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RESEARCH MEMORANDUM

ADHESIVE AND PROTECTIVE CHARACTERISTICS OF CERAMIC COATING A-417 AND ITS EFFECT ON ENGINE LIFE OF FORGED REFRACTALOY-26 (AMS 5760) AND CAST STELLITE 21 (AMS 5385) TURBINE BLADES

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NATIONAL ADVISORY COMMITTEE
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SUMMARY

An investigation was conducted to determine the adhering and protective characteristics of National Bureau of Standards Ceramic Coating A-417 and the effect the coating had on the life of forged Refractaloy 26 and cast Stellite 21 turbine blades. Coated and uncoated blades were run in a full-scale J33-9 engine and were subjected to simulated service operations consisting of consecutive 20-minute cycles (15 min at rated speed and approximately 5 min at idle). The results of the investigation show that the ceramic coating NBS A-417 adhered well to Refractaloy 26 and Stellite 21 turbine blades operated at 1500° F. In addition, the coating was found to prevent corrosion of the Refractaloy 26, a nickel-base alloy known to be corrosion-sensitive, and the Stellite 21, a cobalt-base alloy known to be relatively corrosion-resistant. Even though the coating prevented corrosion of both alloys, it apparently did not affect the engine life of the alloys.

INTRODUCTION

Increasing application of high-temperature alloys has severely drawn upon the available supply of critical alloying elements. Turbojet engines, and in particular turbine-blade alloys, frequently contain considerable quantities of the scarce elements columbium, cobalt, tungsten, molybdenum, chromium, and nickel, several of which contribute to the corrosion resistance of the alloys as well as to high-temperature strength. If, for example, the amount of nickel and chromium were reduced, the alloys would be more susceptible to corrosive attack by hot gases from the combustion chamber. This lack of corrosion resistance has been a principal factor that has limited the use in turbojet engines of several ferrous and low-critical-element alloys which have the necessary strength for this application.

One method of improving the corrosion resistance of turbine blades is by coating with a material which will withstand high operational temperatures. A previous investigation of ceramic coatings on cast Stellite 21 alloy turbine blades (ref. 1) has shown that the National Bureau of Standards Ceramic Coating A-417 adhered well and did not noticeably affect the creep characteristics of the alloy in a full scale, short-time engine test.

The purposes of the investigation reported herein were as follows: (1) to determine whether the ceramic coating NBS A-417 would adhere well to turbine blades being operated for long periods of time in a full-scale turbojet engine, and (2) to determine whether the coating would prevent corrosion of a nickel-base alloy known to be susceptible to high-temperature corrosion (Refractaloy 26, ref. 2). The cobalt-base alloy Stellite 21 was selected because its behavior in engine tests is relatively well known.

Coated and uncoated blades of both alloys were mounted in a turbine wheel and run in a J33-9 engine at a temperature of 1500° F and stress of approximately 21,000 pounds per square inch. The engine test consisting of a repeated 20-minute cycle was employed to simulate service conditions. The engine was shut down at the end of the work day or whenever a blade failed. All failed blades were metallographically examined to determine the protective quality of the A-417 ceramic coating, the adherence of the coating, and the microstructural changes occurring in the alloys during operation. The investigation was conducted at the NACA Lewis laboratory.

MATERIALS

Processing of blades. - Turbine blades of forged Refractaloy 26 in fully aged condition and of cast Stellite 21 in the as-cast condition were submitted to the National Bureau of Standards for coating with the ceramic A-417, the composition of which is given in table I. The coating procedure was as follows:

1. Blades were cleaned by sand blasting, with 60 mesh sand under 80 pounds per square inch air pressure. Care was exercised to keep the surfaces to be coated free of any grease or oil.

2. The cleaned blades were dipped into a slurry of the A-417 ceramic coating and drained. The dipping process was controlled to yield a finished coat 1 to 2 mils in thickness.

3. The coated blades were fired in a furnace for 10 minutes at 1850° F (in air) and were then removed from the furnace and air-cooled to room temperature.

During the firing of the Refractaloy 26 blades, a wrinkled coating was produced on some of them which was attributed to excessive thickness of the coat. In order to correct this, the uneven coat was removed from the blades by sand blasting, and a new coat was applied that had a finished thickness of 0.5 to 1.0 mils. Therefore, some of the Refractaloy 26 blades were fired twice at 1850° F and a variable of double firing (two 10-min periods at 1850° F instead of one) was introduced (table II).

