Si (Vacuum) Pack/Cb-132M. Performance improvement of the remodified TRW coating consisted of an additional 20 test hours at 2400°F for all test specimens. Failure by localized oxidation of the airfoil trailing edge, leading edge, and/or flat airfoil surfaces (Figure 85) occurred after 100 hours at 2200°F plus 60 hours at 2400°F. Coating surface appearance after failure was generally good in areas away from the localized oxidation.

Modified Sylcor Ti-CrTi-Si (Triplex) Pack/D-43. Improvement in the oxidation-erosion performance of the Sylcor modified coating on D-43 alloy consisted of an increase from 80 to 100 hours at 2200°F in which no specimen failures were observed. The maximum coating life was extended from 20 to 60 hours at 2400°F after 100 hours at 2200°F. As in the case of the unmodified coating, the Phase II coating exhibited failures which were localized on the airfoil surfaces. Metallographic examination revealed that the oxide formations extended out of surface craze cracks (Figure 86).

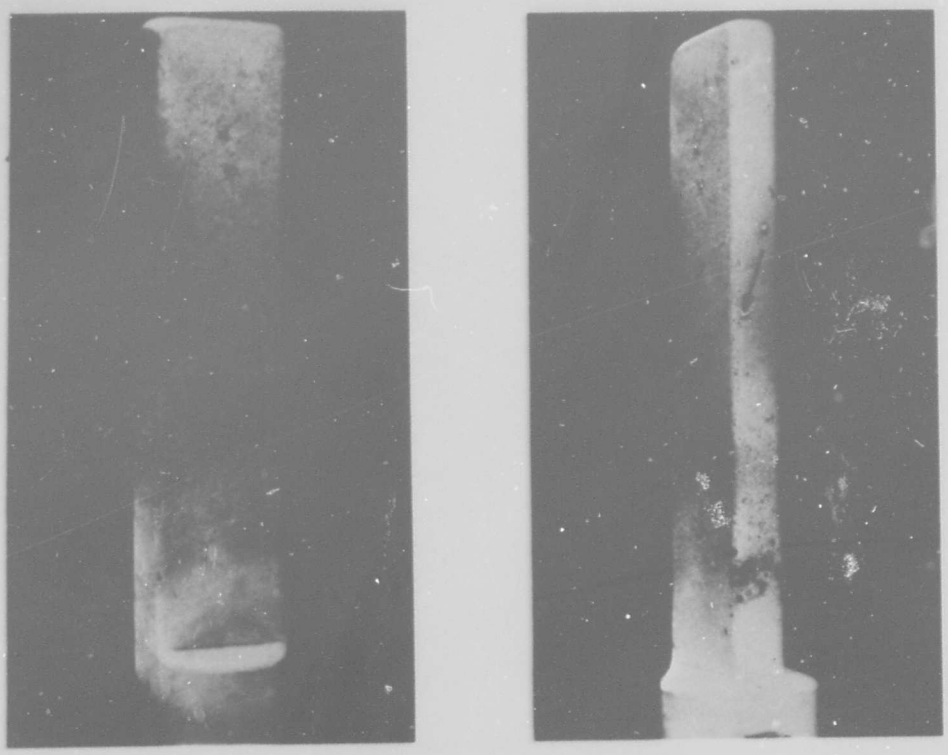
3.3.2.2 Thermal Fatigue Results

Modified TRW TiCr-Si (Vacuum) Pack/C-129Y. The first of two modified TRW TiCr-Si (vacuum) pack coated C-129Y specimens was tested as follows:



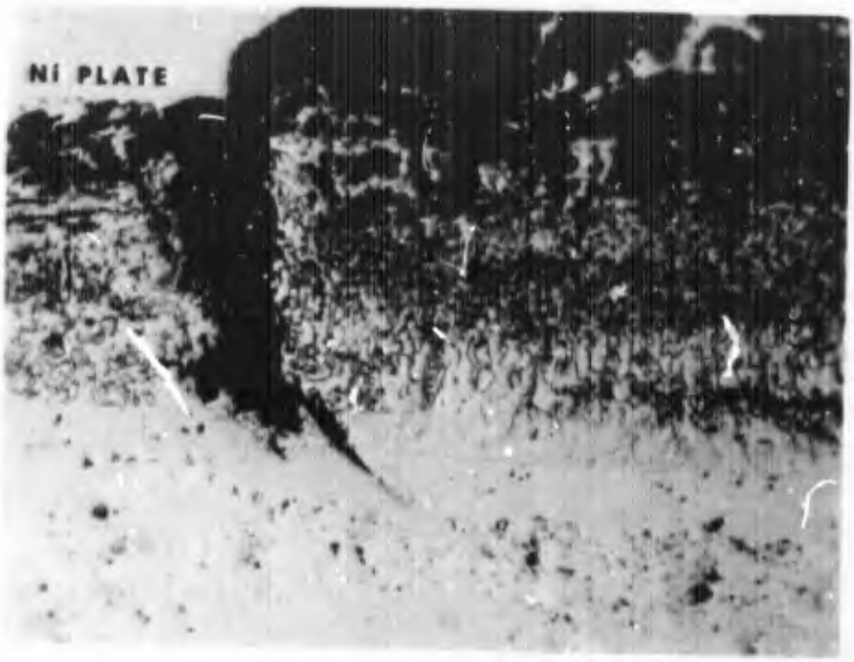
ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER MAG: 500X

Figure 84. Microstructure of the Modified TRW TiCr-Si (Vacuum) Pack Coating on Cb-132M Columbium Alloy After 100 Hours at 2200°F Plus 60 Hours at 2400°F in Oxidation-Erosion



MAG: APPROX. 1.8X

Figure 85. Typical Oxidation-Erosion Bars of Remodified TRW TiCr-Si (Vacuum) Pack Coated Cb-132M Alloy After 100 Hours at 2200°F Plus 60 Hours at 2400°F (arrows locate typical local oxidation failure)



ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33%, 33% WATER MAG: 500X

Figure 86. Microstructure of the Modified Sylcor Ti-CrTi-Si (Triplex) Pack Coating on D-43 Columbium Alloy After 100 Hours at 2200°F Plus 60 Hours at 2400°F in Oxidation-Erosion

- 600 cycles at 2200°F, followed by
- 400 cycles at 2400°F, followed by
- 400 cycles at 2500°F

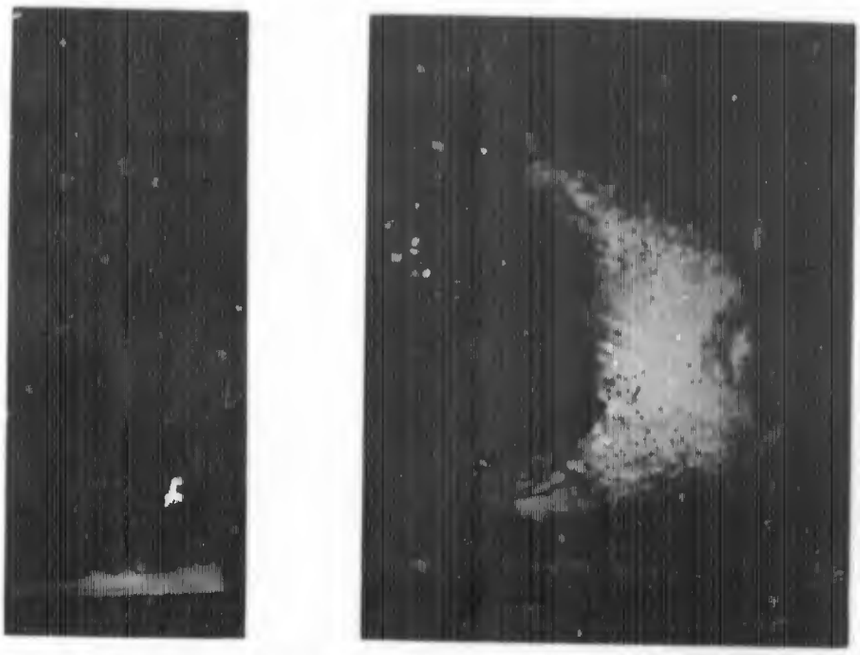
Although no airfoil failures (cracks) were observed at the conclusion of this test, some localized oxidation was noted on airfoil external surfaces (Figure 87). The general performance was superior to that of the unmodified coating in that no gross oxidation was apparent at the trailing edge and the localized oxidation and spalling on the external airfoil surfaces were less severe.

The second thermal fatigue specimen was tested identically. During the last 100 cycles at 2500°F, a hole oxidized through the convex surface of the sheet metal airfoil and a portion of the specimen platform was oxidized (Figure 88). This unusual behavior was believed due to mechanical damage of the test part during the inspection period just prior to the last 100 cycles at 2500°F. In areas away from these failures, local oxidation was evident, but, excluding the mechanically damaged areas, the specimen was in good condition with performance comparable to that of the first test specimen. Localized oxide was metallographically observed to extend out of surface craze cracks (Figure 89).

Modified TRW TiCr-Si (Vacuum) Pack/Cb-132M. No improvement in thermal fatigue performance was noted for the first modification of the TRW coating on Cb-132M alloy. After 400 thermal fatigue cycles at 2200°F, the specimens exhibited localized oxidation and spalling on the airfoil surfaces (Figure 90). Local oxidation resulted from the presence of surface craze cracks, which extended to the substrate in the absence of a continuous protective coating layer at the coating-substrate interface (Figure 91).

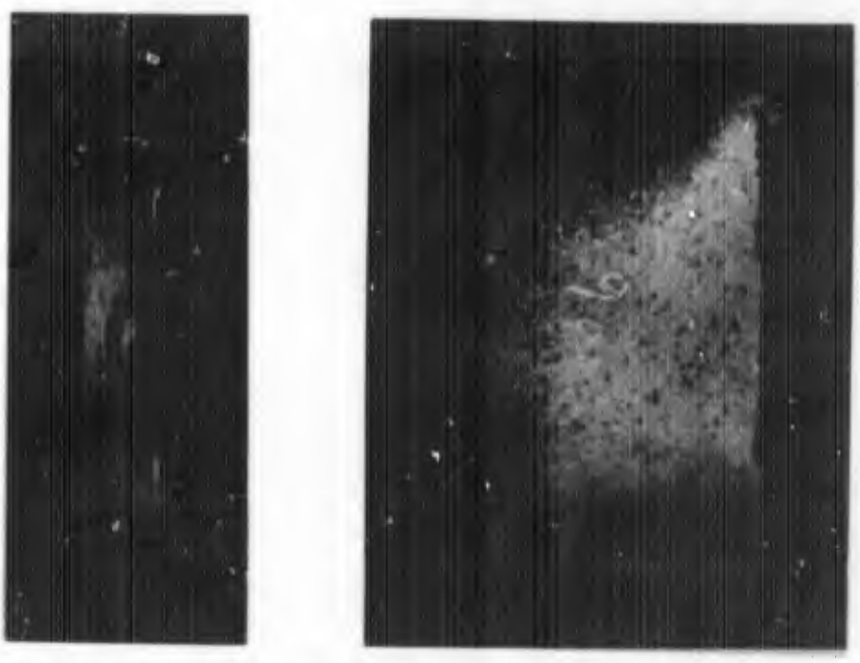
Remodified TRW TiCr-Si (Vacuum) Pack/Cb-132M. The performance of the remodified TRW coating on Cb-132M alloy was significantly better than both the Phase I unmodified and the Phase II modified coatings. While the last two systems failed after only 400 thermal fatigue cycles, no failures were observed on the remodified system until after 600 thermal fatigue cycles at 2200°F and 400 cycles at 2400°F. Failure occurred by local oxidation in spalled areas on the airfoil surfaces (Figure 92).

Modified Sylcor Ti-CrTi-Si (Triplex) Pack/D-43. Two modified Sylcor Ti-CrTi-Si (triplex) pack coated D-43 specimens were tested at 2200°F. After 563 thermal fatigue cycles, the first specimen developed an airfoil crack in a direction parallel to the trailing edge on both the convex and concave surfaces (Figure 93). The second specimen did not exhibit cracking after 600 thermal fatigue cycles (Figure 94). Visible substrate oxide was apparent on



LEADING EDGE SURFACE

CONVEX AIRFOIL SURFACE



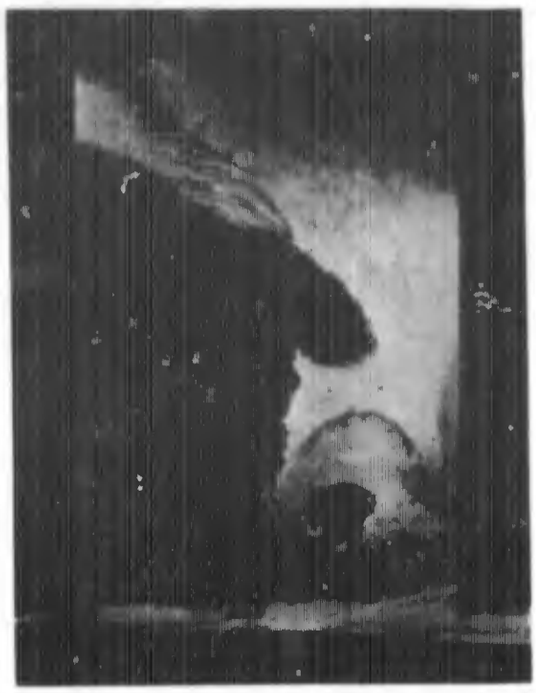
TRAILING EDGE SURFACE

CONCAVE AIRFOIL SURFACE

MAG: 0.8X

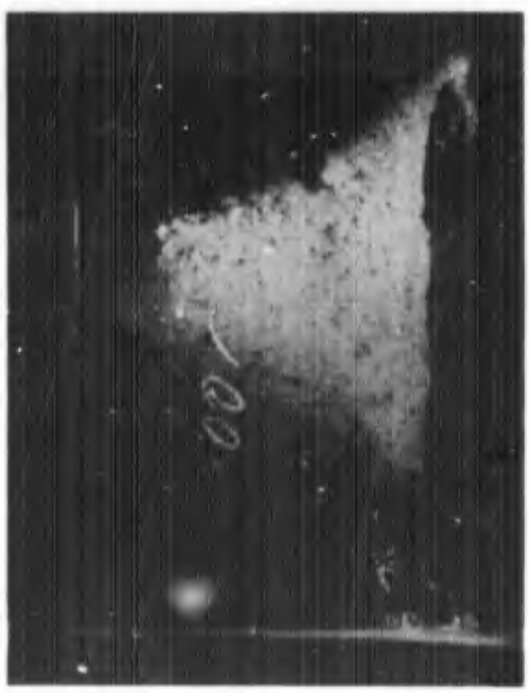
Figure 87. Modified TRW TiCr-Si (Vacuum) Pack Coated C-129Y Columbium Alloy Vane Specimen After 600 Thermal Cycles at 2200°F, 400 Cycles at 2400°F, and 400 Cycles at 2500°F (Specimen No. 1)

LEADING EDGE SURFACE



CONVEX AIRFOIL SURFACE

TRAILING EDGE SURFACE

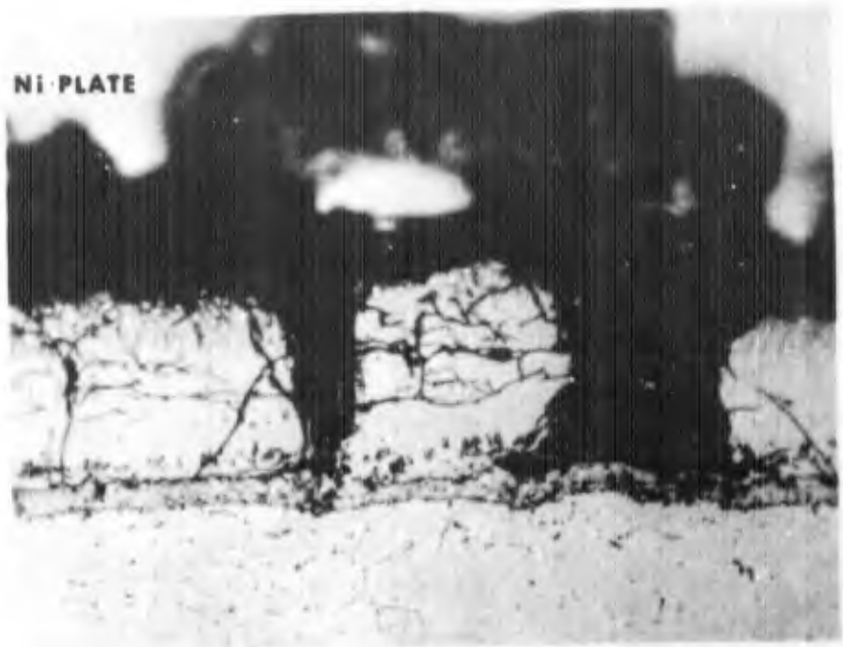


CONCAVE AIRFOIL SURFACE

MAG: 0.9X

Figure 88. Modified TRW TiCr-Si (Vacuum) Pack Coated C-129Y Columbium Alloy Vane Specimen After 600 Thermal Cycles at 2200°F, 400 Cycles at 2400°F, and 400 Cycles at 2500°F (Specimen No. 2)

Hole in airfoil was due to handling damage during interim inspection.



ETCH: 60% LACTIC ACID, 20% HYDROFLUORIC ACID, 20% NITRIC ACID MAG: 500X

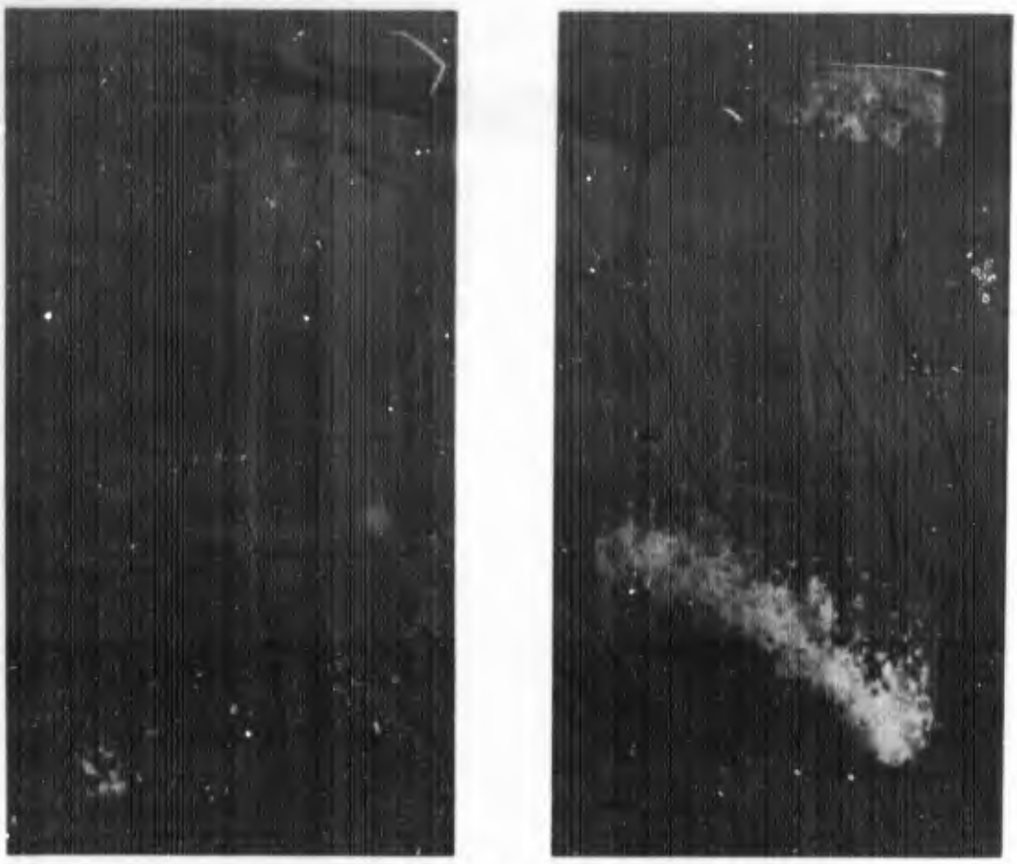
Figure 89. Typical Microstructure of the Modified TRW TiCr-Si (Vacuum) Pack Coating on C-129Y Alloy After Thermal Fatigue Testing for 600 Cycles at 2200°F, 400 Cycles at 2400°F, and 400 Cycles at 2500°F

MAG: 1.2X

Figure 90. Typical Modified TRW TiCr-Si (Vacuum) Pack Coated Cb-132M Alloy Thermal Fatigue Specimens After 400 Cycles at 2200°F (note numerous local oxidation failures)

ETCH: 60% LACTIC ACID, 20% HYDROFLUORIC ACID, 20% NITRIC ACID MAG: 500X

Figure 91. Microstructure of the Modified TRW TiCr-Si (Vacuum) Pack Coating on Cb-132M Alloy After 400 Thermal Fatigue Cycles at 2200°F



CONVEX SURFACE

CONCAVE SURFACE

MAG: 1.8X

Figure 92. Remodified TRW TiCr-Si (Vacuum) Pack Coated Cb-132M Alloy Thermal Fatigue Specimens After 600 Cycles at 2200°F Plus 400 Cycles at 2400°F