For comparative purposes, uncoated blades of Refractaloy 26 and Stellite 21 were subjected to the same firing treatments. The thickness of the ceramic coatings and the number of firing cycles are shown in the following tabulation:

Alloy	Thickness of ceramic coat, mils	Number of 10-min firing cycles at 1850° F
Refractaloy 26	1.0 - 2.0	1
Refractaloy 26	None	1
Refractaloy 26	0.5 - 1.0	2
Refractaloy 26	None	2
Stellite 21	1.0 - 2.0	1
Stellite 21	None	1

Radiographic imspection of blades. - Before any coating was applied, all blades were radiographed and found radiographically sound.

Blades evaluated in engine. - Refractaloy 26 and Stellite 21 blades of each condition tabulated were installed in a J33-9 engine, the operation of which will subsequently be described. Table III shows the number of coated, uncoated, and "as-fired" blades evaluated.

Engine operation. - The blades were evaluated in a full-scale J33-9 turbojet engine. Engine operation consisted of repeated cycles of 20 minutes duration (15 min at full rated speed of 11,539 rpm and 5 min at an idle of 4000 rpm). At full rated speed a temperature of 1500° ±10° F was produced at the middle of the blades and the following centrifugal stresses were also produced in the middle of the blades:

Refractaloy 26, psi 20,500
Stellite 21, psi 21,300

The centrifugal stresses were calculated from the known rotor and material constants of radius, rotational speed, blade volume, and density and vary with the geometry of the blade and the density of the alloy under investigation. A detailed explanation of stress distribution of the blade is found in reference 3. Details of engine operation and blade temperature control may be obtained from reference 4.

METALLURGICAL STUDIES AND EXAMINATION OF BLADES AND CERAMIC COATS

Macroscopic and metallographic examinations were made of several blades in the "as-processed" conditions. Blades which failed during engine operation were macroscopically examined to determine the over-all condition of the coatings and subsequently were sectioned for the microscopic work.

Blade sectioning was dictated by the appearance of the coating, the type and location of the initial failure zone, and the method of propagation of the failure. The areas of interest were mounted, polished, and etched. Refractaloy 26 specimens were etched electrolytically in a 50 percent hydrochloric acid and water solution and the Stellite 21 specimens were etched electrolytically in a 5 percent aqua regia and water solution.

Rockwell hardness determinations were made of as-received, processed, and operated blades of both alloys. In the case of unoperated blades (as received), hardness tests were made on cross sections of the airfoil (2 in. from the base, where critical stresses occur in engine operation). In the case of fractured blades, hardness tests were made on sections 1/8 inch below the failure zone of the operated blades.

RESULTS AND DISCUSSION

Appearance of blades. - Appearances of typical coated blades before and after operation are shown in figure 1. The blade in figure 1(a) is representative of as-coated blades. The coating is continuous over the airfoil surface and has a characteristic chromic oxide green color with a vitrous sheen. During operation, the sheen is removed and the color is lightened by the action of the hot gases. The blade of figure 1(b), Stellite 21, and the blade of figure 1(c), Refractaloy 26, are typical blade failures. Stress-rupture cracking of the Stellite 21 blades was not localized to the critical stress and temperature zone (2 in. above the root flat form) but became general over a wide area, giving an "orange peel" effect. Numerous grain boundaries were opened with a consequent shifting of some grains. This mass movement of metal broke the continuity of the coating, as would be expected, but the ceramic-to-metal bond remained intact. The Refractaloy 26 blade failed completely by stress rupture. The spalling of the ceramic coat noted in the center and right-hand blades was caused by fragments of prior failures impinging on these blades. Macroscopic examination of the coated blades showed that the damage to the ceramic coating by impinging fragments was slight.

Results of engine operation. - The results of the engine operation are plotted in figure 2, and the average life of the alloys is listed in table III. All the Refractaloy 26 blades and more than half of the Stellite 21 blades failed by stress rupture.