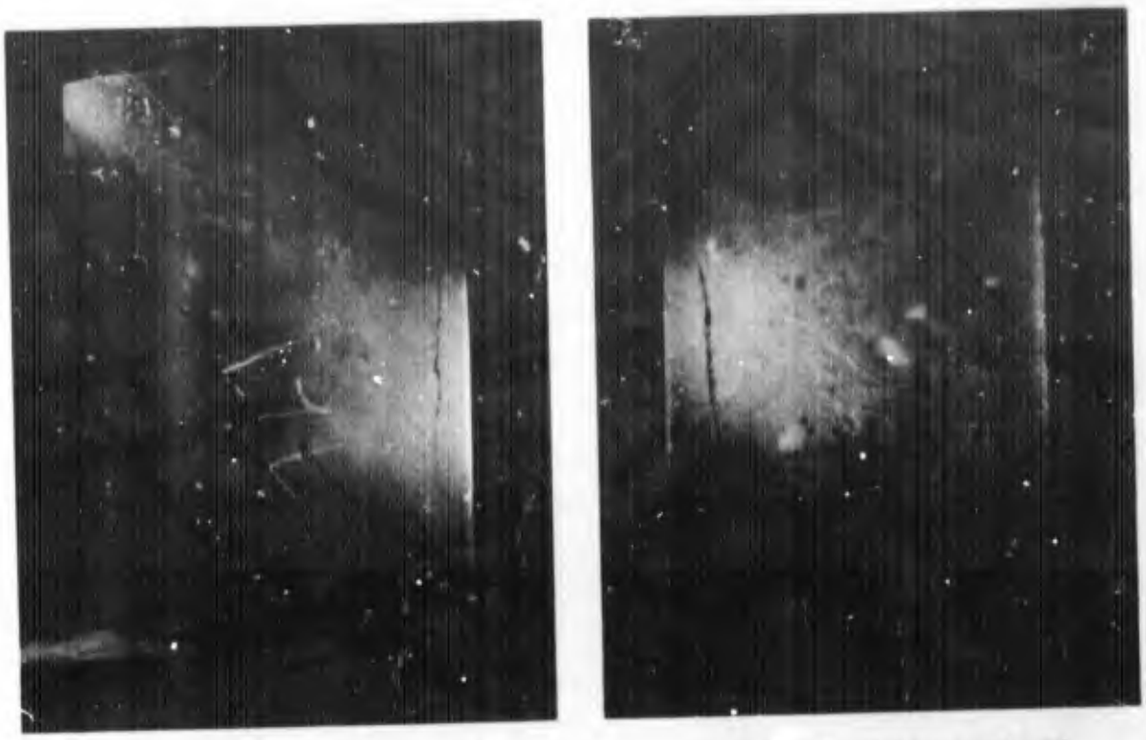
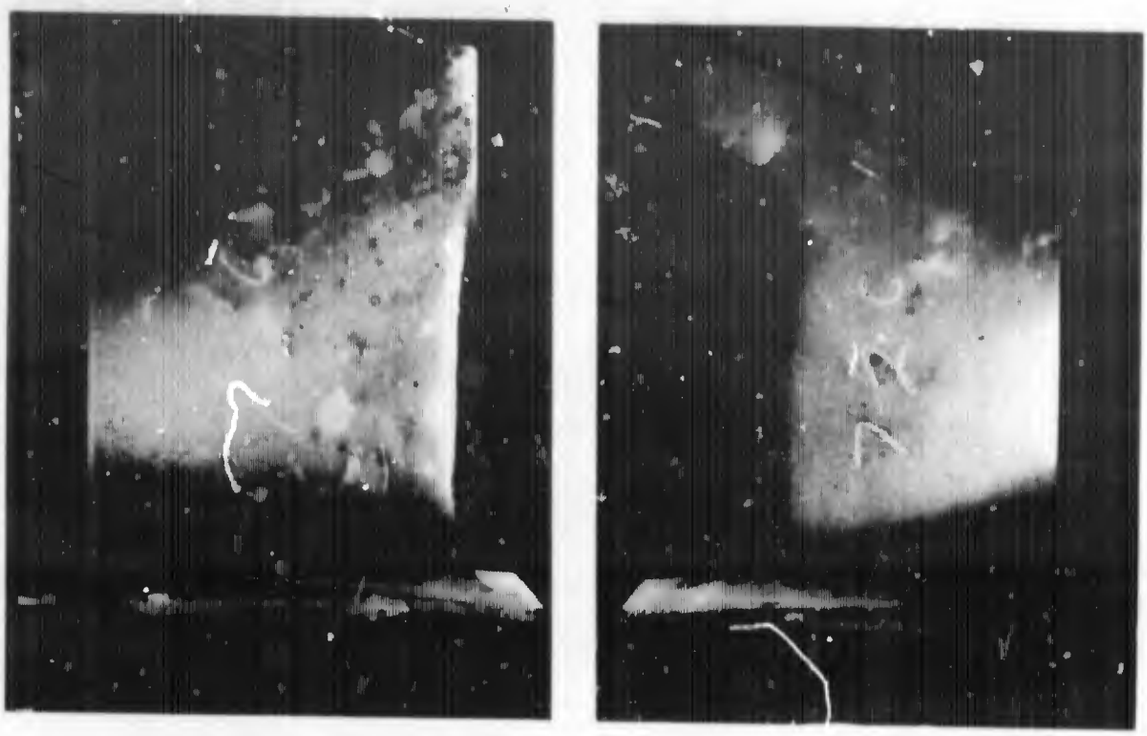


Figure 93. Modified Sylcor Ti-CrTi-Si (Triplex) Pack Coated D-43 Columbium Alloy Vane Specimen After 563 Thermal Cycles at 2200°F (note airfoil crack)



CONCAVE AIRFOIL SURFACE

CONVEX AIRFOIL SURFACE

MAG: 0.9X

Figure 94. Modified Sylcor Ti-CrTi-Si (Triplex) Pack Coated D-43 Columbium Alloy Vane Specimen After 600 Thermal Cycles at 2200°F

the airfoil internal surfaces at the trailing edge after the 2200°F testing, and lack of coating coverage was confirmed by metallographic examination (Figure 95). The appearance of the coating on airfoil external surfaces was very good after testing. However, metallography revealed that oxidation within the titanium-rich solid solution zone was occurring at the base of surface craze cracks (Figure 96). In some cases, craze cracking extended into the substrate (Figure 97).

3.3.2.3 Ballistic Impact Results

Impact characteristics of the Phase II test specimens did not differ appreciably between the applicable coating-substrate systems. After ballistic impacting, the five characteristic zones described previously in paragraph 2.3.2.3 (Phase I Ballistic Impact Results), diagramed in Figure 57, and listed below were noted.

- Zone I, impact depression
- Zone II, scab area surrounding impact depression
- Zone III, unaffected coating
- Zone IV, coating on protrusion opposite the impact depression
- Zone V, scab area surrounding protrusion

Results of postimpact 2200°F oxidation exposure are presented in Table VIII. For all systems, failure occurred in the scab area surrounding the impact depression (Zone II) and/or the scab area surrounding the impact protrusion (Zone V). For all systems except the remodified TRW/Cb-132M, the 2200°F oxidation-exposure life for those specimens impacted at room temperature was less than the life observed after elevated temperature impacting (see Table VIII). The remodified TRW Cb-132M system, however, showed no difference in oxidation life between the room-temperature and 2200°F impacts. The performance of the remodified coating was significantly better than the initial TRW modification.

The generally poorer performance of the Phase II systems in ballistic impact compared to the Phase I data cannot be explained. It is not believed that coating modifications produced this degradation.

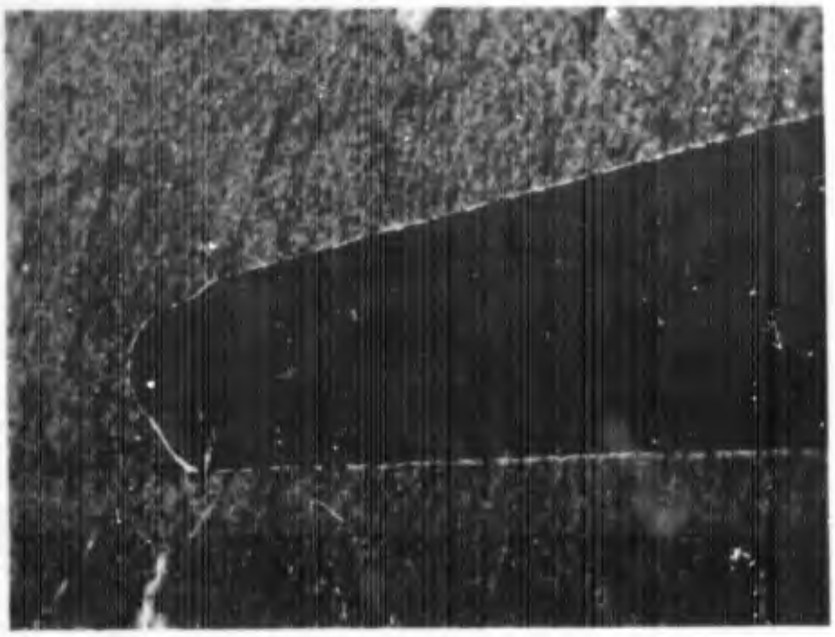


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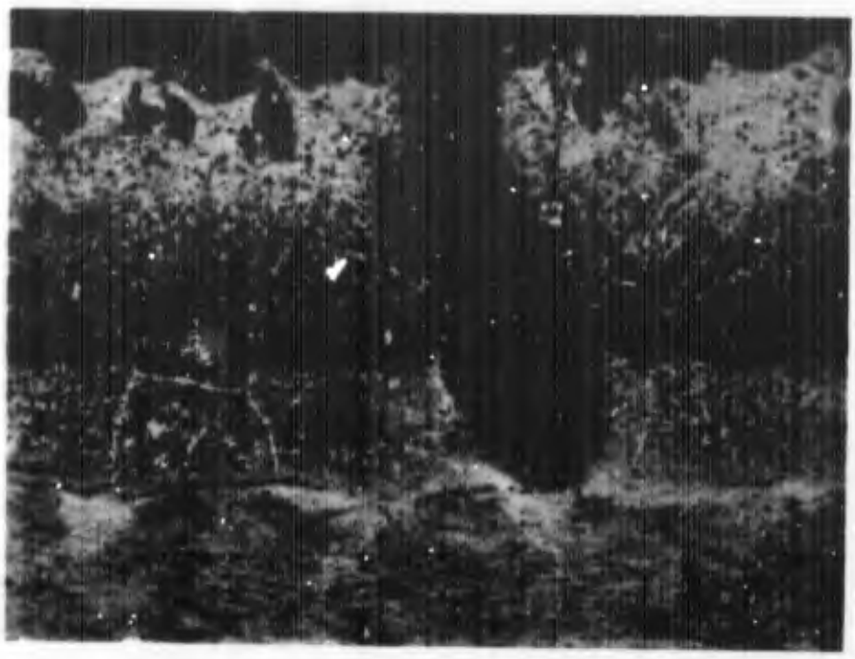
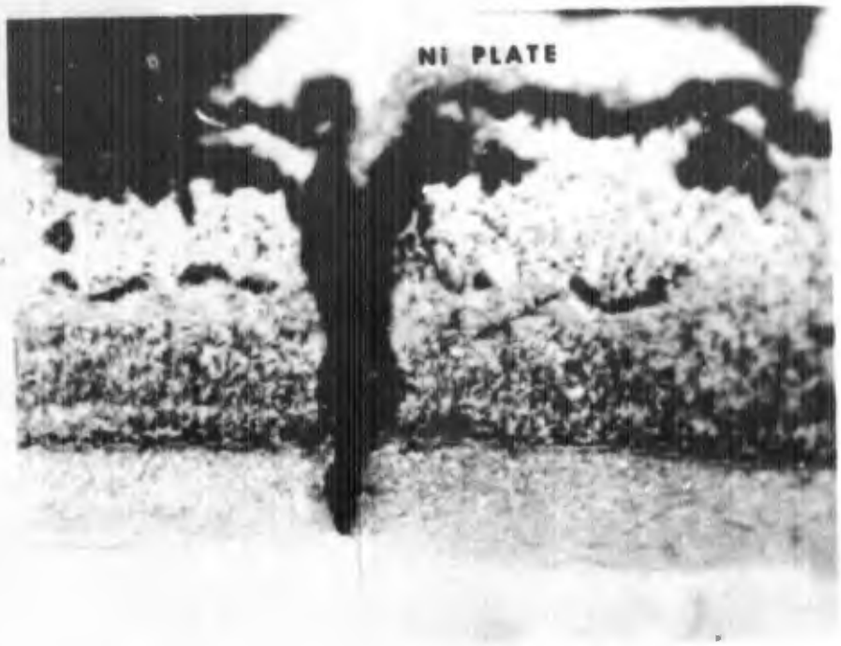
Figure 95. Microstructure at Trailing Edge Internal Surface of Modified Sylcor Ti-CrTi-Si (Triplex) Pack Coated D-43 Columbium Alloy Vane Specimen After 600 Thermal Cycles at 2200°F (note absence of coating)



ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER MAG: 500X

Figure 96. Typical Microstructure at External Surface of Modified Sylcor Ti-CrTi-Si (Triplex) Pack Coated D-43 Columbium Alloy Vane Specimen After 600 Thermal Cycles at 2200°F



ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER MAG: 500X

Figure 97. Microstructure at External Surface of Modified Sylcor Ti-CrTi-Si (Triplex) Pack Coated D-43 Columbium Alloy Vane Specimen After 600 Thermal Cycles at 2200°F (note coating craze crack propagation into substrate)

TABLE VIII

RESULTS OF PHASE II BALLISTIC IMPACT TESTING OF THE VANE AND BLADE AIRFOIL COATING-SUBSTRATE SYSTEMS

Application	Test No.	Specimen No.	Coating	Substrate Alloy	Specimen Temp. at Impact (°F)	Impact Velocity (ft/sec)	Oxidation Life After Impact		Observed Condition After Oxidation Exposure	
							Temp. (°F)	Time (hrs.)	Surface at Impact Point	Surface Directly Behind Impact Point
Vane	1	DBI-2-1	Mod. Sylcor Ti-CrTi-Si (triplex)	D-43	70	200	2200	2	Oxide surrounding impact depression	Oxidation of surface
	2	DBI-2-2	Mod. Sylcor Ti-CrTi-Si (triplex)	D-43	2200	200	2200	3	Oxide surrounding impact depression	No oxide noted
	3	DBI-2-3	Mod. Sylcor Ti-CrTi-Si (triplex)	D-43	70	500	2200	1	Oxide surrounding impact depression	Oxidation of surface
	4	DBI-2-4	Mod. Sylcor Ti-CrTi-Si (triplex)	D-43	2200	500	2200	2	Oxide surrounding impact depression	Oxidation of surface
	5	YBI-2-1	Mod. TRW TiCr-Si (vacuum)	C-129Y	70	200	2200	1	Oxide surrounding impact depression	Oxidation of surface
	6	YBI-2-2	Mod. TRW TiCr-Si (vacuum)	C-129Y	2200	200	2200	2	Oxide surrounding impact depression	No oxide noted
	7	YBI-2-3	Mod. TRW TiCr-Si (vacuum)	C-129Y	70	500	2200	1	Oxide surrounding impact depression	Oxidation of surface
	8	YBI-2-4	Mod. TRW TiCr-Si (vacuum)	C-129Y	2200	500	2200	2	Oxide surrounding impact depression	Oxidation of surface
Blade Airfoil	9	MBI-2-1	Mod. TRW TiCr-Si (vacuum)	Cb-132M	70	200	2200	1	Oxide surrounding impact depression	
	10	MBI-2-2	Mod. TRW TiCr-Si (vacuum)	Cb-132M	2200	200	2200	3	Oxide surrounding impact depression	See Note 3
	11	MBI-2-3	Mod. TRW TiCr-Si (vacuum)	Cb-132M	70	500	--	-	Specimen fractured at impact point	
	12	MBI-2-4	Mod. TRW TiCr-Si (vacuum)	Cb-132M	2200	500	2200	2	Oxide surrounding impact depression	
	13	MBI-2-5	Remodified TRW TiCr-Si (vacuum)	Cb-132M	70	200	2200	5	Oxide beneath an adherent brown skin surrounding impact depression	
	14	MBI-2-6	Remodified TRW TiCr-Si (vacuum)	Cb-132M	2200	200	2200	5	Oxide beneath an adherent brown skin surrounding impact depression	See Note 3
	15	MBI-2-7	Remodified TRW TiCr-Si (vacuum)	Cb-132M	70	500	--	-	Specimen mounted for metallographical observation after impact	
	16	MBI-2-8	Remodified TRW TiCr-Si (vacuum)	Cb-132M	2200	500	2200	5	Oxide beneath an adherent brown skin surrounding impact depression	

Notes:

1. Weight of steel projectile: 0.75 gram each
2. Thickness of substrate: D-43 and C-129Y: 0.050 inch; Cb-132M: 0.125 inch
3. No effects were noted on the surface directly behind the impact point on the coated Cb-132M specimens due to the thickness of these specimens.

3.4 EVALUATION TESTING, LOW-TEMPERATURE SYSTEMS

3.4.1 Test Procedures

To allow comparison of the results, the testing procedures for the Phase II prestrain/oxidation and wear-galling tests were identical to the corresponding Phase I procedure. Although two specimen configurations were screened for each type of test in Phase I, only one type was utilized for Phase II testing. Test procedures were described previously in paragraph 2.4.1.

3.4.2 Test Results

3.4.2.1 Prestrain/Oxidation Results

Sylcor Sn-Al (505-C)/Cb-132M. Two coated Cb-132M modified tensile specimens were prestrained a total of 0.67 and 0.55 percent plastic strain at 1300°F. No visible coating degradation was noted after prestraining or after a subsequent 8-hour static oxidation exposure at 1300°F. Metallographic examination of the gage section after prestraining and exposure revealed no degradation in the outer coating layers, but cracks were observed in coating layers adjacent to the substrate (Figure 98). Substrate oxidation was apparently prevented by the thick, continuous outer coating layers.

Sylcor Ag-Al-Si (508-F)/Cb-132M. Prestraining the Phase II Ag-Al-Si (508-F) coated specimens at 1300°F for a total of 0.51 and 0.60 percent plastic strain resulted in no visible degradation of the coating (see Table IX). Although no oxidation of the specimens was observed after an 8-hour oxidation exposure at 1300°F, metallographic examination revealed cracks in the coating layers adjacent to the substrate (Figure 99). Some areas of base-metal cracking were also noted on these specimens (Figure 100). As in the case of the Phase II Sn-Al system, the outer coating layers provided protection for the ruptured areas near the coating-substrate interface.

3.4.2.2 Wear-Galling Results

Sylcor Sn-Al (505-C)/Cb-132M and Sylcor Ag-Al-Si (508-F)/Cb-132M. Both of the Phase II modified blade root coatings were evaluated using the dovetail wear-galling specimen configuration (Figure 68). The results of the Phase II wear-galling tests are summarized in Table X. After 4.5×10^5 cycles at 1300°F, both Sn-Al coated test specimens suffered minor braze bonding to the nickel-base turbine disk alloy at the contact surfaces.

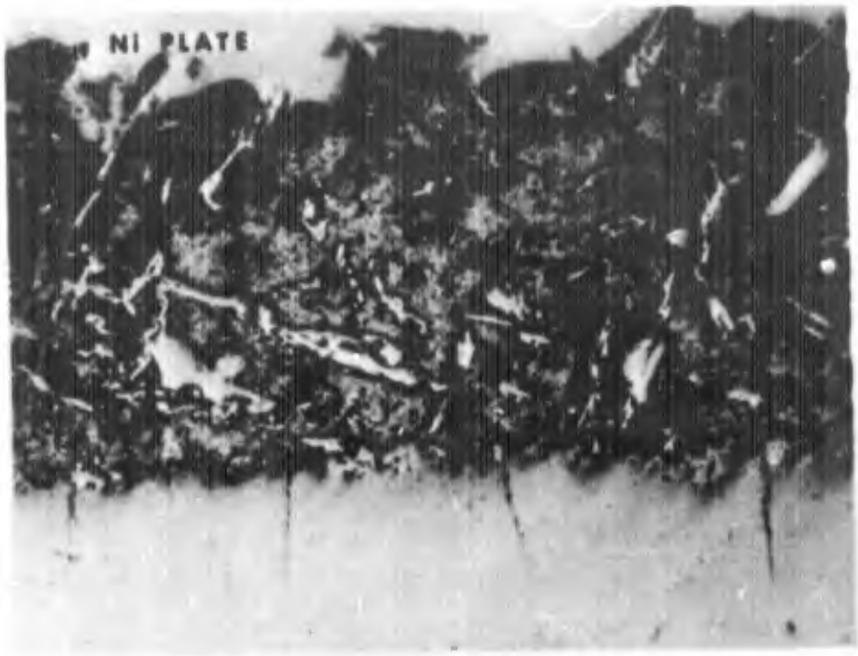


Figure 98. Microstructure of Sylcor Sn–Al (505-J) Coating on Cb–132M Columbium Alloy After 1300°F Prestraining to 0.55-Percent Plastic Strain and 1300°F Oxidation Exposure for 8 Hours (note cracks in coating layer adjacent to substrate)

TABLE IX

PRESTRAIN/OXIDATION TEST RESULTS FOR THE PHASE II COATED Cb-132M ALLOY SPECIMENS FOR BLADE ROOT APPLICATION

Test No.	Specimen	Coating	Prestrain Temp. (°F)	Proportional Limit (psi)	Stress at Termination Of Load (psi)	Total Plastic Strain (percent)	Remarks
1	MTSO-2-1 modified	Sn-Al (505-C)	1300	56,700	66,700	0.67	No visual coating failure after prestraining and subsequent 8-hour oxidation exposure at 1300°F
2	MTSO-2-2 modified	Sn-Al (505-C)	1300	56,100	64,300	0.55	Same as above
3	MAASO-2-1 modified	Ag-Al-Si (508-F)	1300	49,300	64,700	0.51	No visual coating failure after prestraining and subsequent 8-hour oxidation exposure at 1300°F
4	MAASO-2-2 modified	Ag-Al-Si (508-F)	1300	50,200	67,700	0.60	Same as above

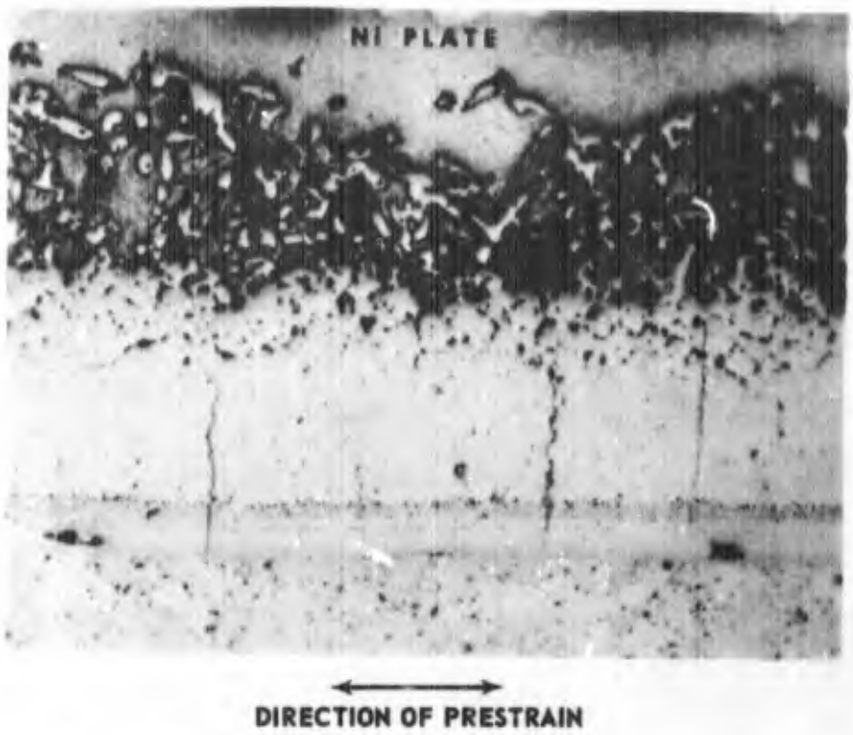
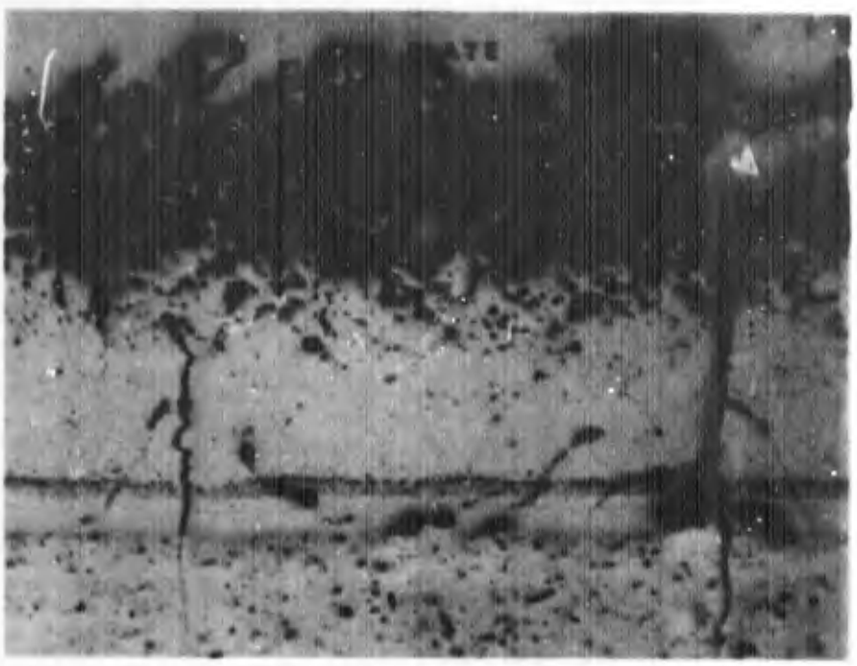


Figure 99. Microstructure of Sylcor Ag-Al-Si (508-F) Coating on Cb-132M Columbium Alloy After 1300°F Prestraining to 0.51-Percent Plastic Strain and 1300°F Oxidation Exposure for 8 Hours (ncte cracks in coating layers adjacent to substrate)



DIRECTION OF PRESTRAIN

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Figure 100. Microstructure of Sylcor Ag-Al-Si (508-F) Coating on Cb-132M Columbium Alloy After 1300°F Prestraining to 0.60-Percent Plastic Strain and 1300°F Oxidation Exposure for 8 Hours (note cracks in both coating and substrate)

TABLE X

WEAR-GALLING TEST RESULTS (1300°F) FOR THE PHASE II COATED Cb-132M ALLOY DOVETAIL SPECIMENS

Rubbing surface: PWA 687 (Waspaloy) nickel-base turbine disk alloy
Tensile load: 2000 psi
Vibratory load: ±200 psi
Specimen vibration frequency: 3600 cycles per minute

Coating	Number of Tests	Total Test Cycles	Specimen Condition After Test	Specimen Condition After 1300°F Oxidation Exposure
Sn-Al (505-C)	2	4.5×10^5	Minor braze bonding in wear-galling areas but more bonding than with the Phase I Ag-Al-Si (508-C); some flow of coating out of load-bearing areas; no visible oxidation but outer coating surfaces removed in some areas	Load-bearing surface unaffected after 4 hours; no noticeable oxidation on the wear-galling surfaces after 24 hours
Ag-Al-Si (508-F)	2	4.5×10^5	Minor braze bonding in the wear-galling areas; coating smeared, but the load-bearing surfaces are in good condition	Yellow discolorations after 4 hours but no observed coating failures; after 24 hours, yellow discolorations extended but no distinct areas of oxide formation

Some smearing and flow of coating away from the load-bearing areas were noted (Figure 101). However, no oxidation was observed on the wear-galling surfaces after a subsequent 24-hour static air exposure at 1300°F. The Ag-Al-Si coating also suffered minor braze bonding in the wear-galling contact areas. Although the coating smeared in the load-bearing areas, the contact surfaces after 4.5×10^5 cycles at 1300°F were in better condition than those on the Sn-Al coated specimens (Figure 102). No coating failures were noted during the 24-hour static air exposures at 1300°F.

3.5 PERFORMANCE COMPARISONS OF THE PHASE I AND PHASE II COATINGS

3.5.1 High-Temperature Systems

3.5.1.1 Oxidation-Erosion

The performances of the Phase II modified coatings were in all cases better than those observed for the Phase I unmodified coatings. All data are graphically presented in Figure 103.

3.5.1.2 Thermal Fatigue

The thermal fatigue performance of the Phase I and Phase II systems is summarized in Figure 104. The only Phase II coating demonstrating a significant improvement over the corresponding Phase I coating was the remodified TRW TiCr-Si (vacuum) pack coating on Cb-132M alloy. In most cases, however, the Phase I unmodified coatings had suffered a greater surface degradation although the coating life may have been equivalent to the Phase II modified coating.

3.5.1.3 Ballistic Impact

The Phase I and Phase II ballistic impact results were summarized in Tables IV and VIII, respectively. As mentioned previously, no significant performance differences between the unmodified and modified coatings were observed.

MAG: APPROX. 4X

Figure 101. Sylcor Sn-Al (505-C) Coated Cb-132M Columbium Alloy Dovetail Specimen Showing Wear-Galling Area (bracket) After 4.5×10^5 Cycles at 1300°F (note coating damage)

MAG: APPROX. 4X

Figure 102. Sylcor Ag-Al-Si (508-F) Coated Cb-132M Columbium Alloy Dovetail Specimen Showing Wear-Galling Area (bracket) After 4.5×10^5 Cycles at 1300°F (note coating damage)

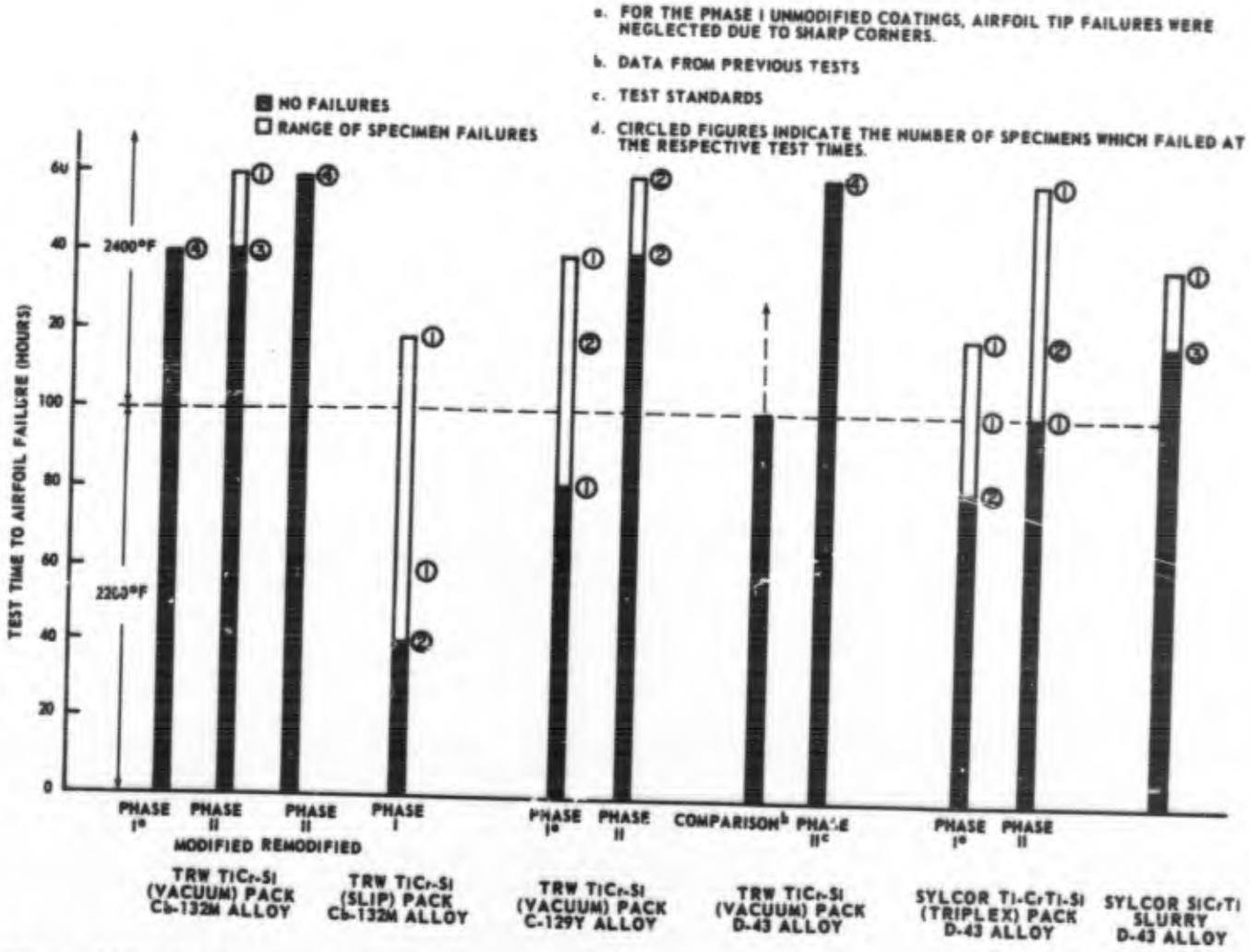


Figure 103. Oxidation-Erosion Performance Comparisons for the Phase I and Phase II Coating-Substrate Systems

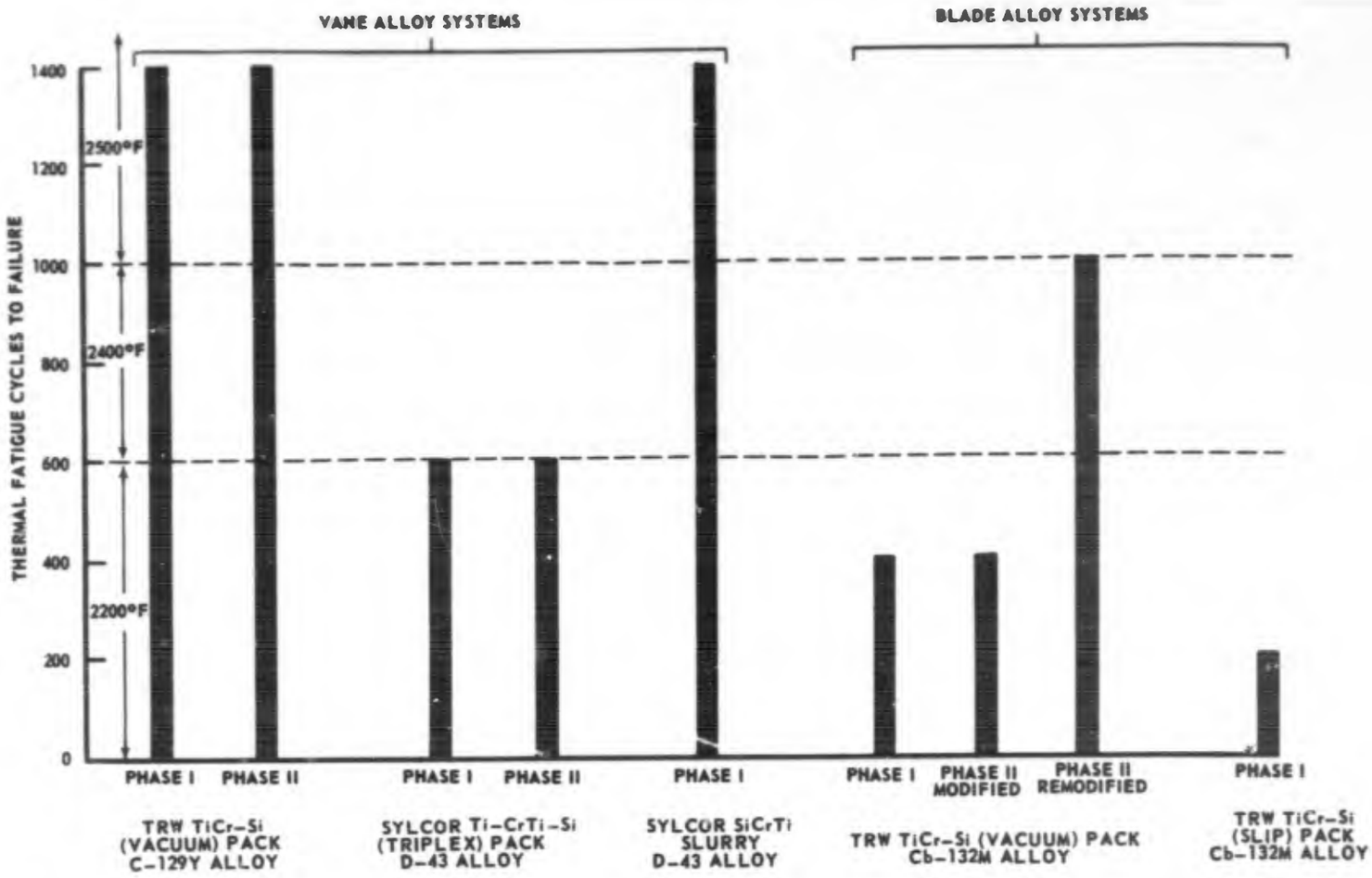


Figure 104. Thermal Fatigue Performance Comparisons for the Phase I and Phase II Coating-Substrate Systems

3.5.2 Low-Temperature Systems

3.5.2.1 Prestrain/Oxidation

The performances of the Phase I and Phase II coatings in the prestrain/oxidation tests did not differ significantly (see Tables V and IX). As discussed previously, the low ductility of the available Cb-132M alloy bar considerably restricted plastic prestraining.

3.5.2.2 Wear-Galling

Although Phase I and Phase II coating performance was generally poor for all the blade root systems, as judged by wear-galling surface appearance after testing, the Sylcor Ag-Al-Si (508-F) coating performance was the best. These results were previously summarized in Tables VI and X.

4. ITEMS 4 AND 5

ADVANCED EVALUATION

Item 4, Coating Life Evaluation, and Item 5, Composite Properties Evaluation, constitute Phase III of the program.

Item 4 requires that the contractor perform both erosion and thermal cycling tests until failure on the optimized blade and vane coating (or coatings), and also static oxidation testing until failure on the optimized blade root coating (or coatings), to determine the protectiveness, diffusional stability, and reliability of the coating system (or systems), as well as ability to withstand transient temperature overshoots.

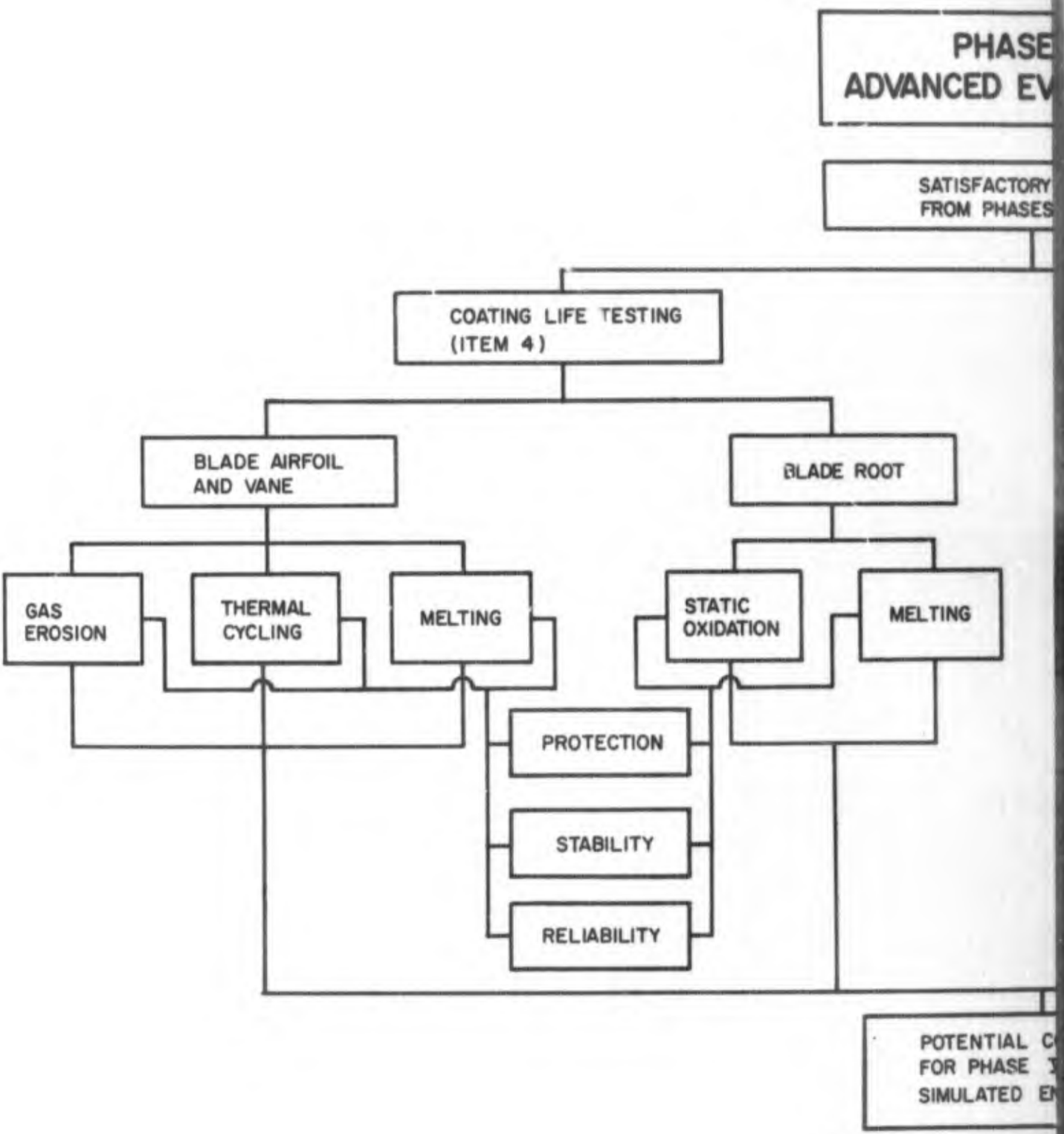
Item 5 requires that the contractor perform the appropriate tests on coated alloy composite specimens to determine their thermomechanical properties: stress-rupture and impact testing on the turbine vane composite(s); fatigue testing, creep testing, and ballistic impact testing on the blade airfoil composite(s); and fatigue testing on the blade root composite(s).

A flow chart summarizing the tests and integration of both Items to form Phase III is presented in Figure 105. The primary purpose of Advanced Evaluation is to obtain quantitative data to assess the advisability of engine testing and aid in the design of components.

4.1 MATERIALS

The selection of the Phase III alloys and coatings was based on the performance of the Phases I and II blade airfoil, blade root, and vane systems. Since Cb-132M was the only blade alloy in the program, it was necessarily forwarded to Phase III. The selection of D-43 as the Phase III vane alloy was based on the following considerations:

- TRW TiCr-Si coated D-43 exhibited the best performance in oxidation-erosion.
- No fabrication difficulties were experienced with D-43, while weld cracking was encountered during construction of C-129Y alloy sheet thermal fatigue specimens.
- Coated D-43 alloy oxidation-erosion bars did not show low-temperature oxidation failures at specimen grips; such failures consistently occurred on C-129Y alloy specimens after only 40 hours at airfoil temperatures of 2200°F.