It is evident from these data that the ceramic coating did not noticeably improve blade performance; nor did the coated blades fail by mechanisms different from those of the uncoated blades, as the symbols of figure 2 indicate. It will subsequently be shown that the ceramic coating protected Refractaloy 26 and Stellite 21 blades from corrosion by the combustion products. In the case of Refractaloy 26, the alloy known to be susceptible to high-temperature corrosion, an improvement in performance would be expected; however, the results indicate that apparently the performance of these blades did not depend on their corrosion resistance.

Metallographic Examination of Refractaloy 26

Before and After Operation

Examination of uncoated blades. - A typical oxide coating produced by solution treating and aging Refractaloy 26 is shown in the photomicrograph of figure 3. The oxide layer was approximately 0.00025 inch thick, and penetration of the grain boundaries of the recrystallized zone was observed although it is not apparent in the photomicrograph. Excessive grain-boundary corrosion of uncoated Refractaloy 26 blades operated for 106 and 143 hours is shown in figure 4. Severe corrosion of this type would be expected to lead to short operational life. However, as has been noted, failure times of coated and uncoated blades are practically the same.

Examination of coated blades. - Metallographic examinations of specimens cut from portions of operated blades that did not elongate severely showed that the ceramic coating prevented corrosion and adhered well to the surfaces of the metal (fig. 5). Some of these sections, which were protected from corrosion, were obtained from the immediate vicinity of the fractures, in the hottest, most critically stressed zones of the blades. In other specimens, in areas near the fractures, corrosion was observed, but the fracturing of the ceramic that permitted this corrosion almost certainly occurred after the blade alloy entered third-stage creep.

A layer of recrystallized grains was observed at the surfaces of the Refractaloy 26 blade airfoils and is believed to have resulted from surface working followed by heat treatment.

Three of the four blades which had a coat thickness of 1.0 to 2.0 mils developed striations on the concave face during operation (see fig. 6). The striae probably formed in these thick coatings during cooling at shut downs of the engine, which produced an elastic recovery of the base metal.

The ceramic is believed to have flowed plastically as the metal elongated both plastically and elastically during heating. On cooling, the metal recovers by a greater amount than the ceramic. To relieve the compressive stresses, ridges are built up on the outer surface by plastic movement of the coating. This analysis is substantiated by an examination of a cross section of the striae. Photomicrographs of this section (fig. 7) show a ridge and valley on the coat surface, but no visible change occurs to the ceramic-metal interface.

The failure zone shown in figure 8 (coated Refractaloy 26) is typical of all failures in this alloy. The mode of failure is stress-rupture which propagates intergranularly. Oxidation of the grain boundaries occurs only after the coating is broken by excessive elongation (stress-rupture).

Metallographic Examination of Stellite 21 Blades

Operated in a Turbojet Engine

Examination of uncoated and as-cast Stellite 21 blades. - A thin oxide layer (approximately 0.0003 in.) was produced on all unprotected Stellite 21 blades by the hot gases (fig. 9(a)) and slight oxide penetration of the grain boundaries occurred (although infrequently) during engine operation.

Examination of coated Stellite 21 blades. - Metallographic examinations of operated blades showed that the adherence of the A-417 coating to Stellite 21 was excellent. No oxide penetration of the coating was detected on any of the coated blades. A typical coating-to-metal interface is shown in figure 9(b).

Precipitation hardening of the Stellite 21 occurred during the firing cycle and operation of the blades in the turbojet engine. Such precipitation is common for the Stellite 21 and is observed near primary carbides, in pearlite colonies, and in slip planes.

Failures of all Stellite 21 blades were by stress-rupture and the fracture paths were found to be intergranular.

HARDNESS SURVEY

Hardness changes measured in Refractaloy 26 and Stellite 21, resulting from the firing treatments and engine operation, are listed in table IV. Refractaloy 26 decreased in hardness from Rockwell-A hardness 64.7 to 49.9. This drop in hardness that occurred in the Refractaloy 26 blades was caused by heating the alloy 50° F above the A.M.S. specified solution-treating temperature of 1800° $\pm$10° F. Thus the Refractaloy 26 specimens were partly solution treated. The decrease in hardness could have been compensated for by reaging the specimen after coating. If this coating were applied commercially to alloys of low strategic content, compensatory heat treatments following the firing cycle would no doubt be necessary. An increase in Rockwell-A hardness of the Stellite 21 from 65 to 67 is attributed to age hardening at the firing temperature of 1850° F.