PHASE
ADVANCED EV
SATISFACTORY
FROM PHASES
COATING LIFE TESTING
(ITEM 4)
BLADE AIRFOIL
AND VANE
BLADE ROOT
GAS
EROSION
THERMAL
CYCLING
MELTING
STATIC
OXIDATION
MELTING
PROTECTION
STABILITY
RELIABILITY
POTENTIAL C
FOR PHASE
SIMULATED E

A

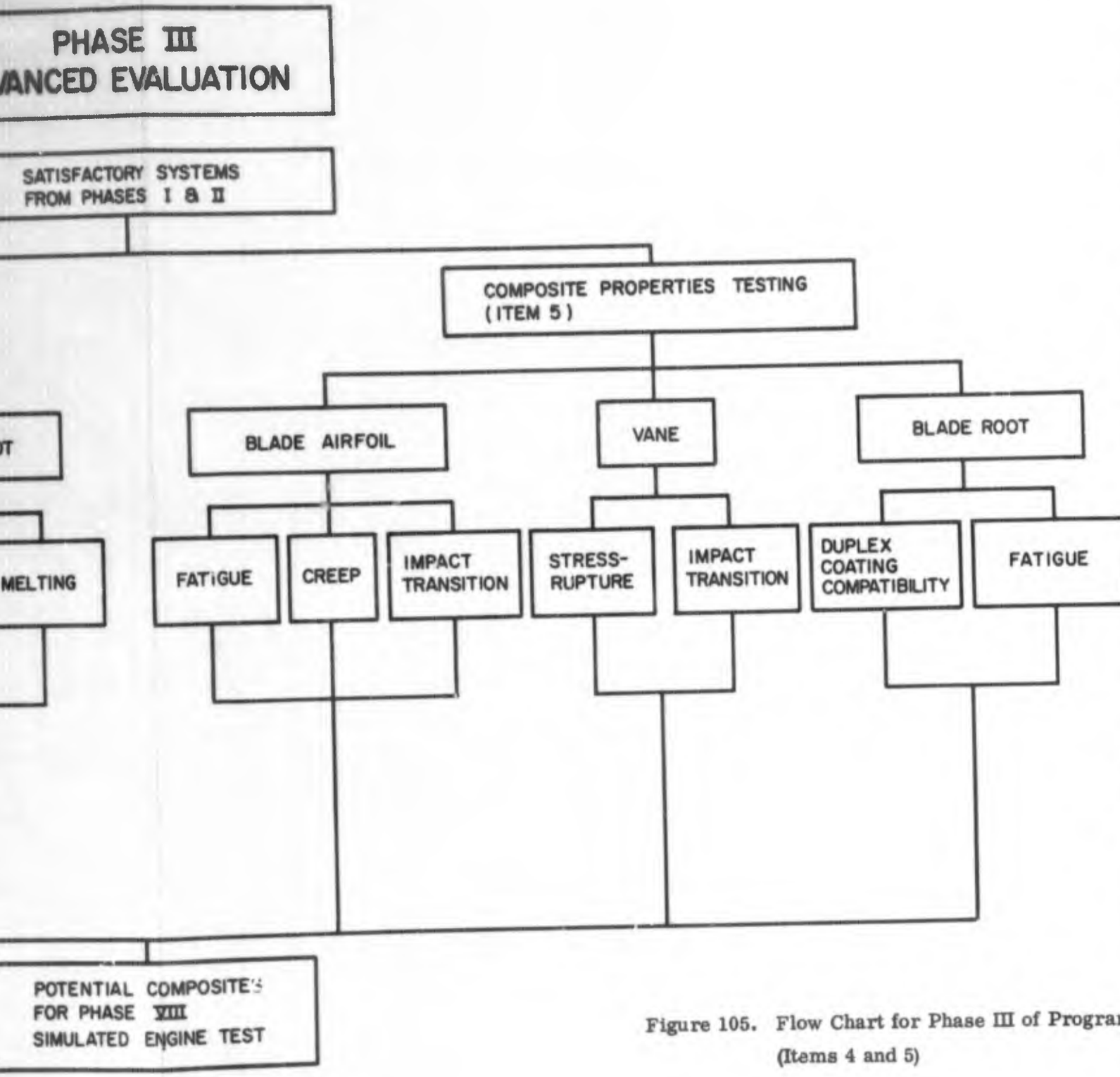


Figure 105. Flow Chart for Phase III of Progra
(Items 4 and 5)

Based on adequate performance in both oxidation-erosion and thermal fatigue, the remodified TRW TiCr-Si (vacuum) pack coating on Cb-132M alloy was selected for advanced evaluation as a blade airfoil system.

The thermal fatigue performance of the modified TRW TiCr-Si (vacuum) pack coating was satisfactory and was the better of the vane system coatings evaluated. Based on this performance, the coating was selected for evaluation in Phase III of the program. Although the Sylcor SiCrTi slurry coating performed well on D-43 alloy in thermal fatigue, as a supplemental system it was not a candidate for Phase III.

The Sylcor Ag-Al-Si (508-F) coating was selected for Phase III blade root evaluation due to its better performance than the Sn-Al coating in the wear-galling tests.

4.2 COATINGS APPLICATION

4.2.1 Blade Airfoil System

Application of the TRW TiCr-Si (vacuum) pack coating for Phase III testing is awaiting the receipt of additional Cb-132M alloy material now being procured. The 2-cycle vacuum pack treatment to be used will utilize the following parameters:

- Cycle 1: A 50Cr-50Ti weight-percent pack, prepared from crushed, prealloyed material, will be used in the first low-pressure cycle at 2200°F for 8 hours with a potassium fluoride activator.

- Cycle 2: A low-pressure siliconizing cycle, also utilizing a potassium fluoride activator, will be performed at 2050°F for a period of 4 hours.

4.2.2 Vane System

The 2-cycle application of the TRW TiCr-Si coating on D-43 alloy was performed in the following manner:

- Cycle 1: A pack alloy of 60Cr-40Ti (weight percent) was utilized in combination with a potassium fluoride activator. The low-pressure cycle was conducted at 2200°F for 8 hours under restricted gas flow to favor the deposition of a higher concentration of titanium.

- Cycle 2: The low-pressure siliconizing cycle was performed at 2050°F for 4 hours with a potassium fluoride activator.

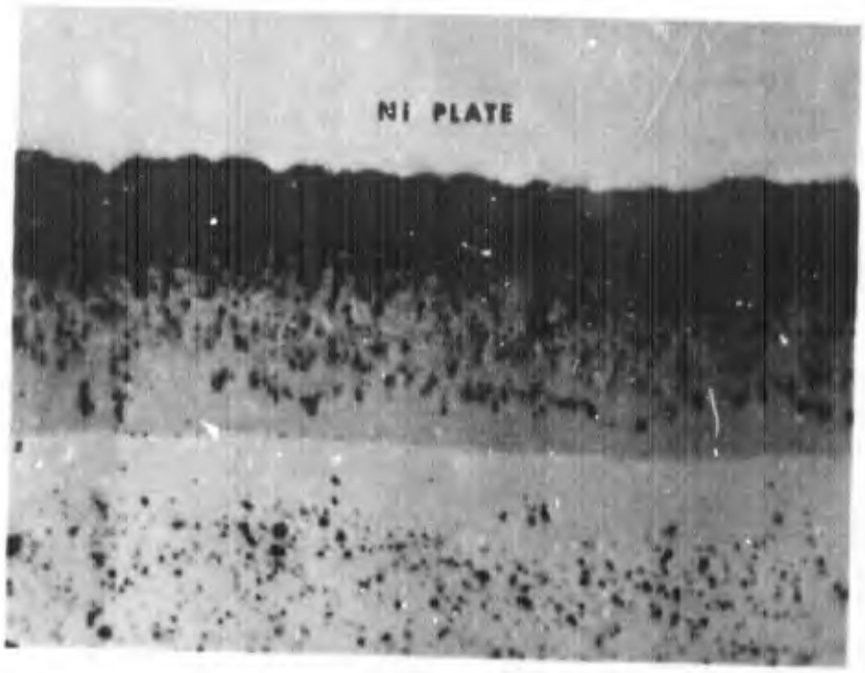
Metallographic examination of the resultant 2.4-mil-thick coating revealed three microstructurally different layers in addition to a titanium-rich solid solution zone extending into the substrate approximately 0.8 mil (Figure 106). The first of these coating layers was a dense 0.2-mil region at the coating-substrate interface. The intermediate layer was 1.3 mils thick and was fairly dense and homogeneous except for the last 0.4 mil, which contained discontinuous regions similar in appearance to portions of the outer layer. The 0.9-mil-thick outer layer was heterogeneous in that it appeared to contain several phases.

Although cracks resulting from coating-substrate thermal expansion differences were observed, these cracks were confined to the outer silicide layers.

Diamond pyramid hardness, utilizing a 20-gram load, was performed in each region with the following results:

Region	Distance from Coating-Substrate Interface (mils)	Average Hardness (DPH)
Outer coating layer	2.0	665
Intermediate coating layer	0.3	1295
Coating layer adjacent to substrate	0.1	1295
Substrate	-0.2	260
Substrate	-2.0	192
Substrate	-3.0	178
Substrate prior to coating	---	189

Concentration profiles obtained by electron microprobe scanning from the base metal through the coating to the nickel-plate overlay produced results which were in good agreement with reported values for chromium, titanium, silicon, and columbium in the TRW TiCr-Si (vacuum) pack coating on D-43 columbium alloy (Ref. 29, 30). The electron microprobe trace across the coating-substrate composite is presented in Figure 107. These



ETCH: 60% WATER, 20% HYDROFLUORIC ACID, 20% NITRIC ACID MAG: 500X

Figure 106. Microstructure of the As-Applied Phase III TRW TiCr-Si (Vacuum) Pack Coating on D-43 Columbium Alloy

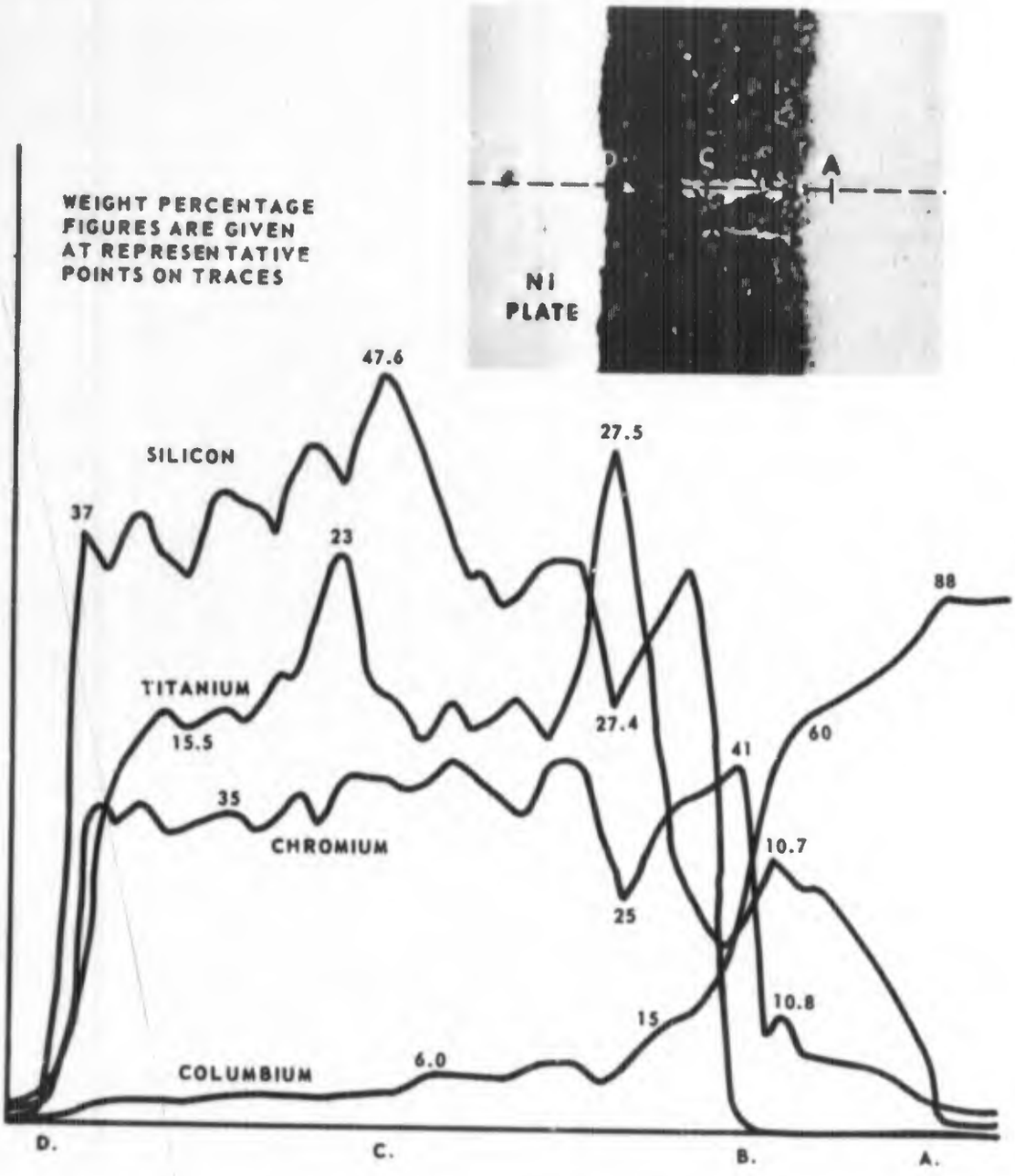


Figure 107. Concentration Profiles for TRW TiCr-Si (Vacuum) Pack Coated D-43 Columbium Alloy as Determined by Electron Microprobe Analyses

results demonstrated that the interfacial coating layer was rich in columbium, titanium, and chromium, probably in the form of (Cb, Ti)Cr_2-type Laves phase, and contained a small quantity of silicon. Both outer layers (extending from the interface outward) contained decreasing amounts of columbium and titanium, an approximately constant concentration of chromium, and slightly increasing amounts of silicon. The outer coating layers were predominantly silicon and have been described compositionally as being silicide regions with varying amounts of chromium, titanium, and columbium (Ref. 30).

4.2.3 Blade Root System

As discussed previously under Phase II Coatings Application (see paragraph 3.2.5), the Ag-Al-Si (508-F) coating contains molybdenum in the outer coating layers. The slurry-applied coating was developed by a high-temperature vacuum diffusion cycle and varied in thickness from 3.9 to 5.5 mils (Figure 108). Although a fairly homogeneous 2.6-mil layer was observed adjacent to the substrate, the outer coating layer contained several phases. Microhardness values for these areas and the adjacent substrate are presented in Figure 109.

Microcracks were observed in the Ta-, W-, Cb-, and Si-rich coating layer adjacent to the substrate; however, these cracks were absent in the outer coating regions.

Electron microprobe X-ray scanning images (Figure 110) revealed the relative concentration of constituents in each region. X-ray diffraction analysis of the coating surface identified the following phases: silver (face centered cubic) molybdenum $(Si,Al)_2$, silicon (diamond cubic), and molybdenum (body centered cubic).

4.3 SYSTEMS EVALUATION

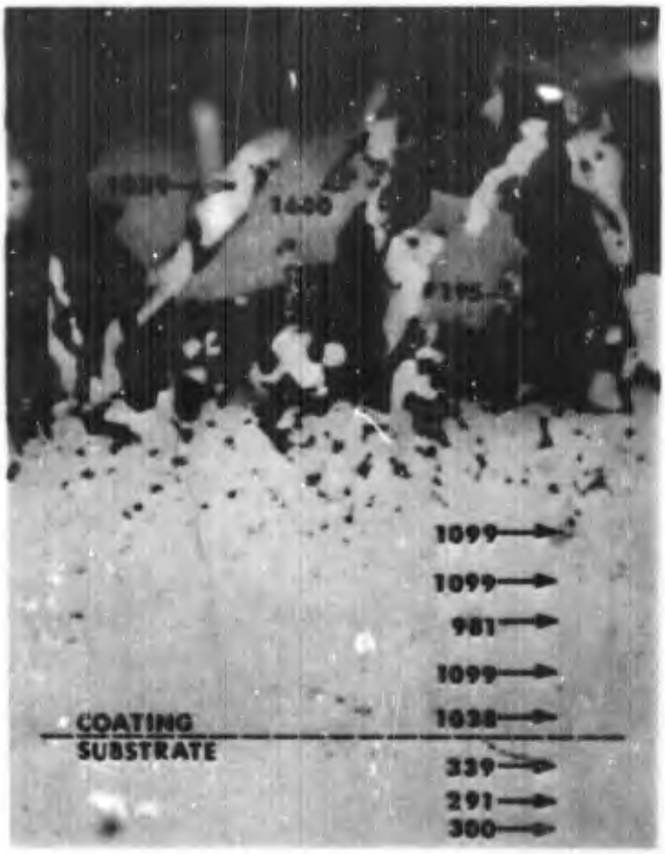
4.3.1 Remodified TRW TiCr-Si (Vacuum) Pack/Cb-132M

The Phase III evaluation of the TRW TiCr-Si/Cb-132M system for blade airfoil application has not been initiated because of the low ductility of the presently available Cb-132M alloy bar stock. Additional material is presently being ordered. The following tests are to be performed on receipt of the additional bar (see Figure 105):

- Oxidation-erosion at 2000°, 2200°, 2400°, and 2500°F
- Thermal fatigue at 2000°, 2200°, 2400°, and 2500°F

UNETCHED MAG: 500X

Figure 108. Microstructure of the As-Applied Phase III Sylcor Ag-Al-Si (508-F) Coating on Cb-132M Columbium Alloy

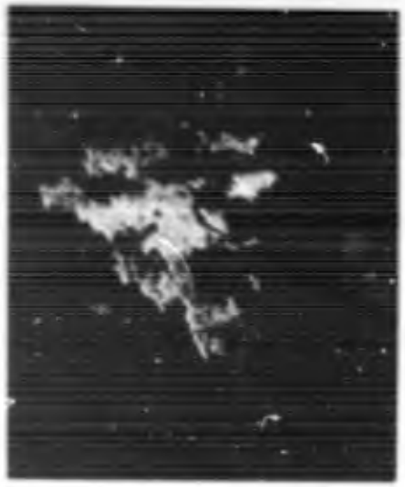


UNETCHED MAG: 500X

Cb-132M COLUMBIUM ALLOY AVERAGE HARDNESS: 267

Cb-132M COLUMBIUM ALLOY AVERAGE SUBSTRATE HARDNESS PRIOR TO COATING: 345

Figure 109. Microstructure of Sylcor Ag-Al-Si (508-F) Coated Cb-132M Columbium Alloy With Diamond Pyramid Hardness (gm/mm^2; 20-gram load) Indicated at Various Locations

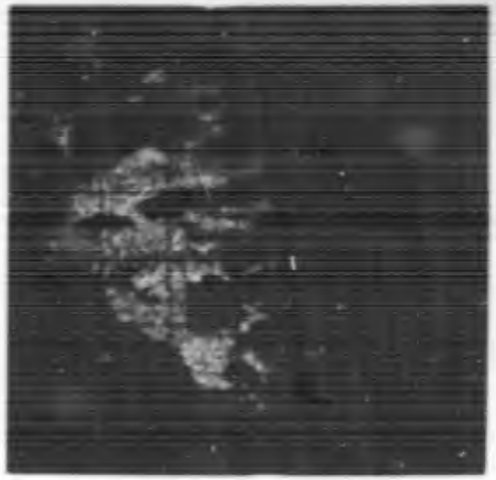


B. BACKSCATTERED ELECTRON IMAGE

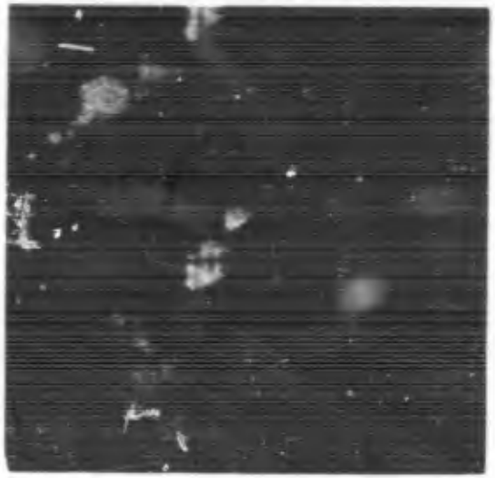
C. ALUMINUM X-RAY IMAGE

A LIGHT PHOTOMICROGRAPH

MAG (ALL PHOTOGRAPHS): 200X

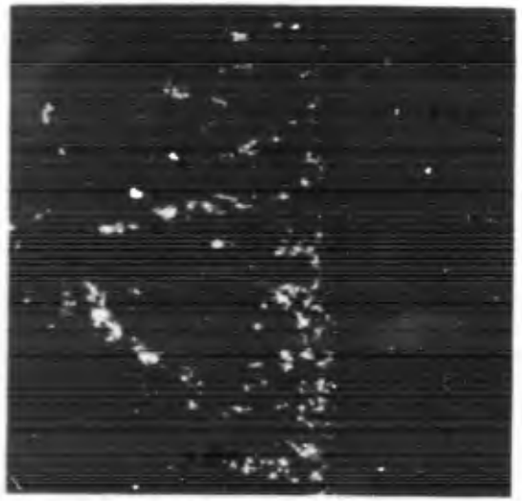


D. SILVER X-RAY IMAGE

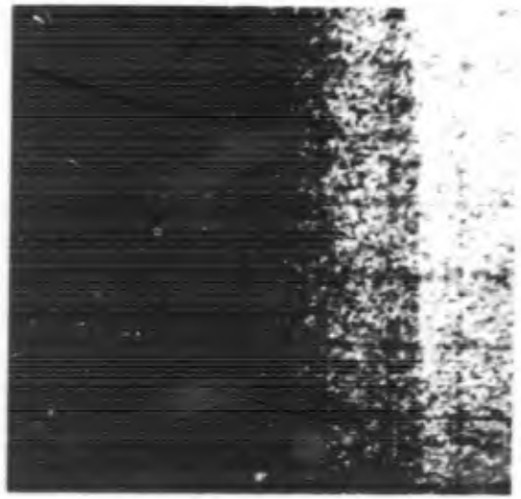


E. SILICON X-RAY IMAGE

Figure 110. Electron Microprobe X-ray Scanning Images for the Sylcor Ag-Al-Si Coating on Cb-132M Columbium Alloy

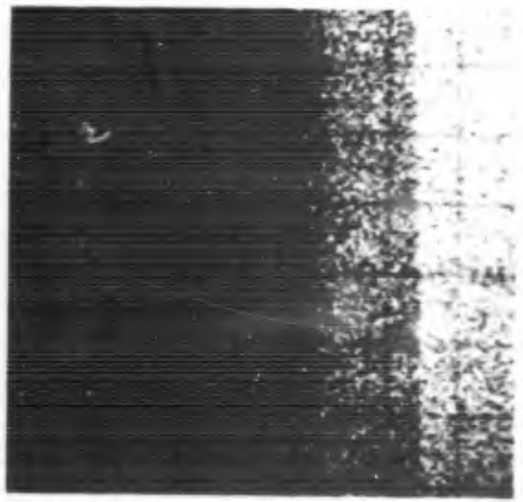


F. MOLYBDENUM X-RAY IMAGE



G. TUNGSTEN X-RAY IMAGE

H. COLUMBIUM X-RAY IMAGE



I. TANTALUM X-RAY IMAGE

Figure 110. (Concluded)

- Mechanical fatigue at 2200°F
- Creep in the 1800° to 2500°F temperature range
- Impact at 70° to 2600°F at a projectile velocity of 500 feet per second

4.3.2 TRW TiCr-Si (Vacuum) Pack/D-43

Phase III evaluation of the TRW TiCr-Si/D-43 vane system has been completed; test results are summarized in the following sections.