SUMMARY OF RESULTS

The results of this investigation, which was conducted to determine whether the ceramic coating NBS A-417 would adhere well to turbine blades being operated for long periods of time in a J33-9 turbojet engine and which was also to determine whether the coating would prevent corrosion of Refractaloy 26 and Stellite 21, may be summarized as follows:

1. The ceramic coating (NBS A-417) adhered well to Refractaloy 26 and Stellite 21 turbine blades operated at 1500° F.

2. The coating was found to prevent corrosion of a blade alloy known to be susceptible to high-temperature corrosion, Refractaloy 26, and also was found to prevent corrosion of Stellite 21, a relatively corrosion-resistant alloy which was used as a basis for comparison.

3. The ceramic coating apparently did not affect the engine life of the Refractaloy 26 and Stellite 21 turbine blades even though the coating prevented corrosion of both alloys.

4. Refractaloy 26 was solution-treated by firing at 1850° F for 10 to 20 minutes.

CONCLUDING REMARKS

This investigation has demonstrated that it is possible to prevent corrosion of turbine blades by coating them with ceramics. The application of ceramic coats to metals usually requires high firing temperatures to bond the ceramic to the metal. These high firing temperatures may adversely affect the mechanical properties of the alloys and compensatory heat treatments may be necessary to restore the lost properties.

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REFERENCES

1. Morse, C. Robert: Comparison of National Bureau of Standards Ceramic Coatings L-7C and A-417 on Turbine Blades in a Turbojet Engine. NACA RM E8I20, 1948.

2. Garrett, F. B., and Yaker, C.: Relation of Engine Turbine-Blade Life to Stress-Rupture Properties of the Alloys, Stellite 21, Hastelloy B, Cast S-816, Forged S-816, X-40, Nimonic 80, Refractaloy 26, N-155, and Inconel X. NACA RM E51G13, 1951.

3. Kemp, Richard H., and Morgan, William C.: Analytical Investigation of Distribution of Centrifugal Stresses and Their Relation to Limiting Operating Temperature in Gas-Turbine Blades. NACA RM E7L05, 1948.

4. Farmer, J. Elmo, Darmara, F. N., and Poulson, Francis D.: Cyclic Engine Test of Cast Vitallium Turbine Buckets. I. NACA RM E7J23, 1948.

TABLE I. - COMPOSITION OF A-417 CERAMIC COATING

(a) Composition of ceramic.

Ingredient	Parts by weight
Frit 331 (see table I(b))	70
Chromic oxide (commercial)	30
Enameler's clay	5
Water	48

(b) Composition of Frit 331.

[Maximum smelting temperature, 2425° F, smelting time, $2\frac{1}{2}$ to 3 hours.]

Ingredient	Parts by weight	Computed oxide composition	
		Oxide	Percent
Flint (SiO_2)	38.00	SiO_2	38.0
Barium carbonate	56.63	BaO	44.0
Boric acid	11.50	B_2O_3	6.5
Calcium carbonate	7.14	CaO	4.0
Beryllium oxide	2.50	BeO	2.5
Zinc oxide	5.00	ZnO	5.0
	120.77		100.0

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TABLE II. - HISTORY OF BLADES USED IN THE INVESTIGATION

Alloy	Number of blades	Number of sandblasts	Thickness of A-417 coating, mils	Firing cycle time at 1850° F, min
[a]Refractaloy 26	4	1	none	[c]20
	4	[d]2	0.5 - 1.0	[c]20
	4	1	1.0 - 2.0	10
	4	1	none	10
[b]Stellite 21	6	1	none	10
	6	1	1.0 - 2.0	10
	4	--	---------	--
Other alloy	20			
Thermocouples	2			
Total	54			

[a]Heat treatment: Solution treat, 1 hr at 2100° F, air cool; age 20 hrs at 1500° F, air cool; age 20 hrs at 1350° F, air cool.

[b]As cast.

[c]Two separate treatments of 10 minutes each.

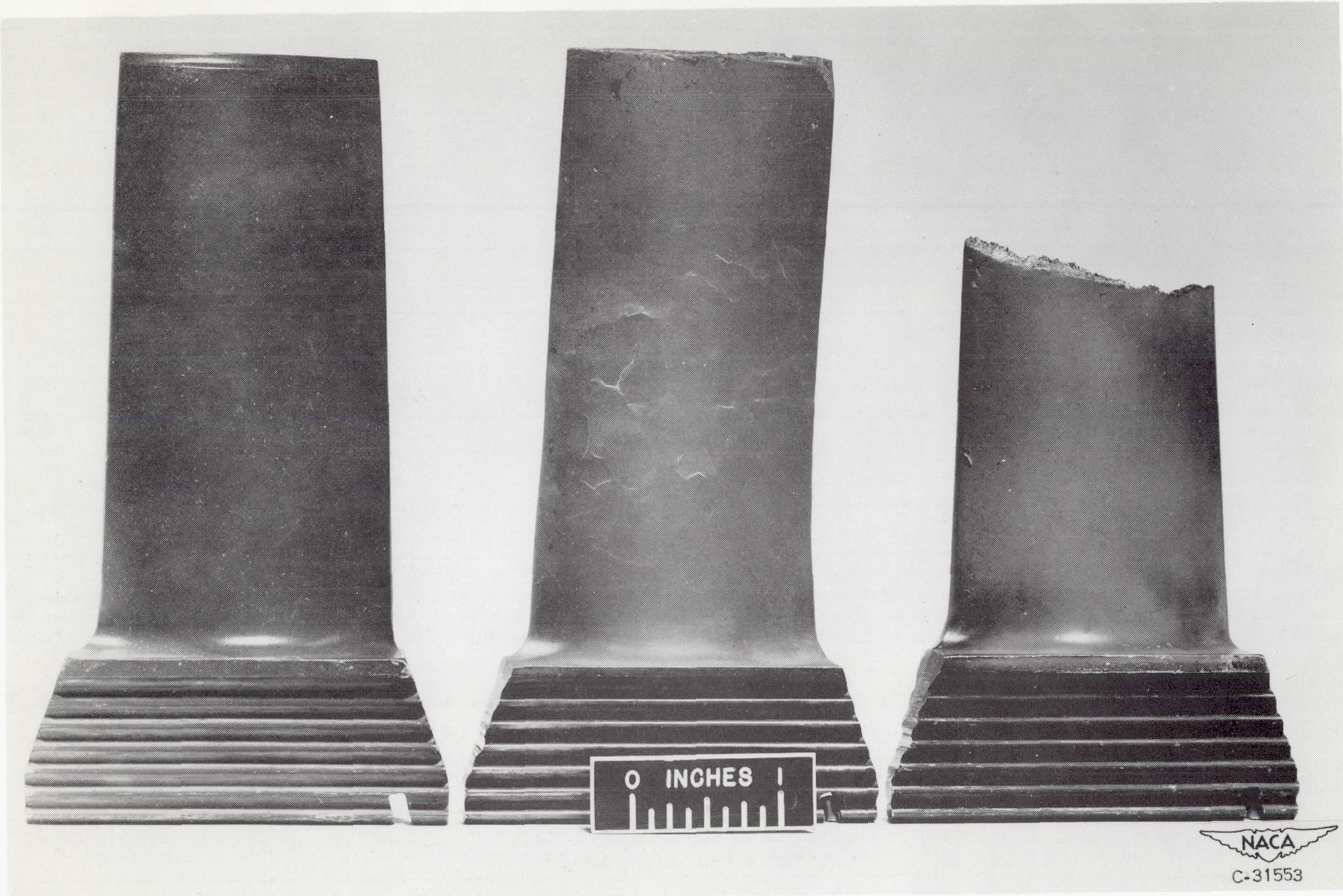
[d]First application removed because of wrinkled appearance of coat.

TABLE III. - MEAN LIFE OF BLADES

Alloy	Coating	Firing cycles	Number of blades	Mean life
Refractaloy 26	A-417	2	4	120
	A-417	1	4	131
	None	2	4	137
	None	1	4	120
Stellite 21	A-417	1	6	85
	None	1	6	107
	As cast	-	4	95

TABLE IV. - CHANGE IN HARDNESS DURING FIRING TREATMENT AND ENGINE OPERATION

Alloy	Condition	Average Rockwell-A hardness		Change	After operation	Change
		Before firing	Before operation (after firing)			
Refractaloy 26	As heat treated	64.7				
	Coated twice fired	64.7	49.9	-14.8	63.0	+13.1
	Coated once fired	64.7	51.7	-13.0	62.9	+11.2
	Uncoated twice fired	64.7	49.9	-14.8	63.2	+13.3
	Uncoated	64.7	51.4	-13.3	63.4	+12.0
Stellite 21	As cast not fired	65			73.1	+ 8.1
	Coated once fired	64	67	+ 2	74.4	+ 7.4
	Uncoated once fired	65	67	+ 2	72.5	+ 5.5



(a) As coated; unoperated.