4.3.2.1 Oxidation-Erosion Test Results

Oxidation-erosion test bars were machined from 0.5-inch-diameter bar to the required "airfoil" geometry (Figure 21). Testing was conducted at 2000°, 2200°, 2400°, and 2500°F until failure, as constituted by the appearance of substrate oxide on the specimen airfoil surfaces. Two specimens were tested at each temperature. Testing in the rig shown in Figure 22 was performed in a combusted JP-5 fuel and air stream with an inspection period after each 20 test hours. At each temperature, dummy specimens were used for rig balancing purposes, and the bars were rotated in an 8-specimen holder at 1750 rpm to maintain temperature uniformity.

The performance of this system is illustrated in Figure 111. In summary, the results were as follows:

- 410 hours at 2000°F: Failure occurred by localized oxidation along the airfoil trailing edge surface.
- 220 hours at 2200°F: First specimen exhibited only one area of localized spalling and oxidation at the trailing edge; second specimen had several areas of localized oxidation along the airfoil trailing edge and one area on the side.
- 60 hours at 2400°F: Localized spalling and oxidation and, in one case, extensive oxidation occurred along the trailing edge surface.
- 40 hours at 2500°F; Localized spalling and oxidation occurred along the trailing edge and, in one case, along the airfoil leading edge.

In general, the surface appearance of the tested specimens was good in that areas away from the localized oxide formations did not suffer gross

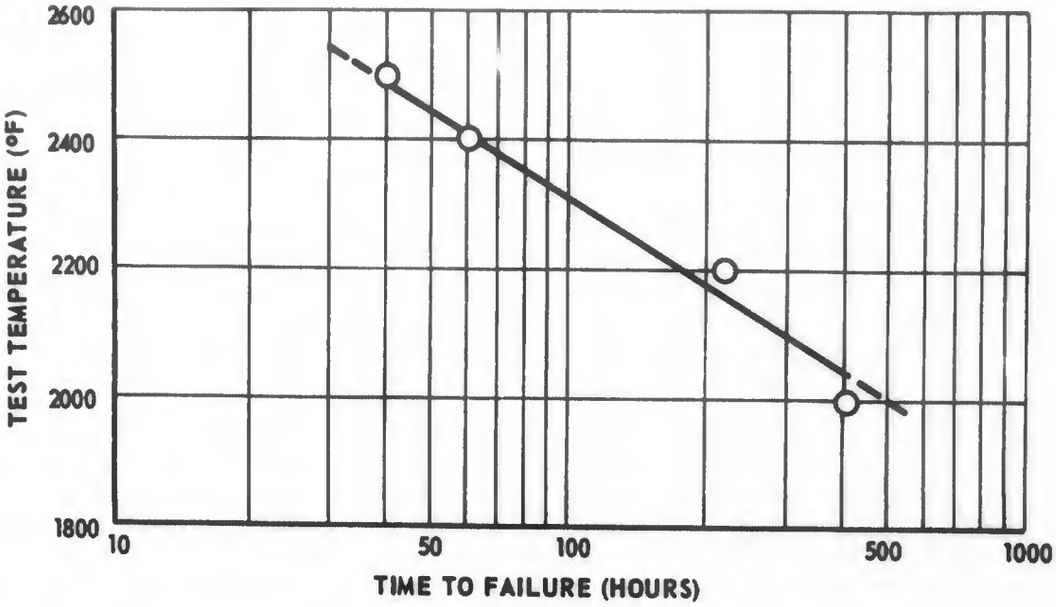


Figure 111. Oxidation-Erosion Life of TRW TiCr-Si (Vacuum) Pack Coated D-43 Columbium Alloy as a Function of Test Temperature

degradation (see Figure 112). The surface oxidation followed the general failure pattern observed for the Phase I and Phase II systems; i.e., oxide extended out of surface craze cracks (Figure 113) and eventually led to considerable oxidation of the substrate beneath the coating.

4.3.2.2 Thermal Fatigue Test Results

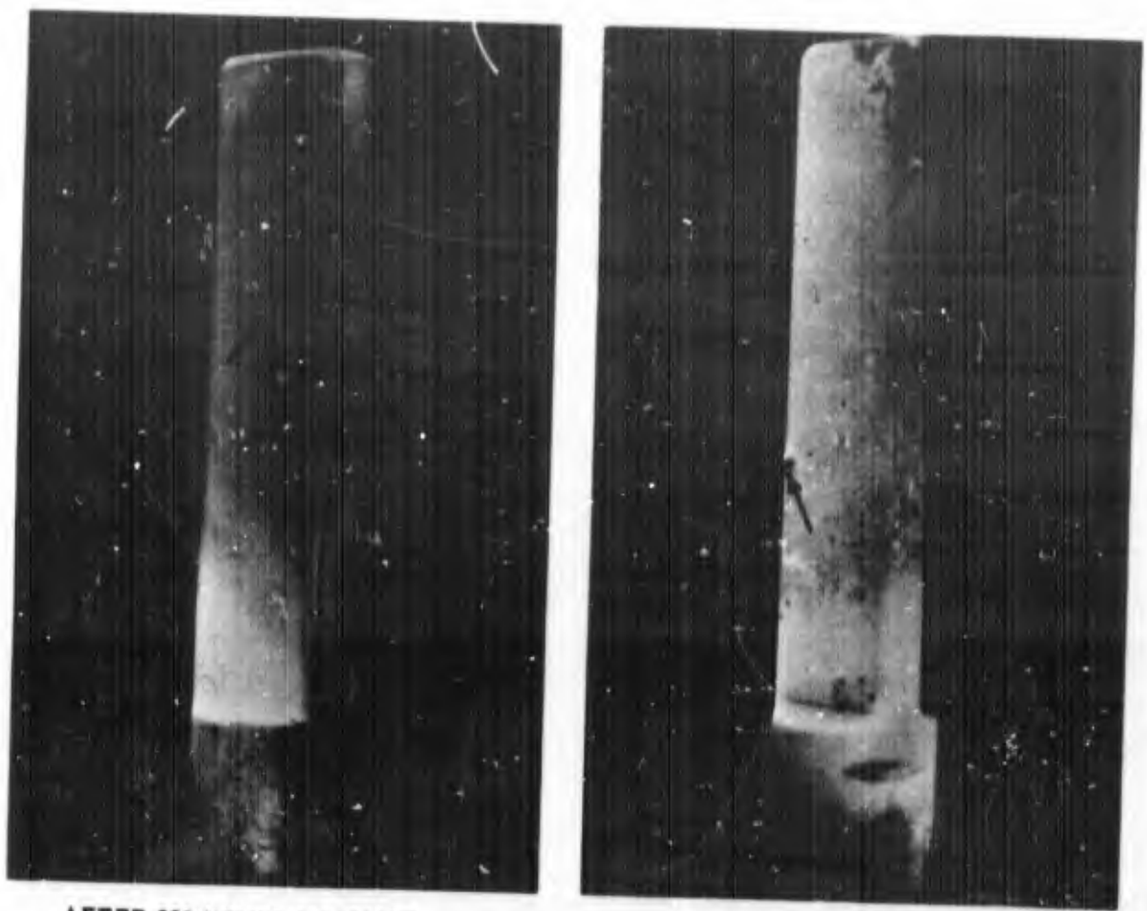
Unlike the Phase I and Phase II vane alloy systems, which utilized a welded sheet metal airfoil configuration (Figure 26), the Phase III thermal fatigue tests were performed on forged D-43 vane alloy paddles of the type shown in Figure 23. Testing at 2000°, 2200°, 2400°, and 2500°F consisted of heating to the desired peak temperature in a combusted JP-5 fuel and air stream, stabilizing at this temperature for 30 minutes, and cycling by alternate heating and cooling to approximately 200°F with an ambient temperature air blast at 60-65 psi. Each thermal cycle consisted of 1 minute at the elevated temperature followed by a 30-second cooling period. For temperature uniformity, the specimens were rotated at 1850 rpm in groups of 12 (TRW TiCr-Si/D-43 test pieces plus dummy specimens). All specimens were inspected at the end of each 200 thermal cycles, and, during continued testing, a 5-minute stabilization period at the elevated temperature was utilized prior to cycling. Two specimens were tested at each temperature. The thermal fatigue test results are summarized in Figure 114, and the following coating characteristics were observed.

- 2000°F, 1200 and 1600 cycles (total time at 2000°F: 1255 and 1665 minutes): The general coating appearance was good, but oxidation had occurred in isolated areas, mostly on convex airfoil surfaces and the trailing edge tip (Figure 115).

- 2200°F, 800 cycles (total time at 2200°F: 845 minutes): Localized spalling and oxidation occurred on the concave airfoil surfaces near the leading and/or trailing edge(s) (Figure 116).

- 2400°F, 400 and 1400 cycles (total time at 2400°F: 435 and 1460 minutes): After 400 cycles, the first specimen developed two small areas of oxidation at the tip of the airfoil. The second specimen (1400 cycles) exhibited localized oxidation along the airfoil trailing edge (Figure 117).

- 2500°F, 1660 and 2200 cycles (total time at 2500°F: 1665 and 2280 minutes): The first specimen developed a small oxide spot at the trailing edge tip and two areas of localized oxidation on the concave surface at the airfoil trailing edge after 1600 cycles. The remaining specimen had a small area of localized oxidation on the convex surface near the trailing edge tip.

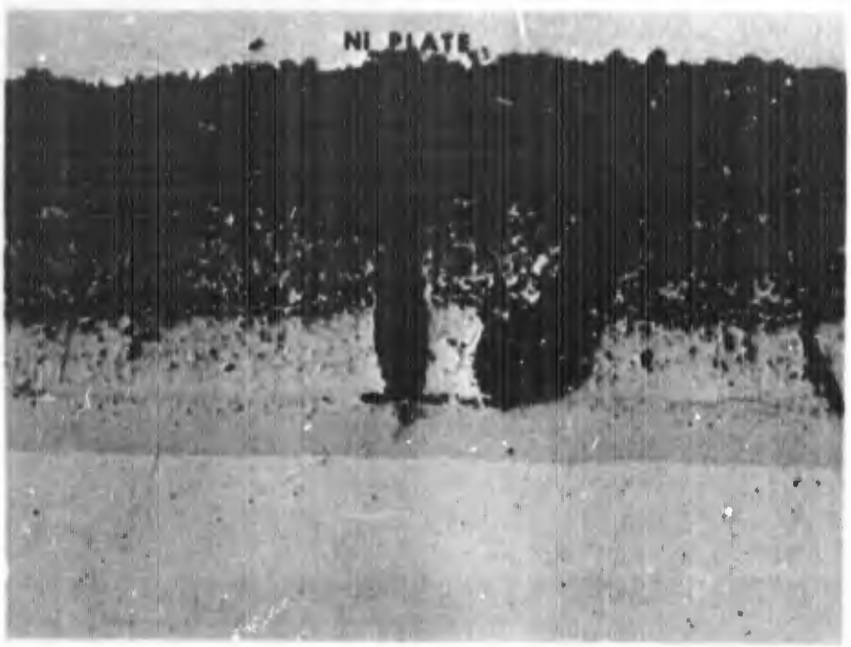


AFTER 220 HOURS AT 2200°F

AFTER 40 HOURS AT 2500°F

MAG: APPROX. 1.5X

Figure 112. Typical Localized Oxidation-Erosion Failures (arrows) on TRW TiCr-Si (Vacuum) Pack Coated D-43 Columbium Alloy



ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER MAG: 500X

Figure 113. Microstructure of the TRW TiCr-Si Coating on D-43 Columbium Alloy After Oxidation-Erosion Testing for 60 Hours at 2400°F

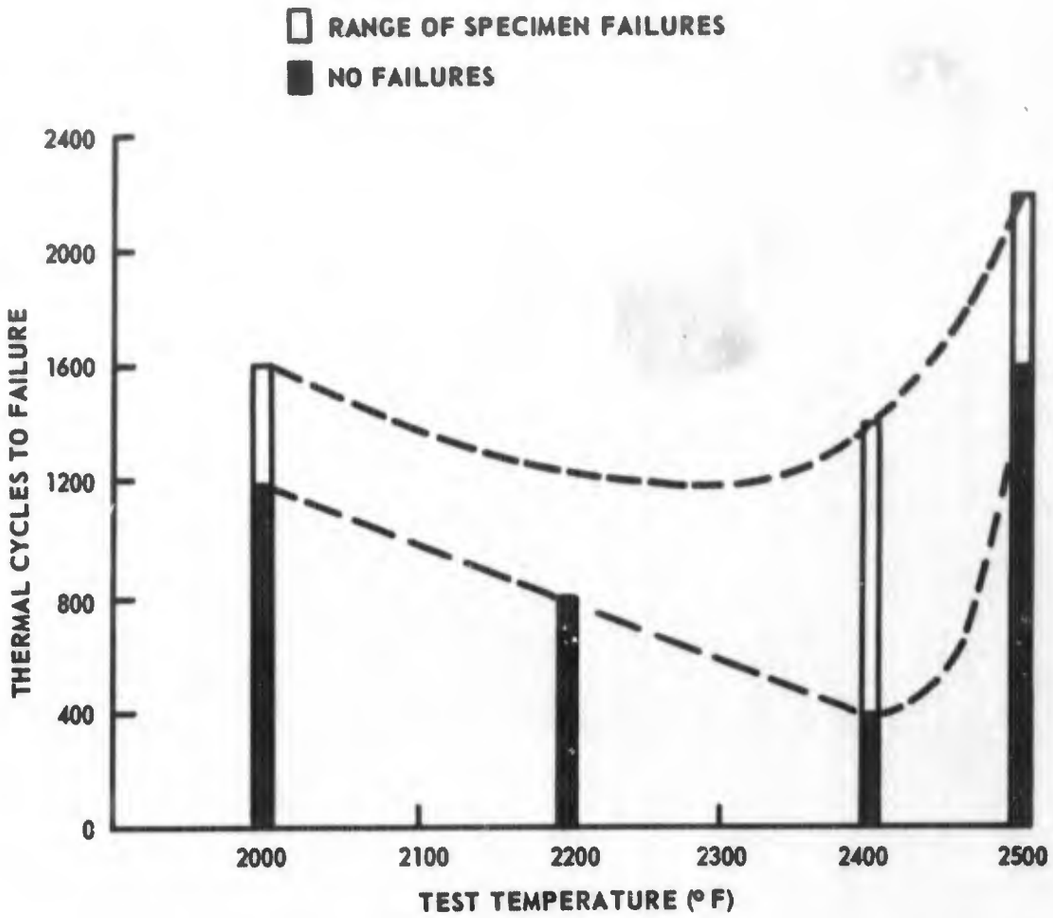


Figure 114. Thermal Fatigue Life of TRW TiCr-Si (Vacuum) Pack Coated D-43 Columbium Alloy as a Function of Test Temperature

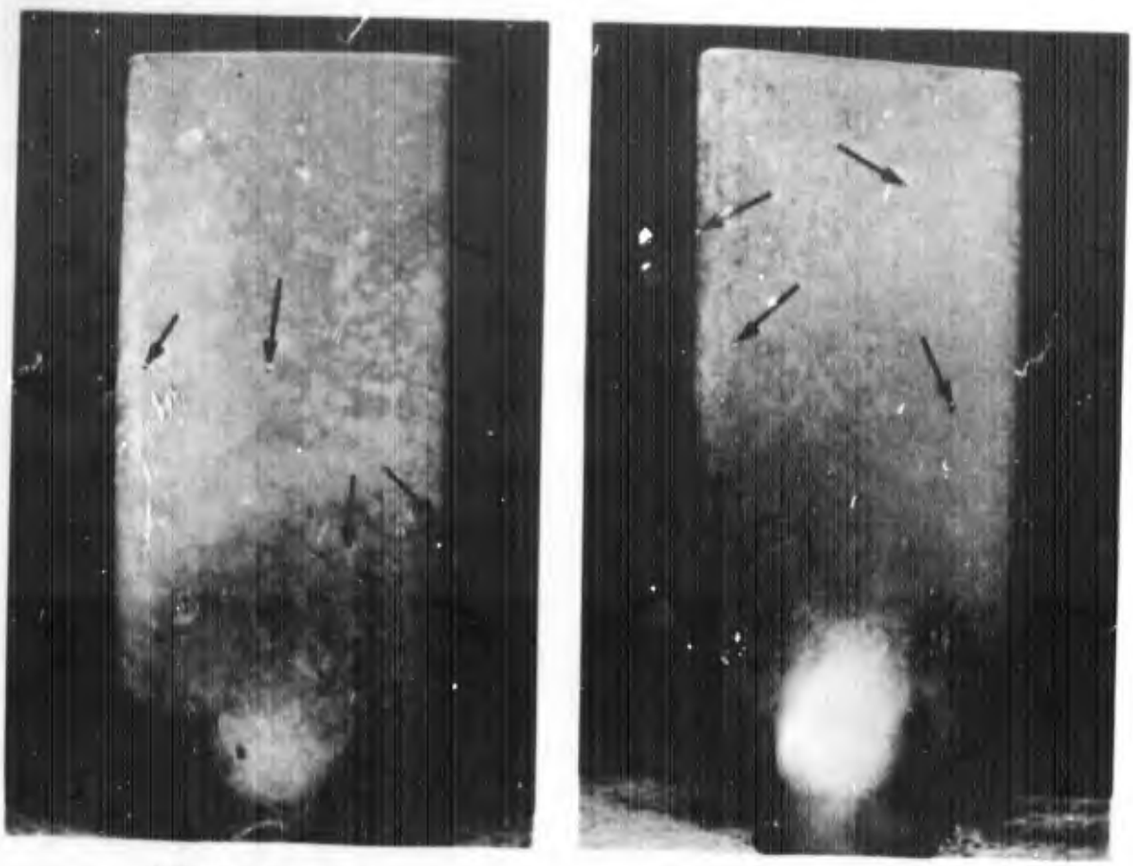
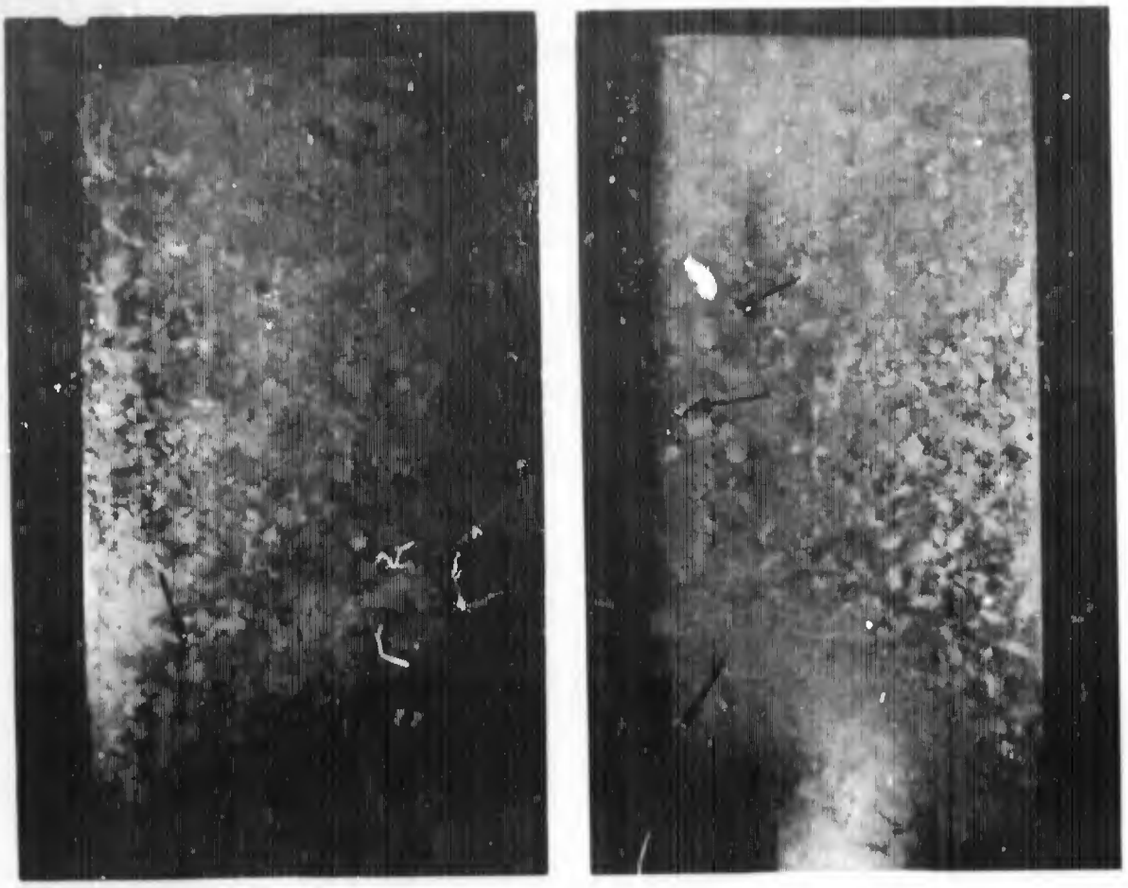


Figure 115. TRW TiCr-Si (Vacuum) Pack Coated D-43 Columbium Alloy Thermal Fatigue Specimen After 1600 Cycles at 2000°F (arrows locate areas of localized oxidation)

CONCAVE SURFACE

MAG: 2X

Figure 116. TRW TiCr-Si (Vacuum) Pack Coated D-43 Columbium Alloy Thermal Fatigue Specimen After 800 Cycles at 2200°F (arrows locate areas of localized oxidation)



CONVEX SURFACE

CONCAVE SURFACE

MAG: 2X

Figure 117. TRW TiCr-Si (Vacuum) Pack Coated D-43 Columbium Alloy Thermal Fatigue Specimen After 1400 Cycles at 2400°F (arrows locate areas of localized oxidation)

In the latter stages of testing, particularly at the 2400° and 2500°F temperatures, the coating developed a mottled surface appearance (Figure 117). However, no failures were observed to occur preferentially in any particular coating area.

The excellent thermal fatigue performance at 2500°F compared to that at 2000°, 2200°, and 2400°F suggests that at 2500°F the TRW TiCr-Si coating thermal fatigue life is relatively unaffected by thermal cycling if the transient time period between the low and high temperatures is small. The total time at 2500°F (38 hours) during one thermal fatigue test compares favorably with the 2500°F oxidation-erosion life of 40 hours. The convergence of the oxidation-erosion and thermal fatigue lives is illustrated in Figure 118. The above behavior may result from a combination of better coating self-healing properties, higher coating ductility, and a more favorable coating-substrate thermal expansion relationship at the higher testing temperature.

4.3.2.3 Melting Test Results

To determine the effects of occasional transient temperature overshoots, a cyclic oxidation-erosion test was performed. Each cycle consisted of the following:

- 10 hours at 2200°F, followed by
- 5 minutes at 2600°F, followed by
- 10 hours at 2200°F

The test specimens were again rotated at 1750 rpm for temperature uniformity in a combusted JP-5 fuel and air stream. The specimens were inspected at the end of each cycle.

Localized oxidation occurred at the leading edge tip of one specimen after two cycles, but the two remaining specimens exhibited a performance life of seven 2200°-2600°F test cycles (Figure 119). Failure of all the remaining specimens occurred as a result of localized oxidation on the airfoil trailing edge surface (Figure 120).

Comparison of the cyclic test results with a 2200°F oxidation-erosion performance revealed that the 5-minute period at 2600°F during each 20 hours of 2200°F testing decreased the average time to failure from 220 to 107 hours, a reduction of approximately 51 percent.

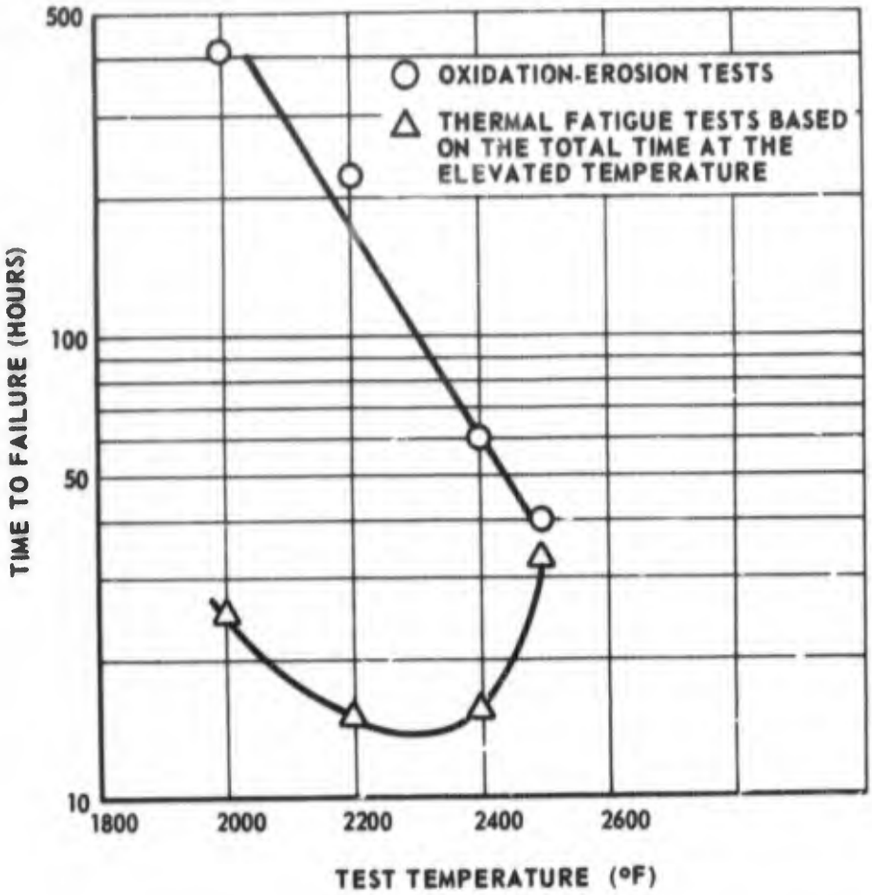


Figure 118. Comparison of the Oxidation-Erosion and Thermal Fatigue Lives of TRW TiCr-Si (Vacuum) Pack Coated D-43 Columbium Alloy

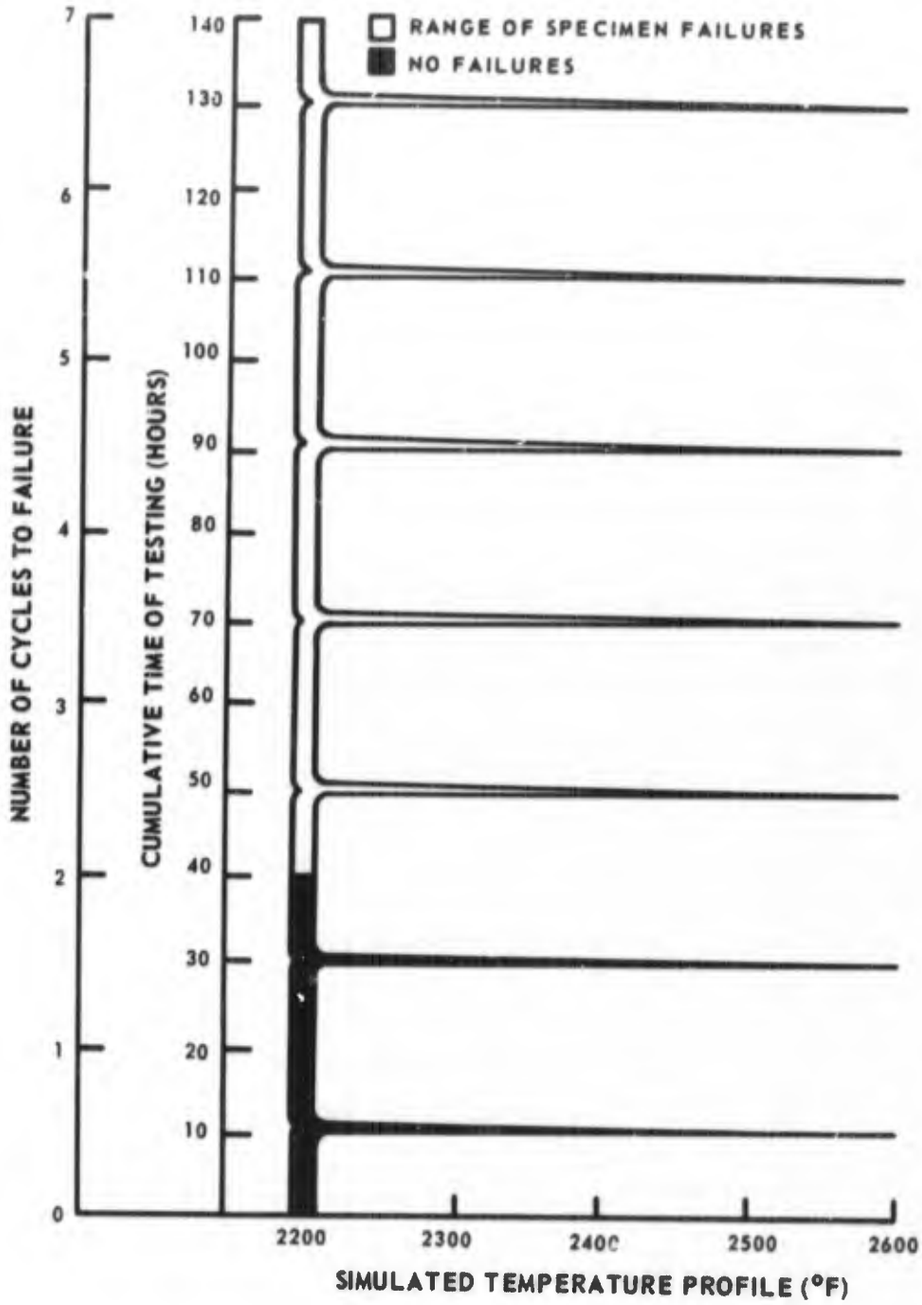
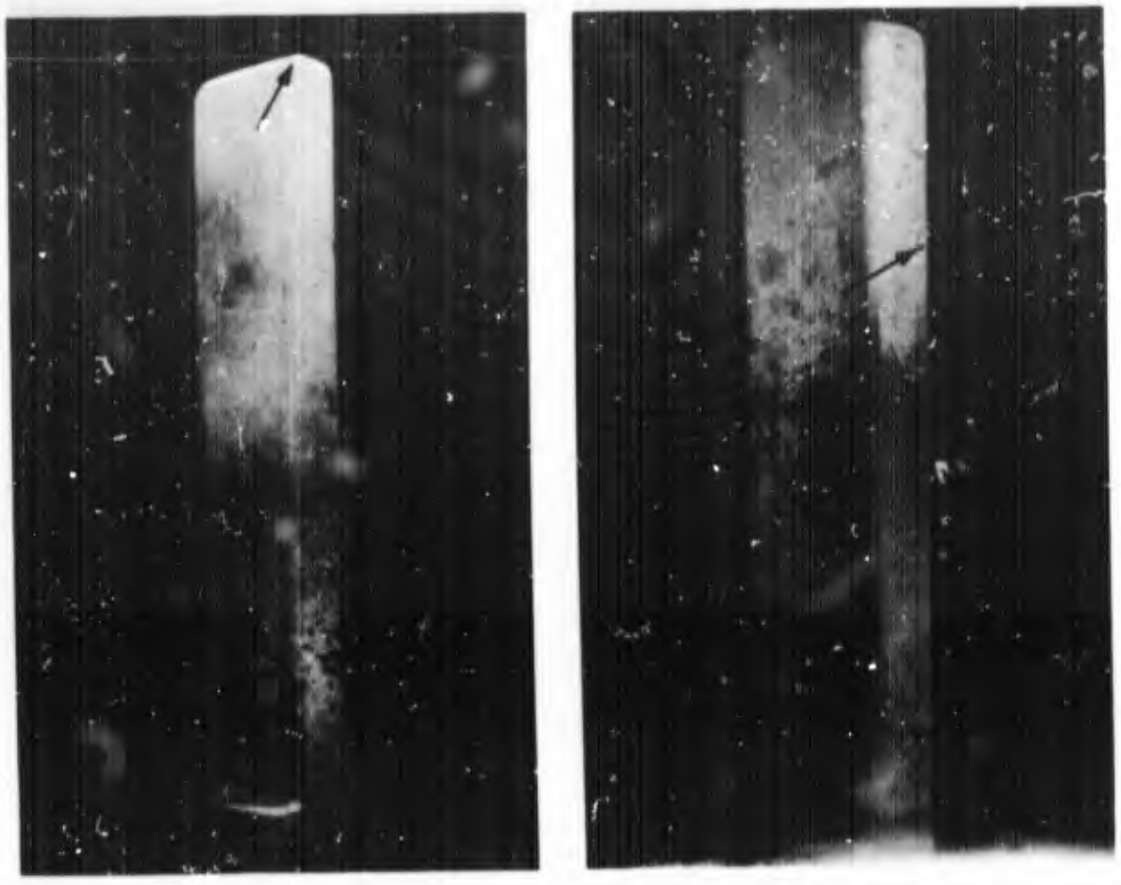


Figure 119. Cyclic Oxidation-Erosion Life of TRW TiCr-Si (Vacuum) Pack Coated D-43 Columbium Alloy



AFTER 2 CYCLES

MAG: 1.8X

AFTER 7 CYCLES

MAG: 2X

Figure 120. Oxidation-Erosion Specimens of TRW TiCr-Si (Vacuum) Pack Coated D-43 Columbium Alloy After Cyclic Testing for the Indicated Number of Cycles (arrows locate areas of localized oxidation)

4.3.2.4 Ballistic Impact Test Results

Rectangular 0.050-inch-thick specimens were impacted at a velocity of 500 feet per second with a 0.75-gram steel pellet after a 5-minute stabilization at 70°, 1600°, 2200°, 2400°, and 2600°F. In all cases, the impact point was located 0.5 inch above the secured portion of the specimen to avoid any differences in specimen bending moment. Two specimens per system were tested at each temperature, and each was impacted at two locations 1 inch apart.

Appearance of the 70°, 1600°, and 2200°F impact areas was characterized by the condition of the outer silicide layers. Five zones were defined as detailed previously in paragraph 2.3.2.3 (Phase I Ballistic Impact Results) and illustrated in Figure 57.

The 2400°F impacts exhibited very small scab areas (Zones II and V), while no scab areas were observed on the specimens impacted at 2600°F.

After impacting and subsequent inspection, all specimens were subjected to static oxidation exposure at 2200°F. The first appearance of substrate oxide constituted failure. Oxidation test results are presented in Table XI. The protective life at 2200°F as a function of impact temperature is presented in Figure 121 along with illustrations of typical as-impacted surfaces at 70°, 2200°, and 2600°F. The surface and microstructural appearance of a typical failure is shown in Figure 122.

The relationship between temperature at impact and subsequent protective life at 2200°F (Figure 121) indicates a transition between 2200° and 2600°F. In all cases, the average protective life increased at least slightly as the impact temperature increased. However, above 2200°F these increases were significant. This behavior was probably due to two factors:

- At temperatures above 2200°F, the ductility of the coating and the coating-substrate composite increases significantly.
- At lower temperatures, the higher yield strength of the substrate results in a greater reflected elastic shock, thereby leading to a larger scab area (Zone II) surrounding the impact depression. As mentioned previously, all failures occurred within this area.

4.3.2.5 Stress-Rupture Test Results

Stress-rupture testing of the TRW TiCr-Si/D-43 system was performed at 2200° and 2500°F by New England Materials Laboratory. The test specimen configuration is shown in Figure 123. A resistance-heated furnace was employed for this testing and was stabilized at the desired

TABLE XI

RESULTS OF PHASE III BALLISTIC IMPACT TESTING OF THE TRW TiCr-Si (VACUUM) PACK COATED D-43 COLUMBIUM ALLOY

Test No.	Specimen No.	Specimen Temp. at Impact (°F)	Impact Velocity (ft/sec)	Oxidation Life After Impact		Observed Condition After Oxidation Exposure	
				Temp. (°F)	Time (hrs.)	Surface at Impact Point	Surface Directly Behind Impact Point
1	DBI-3-1	70	500	2200	1	Oxide surrounding impact depression in all cases; after impact, extensive scabbing around impact depression	No failures
2	DBI-3-1	70	500	2200	1		
3	DBI-3-2	70	500	2200	1		
4	DBI-3-2	70	500	2200	1		
5	DBI-3-3	1600	500	2200	2	Oxide beneath an adherent brown skin surrounding the impact depression; point breakthroughs of oxide to surface. In all cases the outer coating layers were removed from around the impact depression after impact.	No failures
6	DBI-3-3	1600	500	2200	2		
7	DBI-3-4	1600	500	2200	2		
8	DBI-3-4	1600	500	2200	3		
9	DBI-3-5	2200	500	2200	2	Oxide beneath an adherent brown skin surrounding the impact depression; point breakthroughs of oxide to surface. In all cases the outer coating layers were removed from around the impact depression after impact.	No failures
10	DBI-3-5	2200	500	2200	3		
11	DBI-3-6	2200	500	2200	2		
12	DBI-3-6	2200	500	2200	3		
13	DBI-3-7	2400	500	2200	3	Oxide beneath an adherent brown skin surrounding the impact depression; point breakthroughs of oxide to surface. In some cases the outer coating layers were removed from around the impact depression after impact.	No failures
14	DBI-3-7	2400	500	2200	5		
15	DBI-3-8	2400	500	2200	3		
16	DBI-3-8	2400	500	2200	5		
17	DBI-3-9	2600	500	2200	7	Oxide beneath an adherent brown skin surrounding the impact depression; point breakthroughs of oxide to surface. The outer coating layers were not removed from around the impact depression after impact.	No failures
18	DBI-3-9	2600	500	2200	5		
19	DBI-3-10	2600	500	2200	6		
20	DBI-3-10	2600	500	2200	6		

Notes:

a. Weight of steel projectile: 0.75 gram each
b. Specimen thickness before coating: 0.050 inch

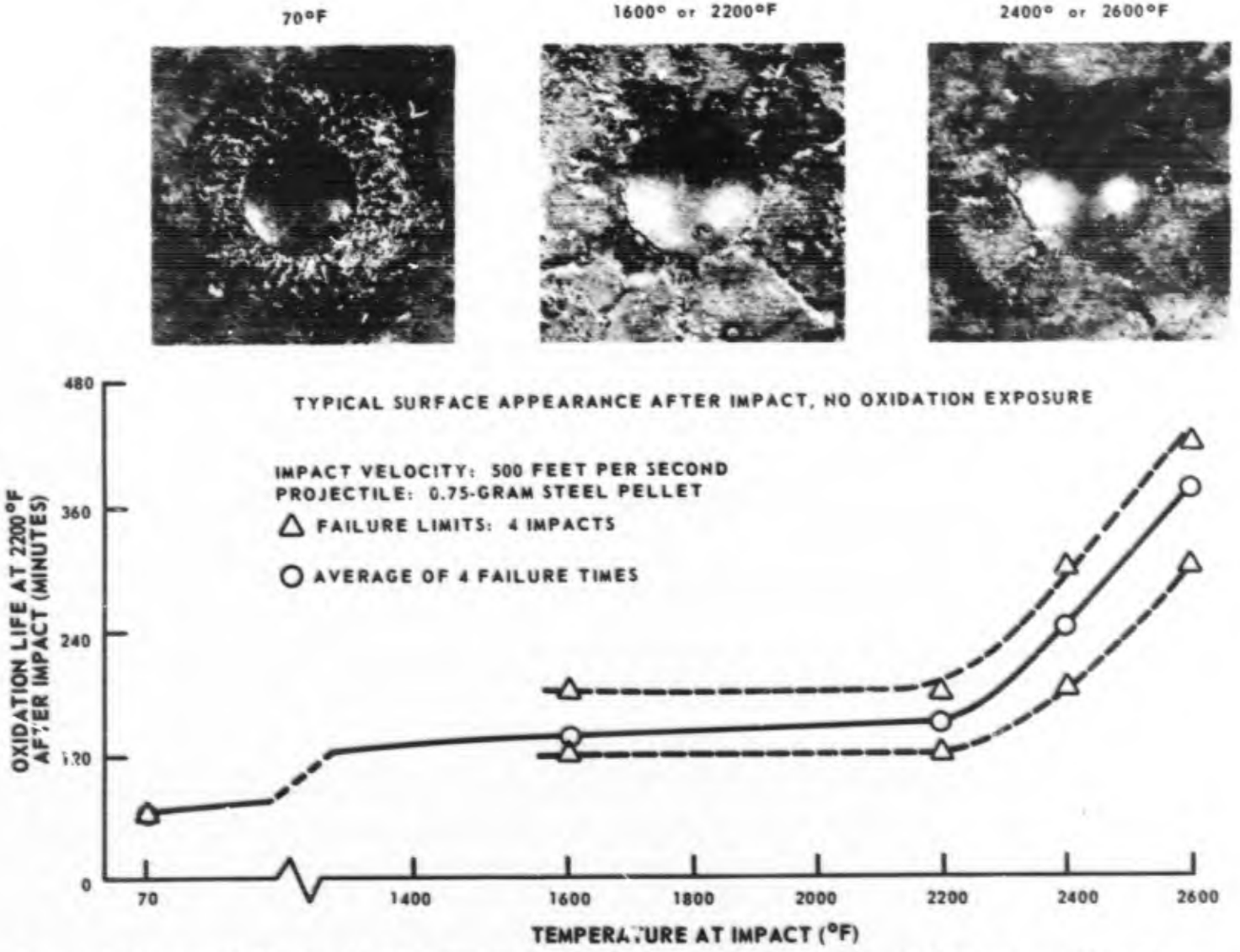
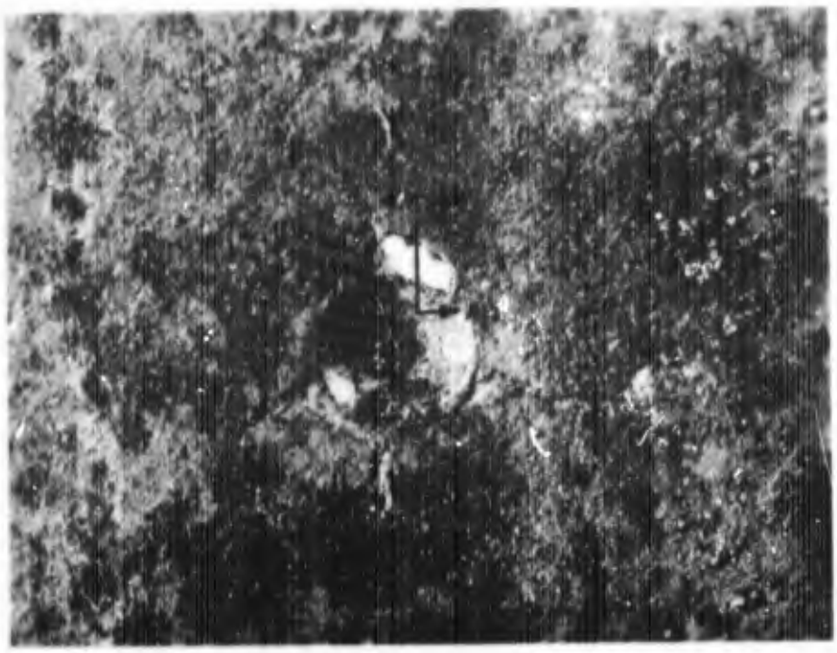
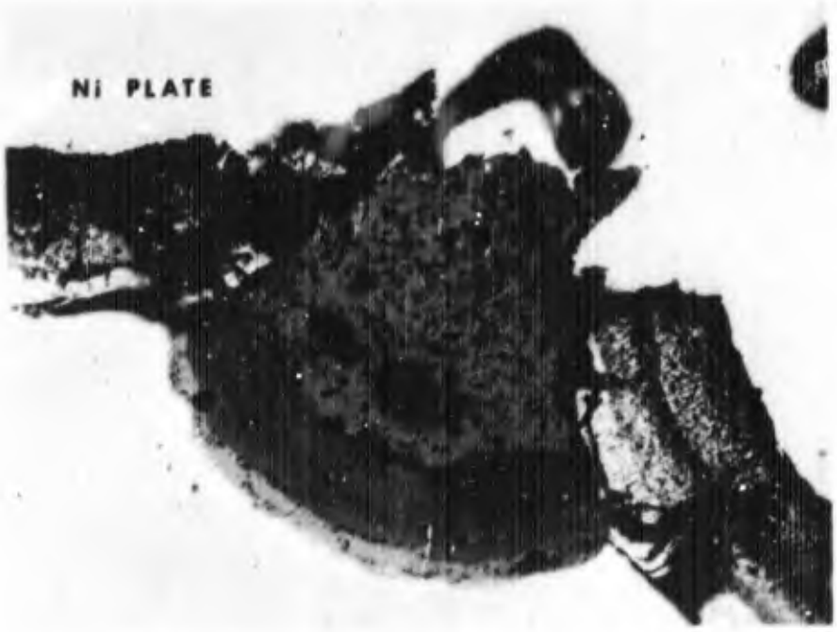


Figure 121. The Oxidation Life of TRW TiCr-Si (Vacuum) Pack Coated D-43 Columbium Alloy After 500 Feet Per Second Ballistic Impact at 70°-2600°F



SURFACE APPEARANCE

MAG: 10X



MICROSTRUCTURE OF FAILURE AT SECTION A-A

UNETCHED

MAG: 250X

Figure 122. Surface and Microstructural Appearance of a Typical 2200°F Static Oxidation Failure of TRW TiCr-Si (Vacuum) Pack Coated D-43 Columbium Alloy After 2600°F Ballistic Impact.