(b) Coated Stellite 21; failed by cracking after 99.7 hours.

(c) Coated Refractaloy 26; failed by rupturing after 99.7 hours.

Figure 1. - Comparison of ceramic coated blades.

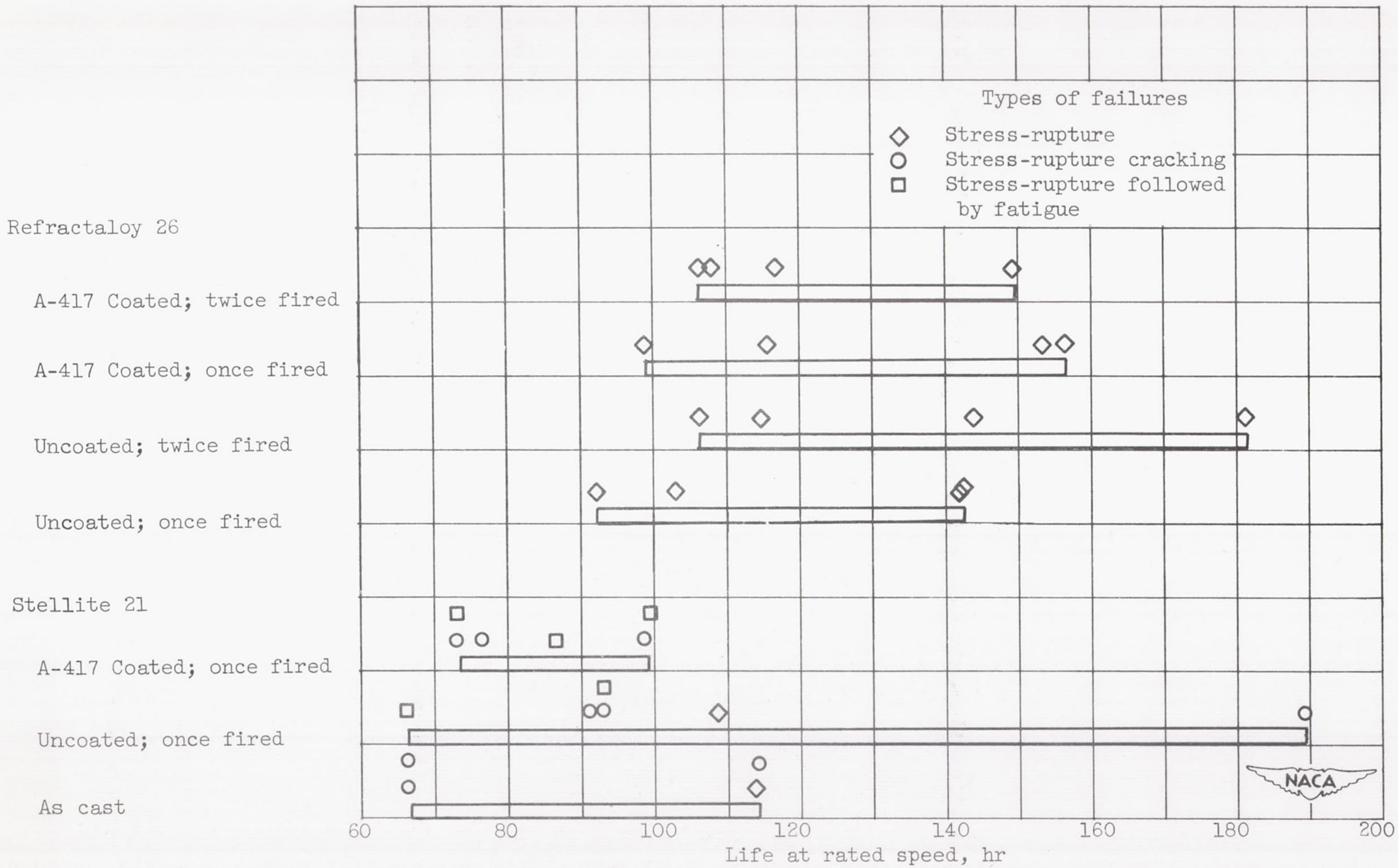


Figure 2. - Classification of A-417 coated and uncoated Refractaloy 26 and Stellite 21 blade failures.