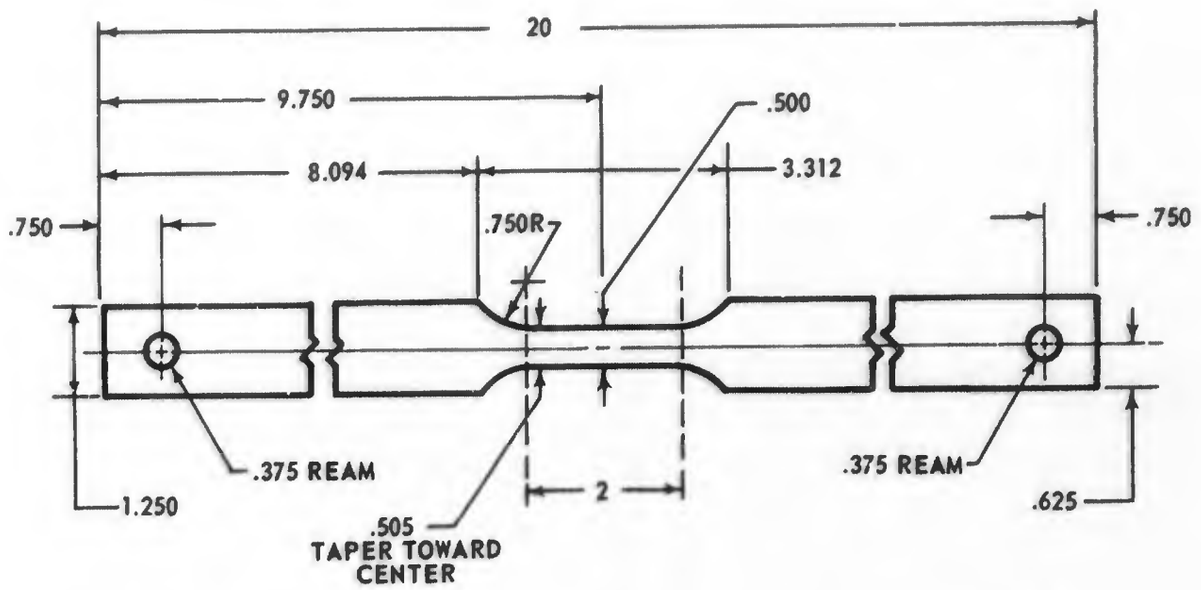


Figure 123. Sheet Stress-Rupture Specimen Configuration

temperature prior to the insertion of each specimen. After stabilization, each specimen was placed in the test chamber and the temperature again stabilized. The total stabilization period was generally about 30 minutes. Two sheathed chromel/alumel thermocouples were used for continuous temperature monitoring at 2200°F and two sheathed platinum/platinum-rhodium couples were utilized at 2500°F.

Results of the stress-rupture testing at 2200° and 2500°F are summarized in Table XII and Figure 124. No anomalies were observed at either testing temperature in the time to rupture or in the deformation and fracture behavior characteristics of the specimens, and the data are in good agreement with published information (Ref. 31). The appearance of typical gage sections after failure at 2200° and 2500°F is shown in Figure 125. Fracture surfaces were destroyed by specimen oxidation after failure. Metallographic examination of specimen gage sections did not reveal cavitation or distinct indications of void formation. In cases of considerable elongation prior to failure, multiple separation of the coating was observed (Figure 125, upper photograph).

4.3.3 Sylcor Ag-Al-Si (508-F)/Cb-132M

Phase III evaluation of the Sylcor Ag-Al-Si/Cb-132M system for blade root application involved the following tests:

- Oxidation exposure at 1300° and 1600°F to determine coating protection, stability, and reliability.
- Melting tests at 2000°F, a test to ascertain the effects of occasional transient exposures at temperatures above the anticipated operating range (1300°-1600°F).
- Duplex coating compatibility for cases where the blade airfoil coating differs compositionally from the blade root coating.
- Mechanical fatigue testing to assure composite capability under blade root loading.

Static oxidation, melting, and duplex coating compatibility tests have been completed; the results are summarized in the following sections. Fatigue tests have not been conducted because of the low ductility of the Cb-132M alloy bar presently on hand. These tests will be performed on additional material presently being procured.

TABLE XII

PHASE III 2200° AND 2500°F STRESS-RUPTURE PROPERTIES OF TRW TiCr-Si (VACUUM) PACK COATED D-43 COLUMBIUM ALLOY

Test No.	Test Temperature (°F)	Stress (psi)	Rupture Life (hours)	Elongation (percent)
1	2200	18,200	8.1	55.2
2	2200	15,500	16.8	74.3
3	2200	14,100	41.7	36.8
4	2200	12,400	68.8	23.4
5	2200	11,300	148.8	26.7
6	2500	10,400	6.0	55.5
7	2500	8,300	14.1	47.2
8	2500	6,700	32.0	25.6
9	2500	5,600	118.6	39.1

Note: Stress based on cross-sectional area before coating.

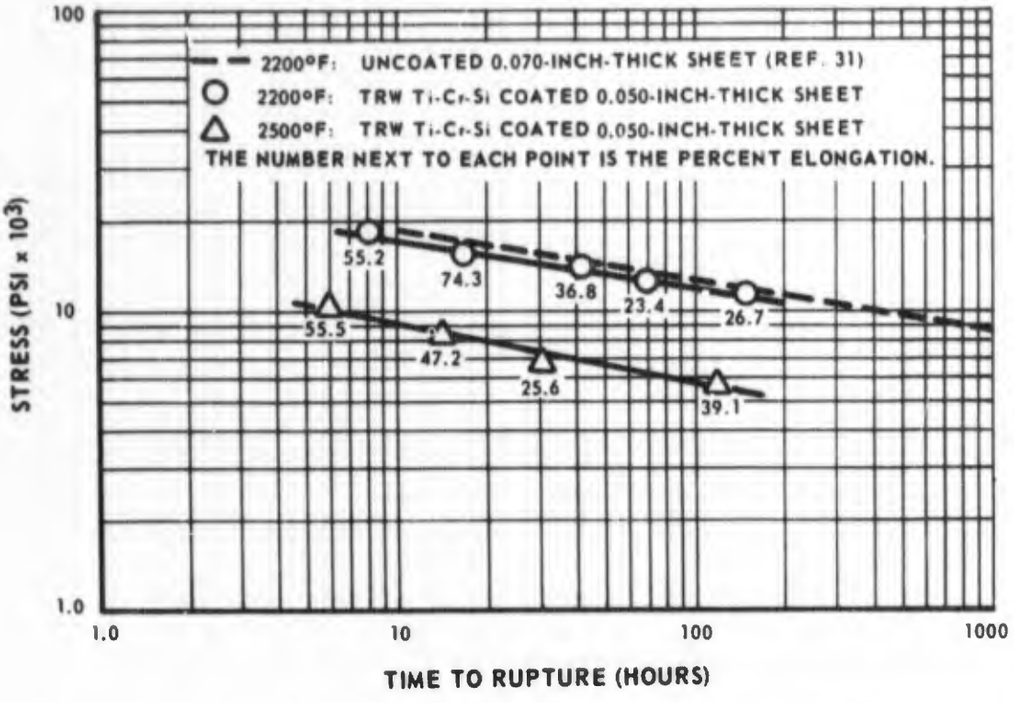
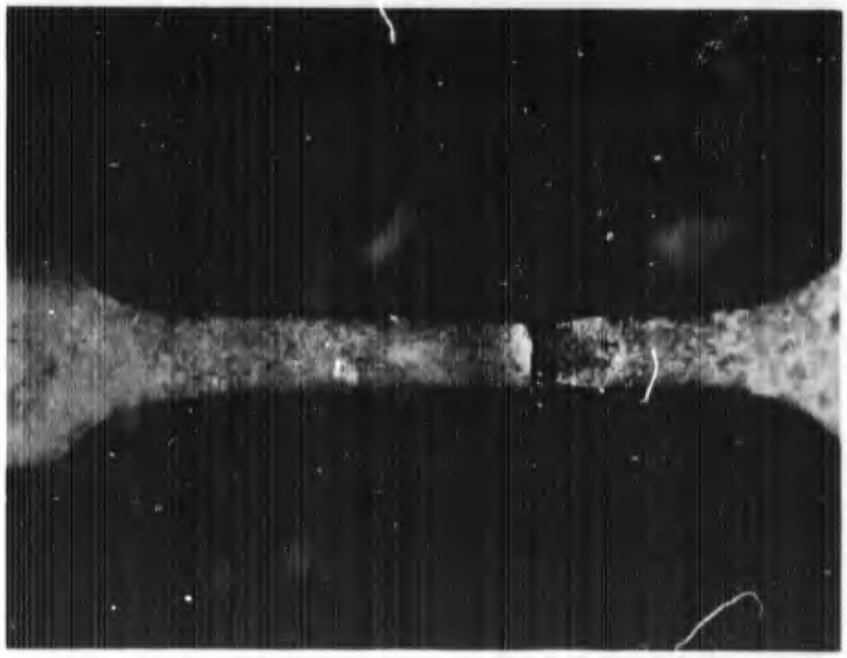


Figure 124. Stress-Rupture Properties of TRW TiCr-Si (Vacuum) Pack Coated D-43 Columbium Alloy at 2200°F and 2500°F

TEMPERATURE: 2200°F
STRESS: 15,510 PSI

TIME TO RUPTURE: 16.8 HOURS
TOTAL ELONGATION: 74.3%
MAG: 1X



TEMPERATURE: 2500°F
STRESS: 10,430 PSI

TIME TO RUPTURE: 6.0 HOURS
TOTAL ELONGATION: 55.5%
MAG: 1X

Figure 125. Typical Failed Stress-Rupture Specimens of TRW TiCr-Si (Vacuum) Pack Coated D-43 Columbium Alloy Sheet

4.3.3.1 Oxidation Test Results

Oxidation testing was performed at 1300° and 1600°F in still air using 0.125x0.5x2-inch coupons. During the first 24 hours of testing, silver beads formed on the coated surfaces (Figure 126), but gradually disappeared during continued exposure.

Failures were observed after 48, 120, 144, 144, and 168 hours for the five specimens tested at 1300°F. In all cases, the coating on flat specimen surfaces remained intact. Failures occurred by coating separation along edges and corners which led to eventual substrate oxidation in these areas (Figure 127). At 1600°F, the failure times for the five test specimens were 120, 474, 498, 600, and 630 hours. Except for the 120-hour failure, all specimens exhibited coating separation at edges and corners after 190 to 250 hours. However, no substrate oxide was observed in the separated areas until those failure times reported above. During continued exposure at 1300° or 1600°F after the first sign of substrate oxidation, either additional failures continued to occur along edge surfaces (Figure 128) or the coating separated and peeled away from the substrate (Figure 129).

4.3.3.2 Melting Test Results

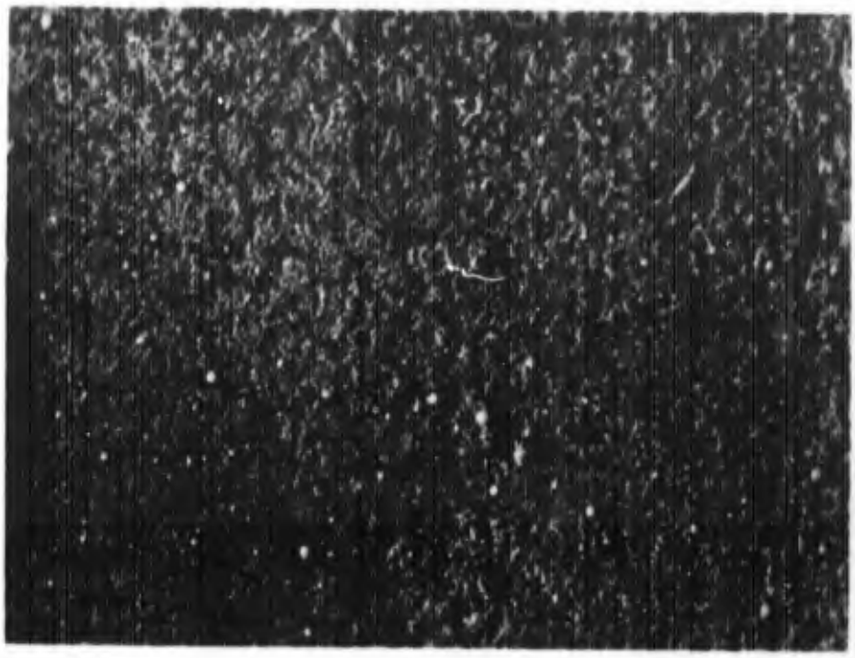
Coupons similar to those described above were cycled from 1300° and 1600°F to 2000°F. Cycling was performed by transferring between furnaces stabilized at the appropriate temperatures. Transfer time was approximately 1 minute. After 3 cycles, each consisting of 1 hour at 1300° or 1600°F followed by 1 hour at 2000°F, the specimens were exposed in still air at the lower temperature until failure.

For the 1300°-2000°F tests, no failures were observed after cycling; however, all four test coupons failed during subsequent 1300°F oxidation exposure after 73, 87, 127, and 127 hours by edge and/or corner separation of the coating and subsequent substrate oxidation.

The 1600°-2000°F cycled specimens exhibited edge and corner coating separation after cycling and 73 to 127 hours at 1600°F. However, failure of the four specimens by visible substrate oxidation did not occur until 355, 355, 409, and 802 hours.

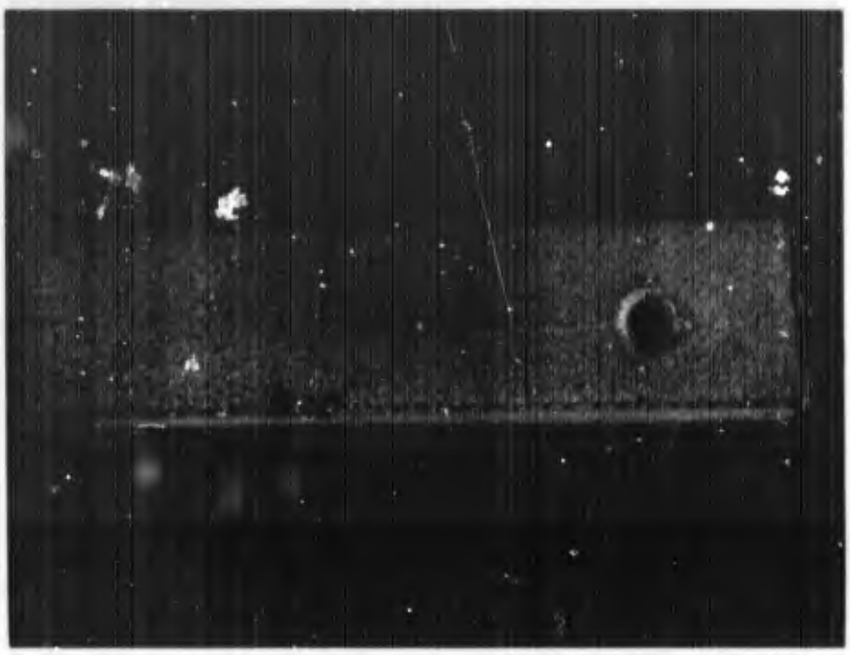
4.3.3.3 Coating Compatibility Test Results

Compatibility testing of the Sylcor 508-F Ag-Al-Si and the TRW TiCr-Si (vacuum) pack coatings was conducted using four 0.5-inch-diameter by 3.25-inch-long specimens. On each specimen, the Ag-Al-Si coating overlapped the TiCr-Si coating for a 0.5-inch length along the bar at each of



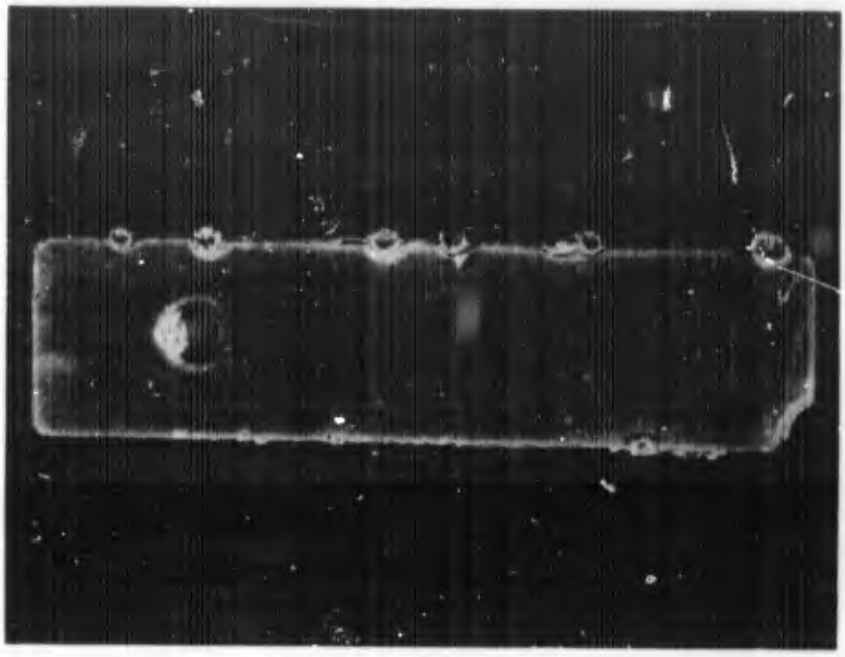
MAG: 10X

Figure 126. Surface of Sylcor Ag-Al-Si Coated Cb-132M Columbium Alloy After 24-Hour Oxidation Exposure at 1300°F Showing Silver Beads (arrows)



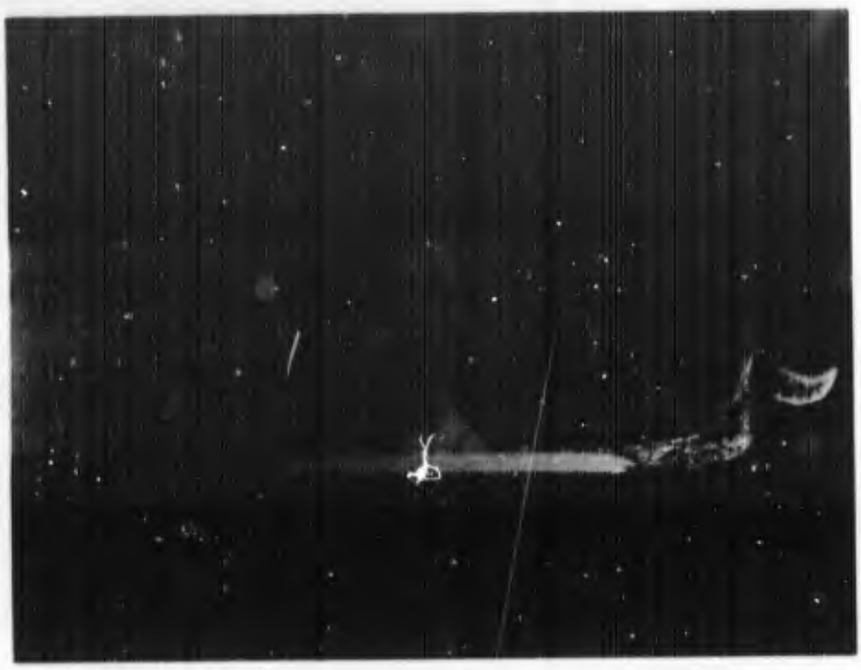
MAG: 2X

Figure 127. Typical Sylcor Ag-Al-Si Coated Cb-132M Columbium Alloy Coupon After 1300°-1600°F Oxidation Exposure Showing Edge Separation and Oxidation Failure (arrow)



MAG: 2X

Figure 128. Typical Sylcor Ag-Al-Si Coated Cb-132M Columbium Alloy Coupon After 1300°-1600°F Oxidation Exposure Showing Edge Failures in Areas of Prior Coating Separation



MAG: 2X

Figure 129. Typical Sylcor Ag-Al-Si Coated Cb-132M Columbium Alloy Coupon After 1300°F-1600°F Oxidation Exposure Showing Separation and Peeling in Area of Prior Substrate Oxidation

two locations (Figure 130). One test specimen (two overlap areas) was evaluated under the four test conditions described previously for the oxidation and melting tests. The test conditions are repeated below:

- 1300°F oxidation exposure for 120 hours maximum
- 1600°F oxidation exposure for 120 hours maximum
- 1300°-2000°F cycling followed by 1300°F oxidation exposure to failure
- 1600°-2000°F cycling followed by 1600°F oxidation exposure to failure

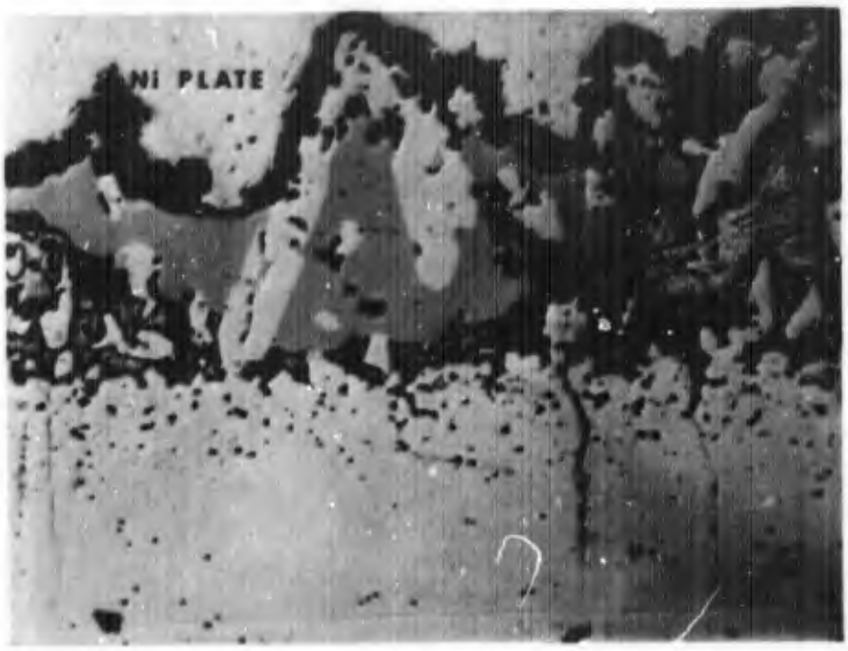
No failures were noted after 120 hours at 1300° or 1600°F. Metallographic examination of the microstructure of each coating and the overlap areas revealed the following:

- The microstructure of the Ag-Al-Si (508-F) coating away from the overlap areas was normal (Figure 131).
- Alloying of the TiCr-Si and Ag-Al-Si coatings occurred within the overlap regions. The outer (Ti,Cr)-silicide layers appeared "solutioned" by this interaction (Figure 132).
- The microstructure of the TiCr-Si coating in areas away from the overlaps was not normal, probably because of contamination during the 508-F coating application process and subsequent diffusion during oxidation exposure (Figure 133).

Cyclic compatibility testing resulted in failures (after 79 hours at 1300°F and 127 hours at 1600°F) which appeared as ruptures within the overlap regions or in the TiCr-Si coating immediately adjacent to the overlap (Figure 134). Although no substrate oxide was visible on the surfaces, metallographic examination of the failed areas revealed substrate oxidation (Figure 135).

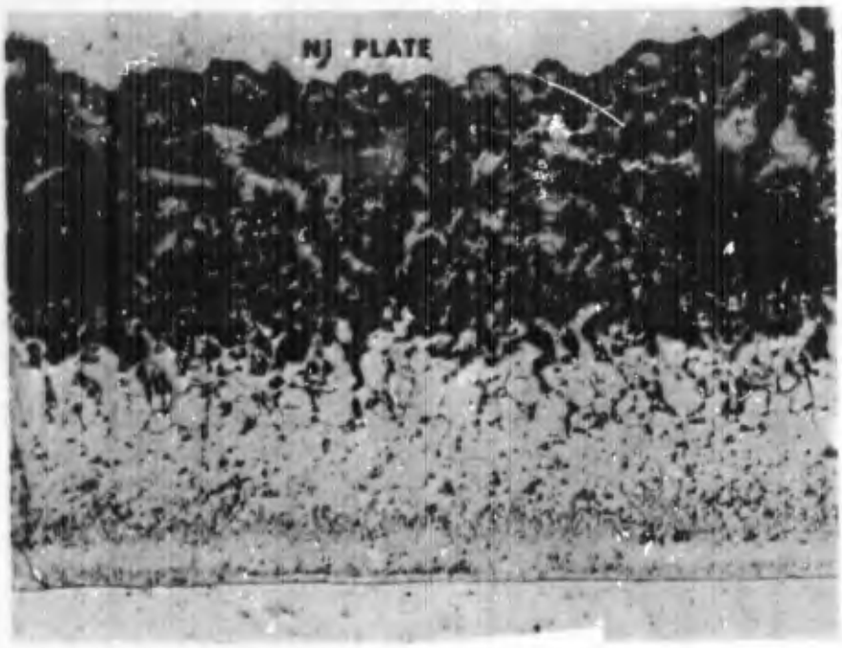
MAG: APPROX. 3X

Figure 130. Sylcor Ag-Al-Si and TRW TiCr-Si Coated Cb-132M Columbium Alloy Compatibility Test Specimen



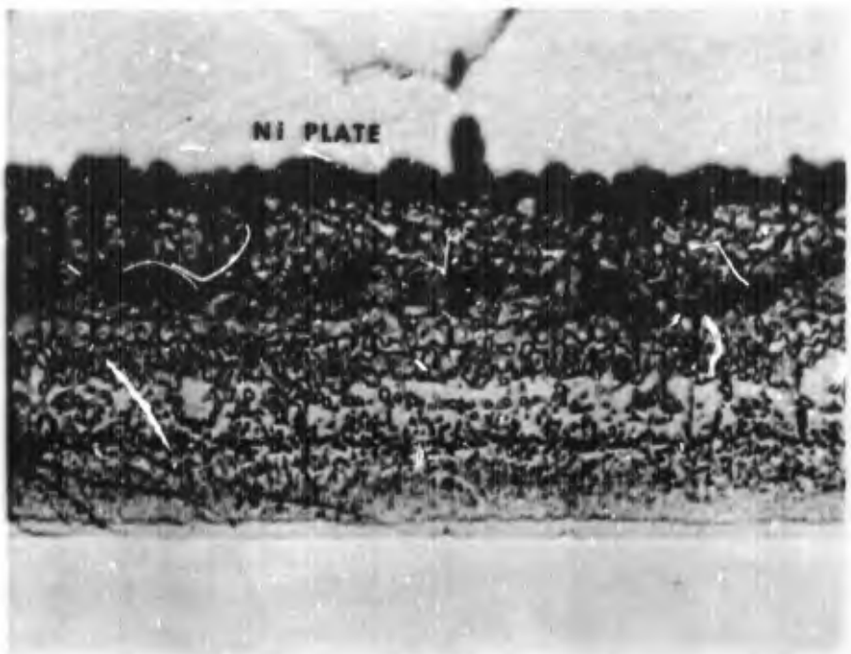
UNETCHED MAG: 500X

Figure 131. Microstructure of the Sylcor Ag-Al-Si (508-F) Coating on Cb-132M Columbium Alloy Compatibility Test Specimen (away from overlap) After 120 Hours at 1600°F in Still Air



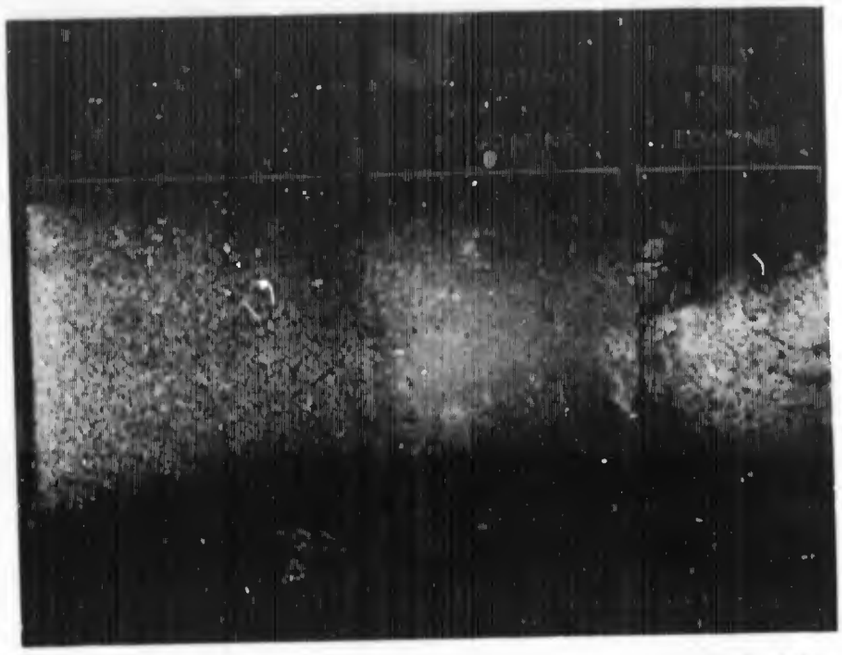
ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER MAG: 250X

Figure 132. Microstructure of a Sylcor Ag-Al-Si Coating/TRW TiCr-Si Coating Overlap Area on Cb-132M Columbium Alloy After 120 Hours at 1300°F in Still Air (note alloying in outer silicide layer of TiCr-Si coating)



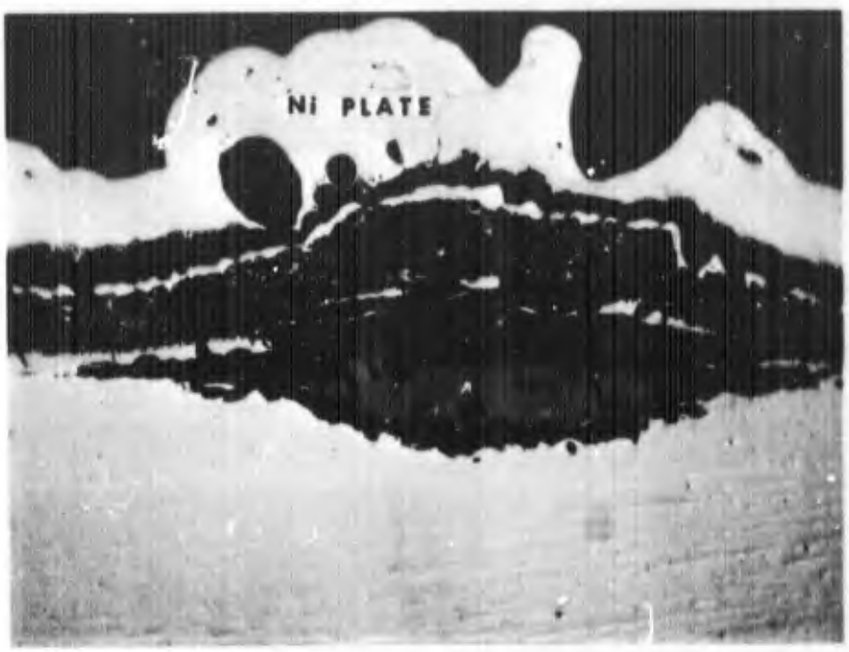
ETCH: 33% HYDROFLUORIC ACID, 33% NITRIC ACID, 33% WATER MAG: 500X

Figure 133. Microstructure of the TRW TiCr-Si Coating on Cb-132M Columbium Alloy Compatibility Test Specimen (away from overlap) After 120 Hours at 1600°F in Still Air



MAG: 3.5X

Figure 134. Compatibility Test Specimen After 79 Hours at 1300°F in Still Air Showing Surface Ruptures In and Adjacent to the Overlap Area



UNETCHED MAG: 50X

Figure 135. Microstructure of Ruptured Region in Overlap Area on Compatibility Test Specimen After 79 Hours at 1300°F in Still Air

SECTION IV

CONCLUSIONS AND RECOMMENDATIONS

1. CONCLUSIONS

The following conclusions have been drawn as a result of the coating-substrate systems evaluation conducted in Phases I through III of the program:

- TRW TiCr-Si (vacuum) pack coated D-43 columbium alloy is superior to similarly coated C-129Y alloy and Sylcor Ti-CrTi-Si (triplex) pack coated D-43 alloy for sheet metal turbine vane application.

- The Sylcor SiCrTi slurry coating has considerable potential for turbine vane application based on the results of preliminary oxidation-erosion and thermal fatigue screening tests. Since this coating was not a candidate for complete evaluation (coating improvement and advanced evaluation) in this report period, it cannot be accurately compared with the other potential vane coatings at this time.

- TRW TiCr-Si (vacuum) pack coated D-43 alloy is a candidate for engine testing as a turbine vane composite in this program.

- TRW TiCr-Si (vacuum) pack coated Cb-132M alloy is a candidate for advanced evaluation as a turbine blade airfoil system.

- The TRW TiCr-Si (slip) pack coating does not show promise for turbine blade airfoil application because of poor oxidation-erosion and thermal fatigue performance in preliminary screening tests.

- The general performance of the TiCr-Si family of coatings in oxidation-erosion and thermal fatigue in the 2000° to 2500°F temperature range is enhanced by the presence of a continuous $(Cb,Ti)Cr_2$ Laves phase layer at the coating-substrate interface and/or accompanying microstructural characteristics.

- The resistance of basic TiCr-Si coated columbium alloy composites to foreign object damage in a turbine engine environment is poor and independent of coating composition or application process as judged by 2200°F static oxidation lives of less than 10 hours for all systems after ballistic impact at 70°-2200°F.

- The Naval Research Laboratory zinc coating does not show promise for turbine blade root application because of poor reproducibility during thermal conditioning following dip application.

- The Sylcor Ag-Al-Si (508-F) coating is superior to the Sylcor Sn-Al (505-C) coating for turbine blade root application based on 1300°F wear-galling resistance and subsequent 1300°F static oxidation life, but the performance of both coatings is marginal.

- Extrusion parameters (temperature and reduction) for Cb-132M columbium alloy must be controlled to obtain the worked (unrecrystallized) microstructure necessary for adequate forgeability or ductility in the final wrought form.

2. RECOMMENDATIONS

Based on the results obtained during this annual report period, it is recommended that:

- TRW TiCr-Si (vacuum) pack coated D-43 columbium alloy be considered as a turbine vane system for engine testing during the final phase of this program, final selection to be based on comparison with other systems evaluated during the next year.

- The TRW TiCr-Si (vacuum) pack coating on Cb-132M alloy, as a turbine blade airfoil composite, and the Sylcor Ag-Al-Si (508-F) coating on Cb-132M alloy, as a blade root system, be fully investigated through advanced evaluation testing as promising candidates for the respective applications.

- The Sylcor SiCrTi slurry coating, modified as based on existing data, be fully evaluated for turbine vane and possibly blade airfoil application during the next year.

- Emphasis of the next year's effort be placed on evaluation of systems for the turbine blade root application which is the most critical application based on past engine testing and the results obtained during this annual report period.

SECTION V

FUTURE WORK

Following the end of the period covered by this report, a contract extension of one year duration was awarded to permit the evaluation (Items 8 through 11 below) of seven additional coating-substrate systems before selections are made for the engine testing of turbine blades and vanes. A brief description of all future work under this contract (as amended) is given in the following paragraphs.

1. ITEMS 4 and 5, ADVANCED EVALUATION

Item 4, Coating Life Evaluation, has been started, as reported in Section III, Technical Discussion. The remaining work to be done under this Item is the Phase III evaluation of the remodified TRW TiCr-Si (vacuum) pack coated Cb-132M alloy as a blade airfoil system. The evaluation will include oxidation-erosion and thermal fatigue tests at 2000°, 2200°, 2400°, and 2500°F and "melting" tests to evaluate the effect of 2600°F transient overshoots on 2200°F oxidation-erosion life.

The work to be performed under Item 5, Composite Properties Evaluation, as described in the Statement of Work in the original contract, was started during the report period but was not completed. However, the contract amendment which has just taken effect transfers all of the remaining Item 5 work (and some of the already completed Item 5 work) to the new Items 6 and 7. Therefore, the work of Item 5 is now considered to be complete.

2. ITEMS 6 and 7, COMPOSITE PROPERTIES EVALUATION

Item 6, Blade Airfoil Composite Properties, and Item 7, Blade Root Composite Properties, constitute Phase IV of the program.

Under Item 6, the properties of remodified TRW TiCr-Si (vacuum) pack coated Cb-132M alloy will be determined for blade airfoil application. The tests will consist of mechanical fatigue in air at 2200°F, creep testing in air in the 1800°-2500°F range, and ballistic impact at 70°-2600°F to establish a transition temperature based on postimpact 2200°F static oxidation life. The remaining work under Item 7, partially completed as Item 5 work as described previously in this report (Section III, Technical Discussion; paragraph 4.3.3) will consist of mechanical fatigue testing of Sylcor Ag-Al-Si (508-F) coated Cb-132M alloy in the 1300°-1600°F temperature range in

air. Similar tests will be conducted on specimens with a root-airfoil coating overlap. Material for the above testing under Items 6 and 7 is presently being procured.

3. ITEM 8, ADDITIONAL PRELIMINARY SCREENING EVALUATION

Item 8, Phase V of the program, will involve the evaluation of seven additional coating-substrate systems using the same preliminary screening tests described in this report under Item 2 in Section III. Two of these systems will not be candidates for further evaluation under Item 9 through 11 described below. Candidate systems for inclusion in this phase as vane, blade airfoil, or blade root composites are presently being reviewed.

4. ITEM 9, COATING IMPROVEMENT OF ADDITIONAL SYSTEMS

Under this item, Phase VI of the program, appropriate coating composition and/or process modifications will be made on those systems whose performance was unsatisfactory but promising in preliminary screening tests (Item 8), but not on more than five of those systems. Phase V tests will be repeated on each of the modified systems to establish the degree of improvement and justify their promotion to Phase VII (Items 10 and 11).

5. ITEMS 10 and 11, ADDITIONAL ADVANCED EVALUATION

Item 10, Coating Life Evaluation, and Item 11, Composite Properties Evaluation, represent Phase VII of the program. The systems to be evaluated will be selected on the basis of Phase V and Phase VI results and after consultation with the Air Force Materials Laboratory Project Engineer.

Work under Item 10 will consist of oxidation-erosion and thermal fatigue testing (to failure) of the optimized blade and vane coating (or coatings), and also static oxidation testing (to failure) of the optimized blade root coating (or coatings), to determine the protectiveness, diffusional stability, and reliability of the coating system (or systems), as well as the ability to withstand transient temperature overshoots.

Under Item 11 the following tests will be conducted: stress-rupture and impact on turbine vane composite(s); mechanical fatigue, creep, and ballistic impact on blade airfoil composite(s); and mechanical fatigue on blade root composite(s).

6. ITEMS 12 and 13, COMPONENT DESIGN AND SIMULATED ENGINE TEST

Item 12, Preliminary Test Design and Material Procurement, and Item 13, Simulated Engine Test, are Phase VIII of the program. Turbine vane, blade airfoil, and blade root systems for engine testing will be selected with approval of the Air Force Materials Laboratory Project Engineer.

Turbine vanes and blades will be designed with the aid of the Phase VII mechanical test data and the necessary substrate material purchased for the fabrication of engine test components under Item 12. Upon receipt of specific authorization from the Air Force, a number of vanes and blades will be fabricated under Item 13. These components will be installed in a turbine development engine, and tested at temperatures approaching substrate alloy limits to establish short-time, high-performance capability under actual engine conditions and to provide an indication, by correlation with longer-term laboratory test data, of the multihundred hour performance of this hardware.

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11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY
N/A	Air Force Materials Laboratory (MAMP) Wright-Patterson AFB, Ohio 45433

13. ABSTRACT

Data generated using laboratory tests such as oxidation-erosion, thermal fatigue, ballistic impact, and wear-galling to simulate turbine engine environments are reported for eight coated columbium alloy composites. Applications considered were turbine vane and turbine blade airfoils operating in the 1800°-2500°F temperature range and turbine blade roots at 1300°-1600°F. Five of the eight coatings were modified after preliminary screening tests and re-evaluated. Complete evaluation of TRW TiCr-Si (vacuum) pack coated D-43 alloy, including stress-rupture testing, established the system as a turbine vane candidate for engine testing in the last phase of the program. Preliminary results on Sylcor SiCrTi slurry coated D-43 alloy indicated promise for turbine vane use and continued testing was recommended. TRW TiCr-Si (vacuum) pack coated Cb-132M alloy demonstrated potential as a blade airfoil composite and is being fully evaluated. The Naval Research Laboratory zinc coating selected for possible blade root application was deleted from the program because of nonreproducibility. Sylcor Ag-Al-Si (508-F) coated Cb-132M was the most promising turbine blade root composite in screening tests and will be fully investigated for that purpose. Performance of all candidate blade root coatings was marginal, and emphasis on this area during the next yearly effort was recommended.

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UNCLASSIFIED

Security Classification

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Columbium Alloys Coatings (Oxidation-Resistant) Gas Turbine Engines						

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