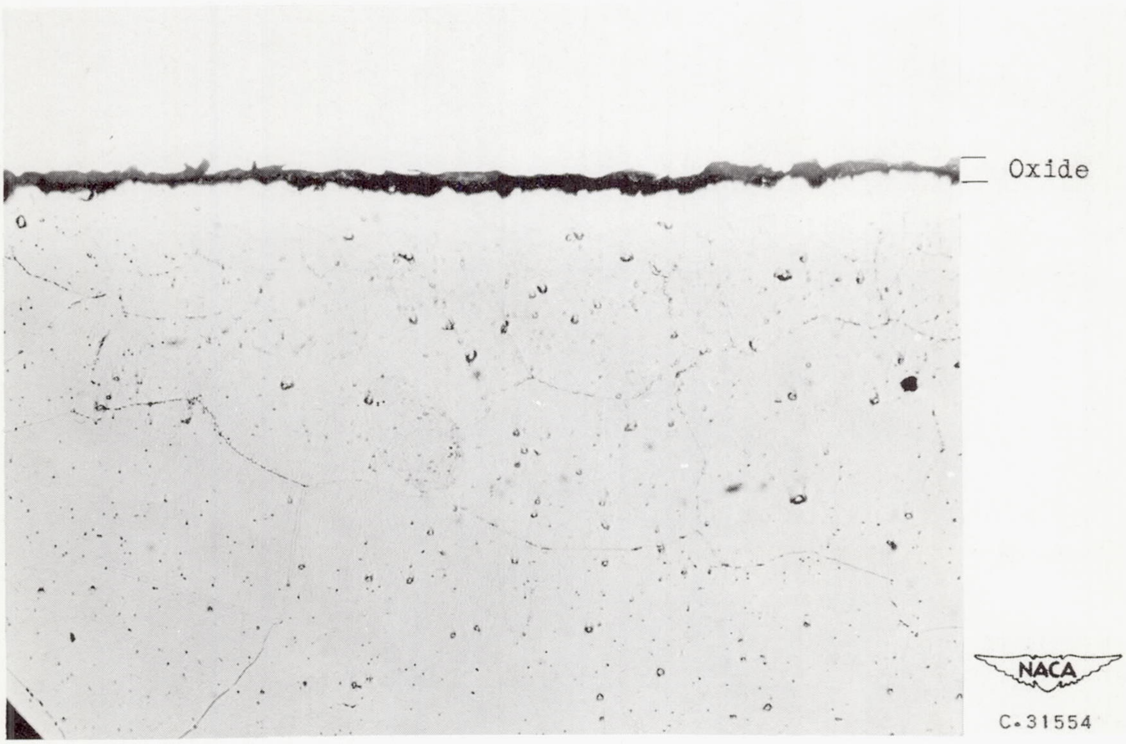
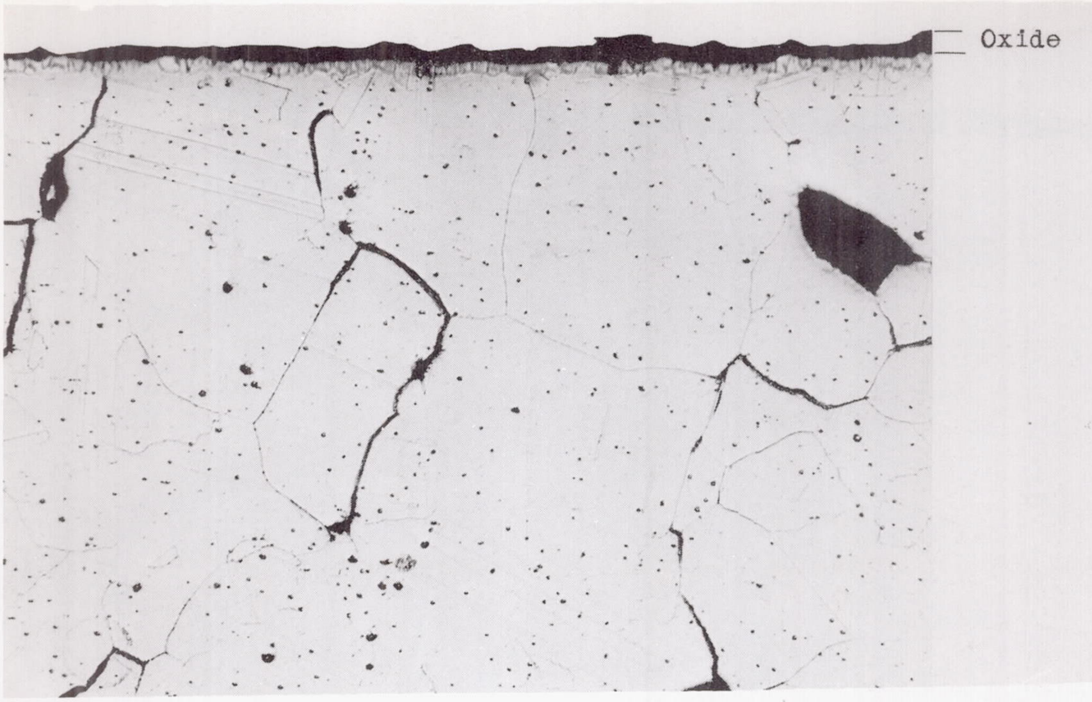
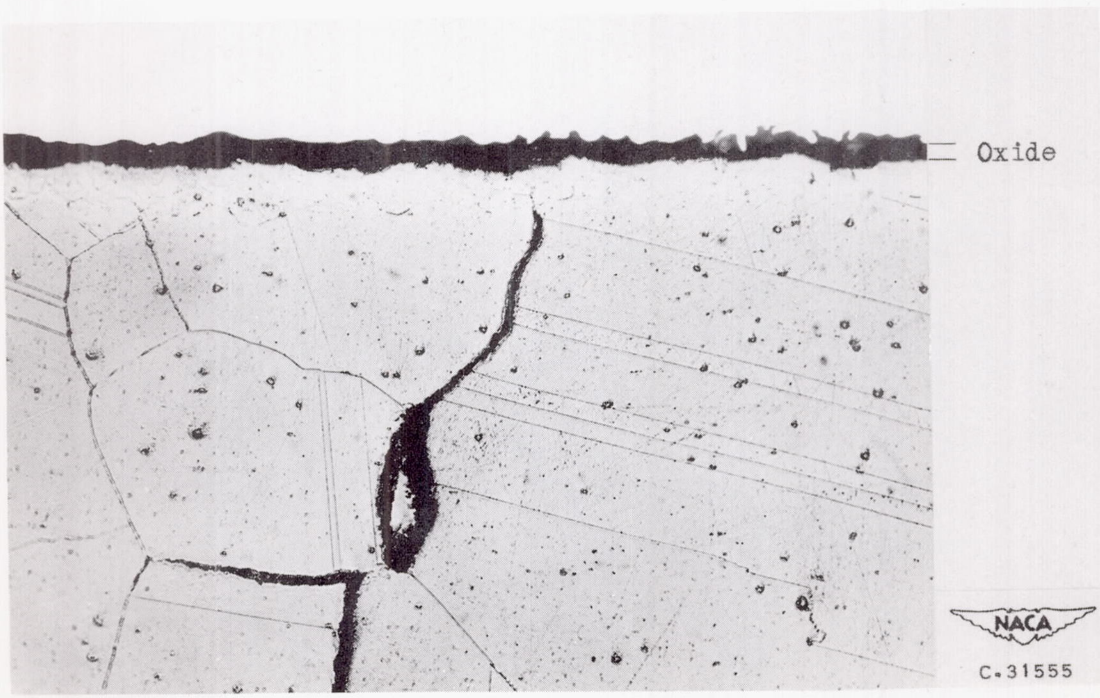


Figure 3. - Oxidation of Refractaloy 26 during heat treatment. Electrolytically etched in 50-percent hydrochloric acid. X250.

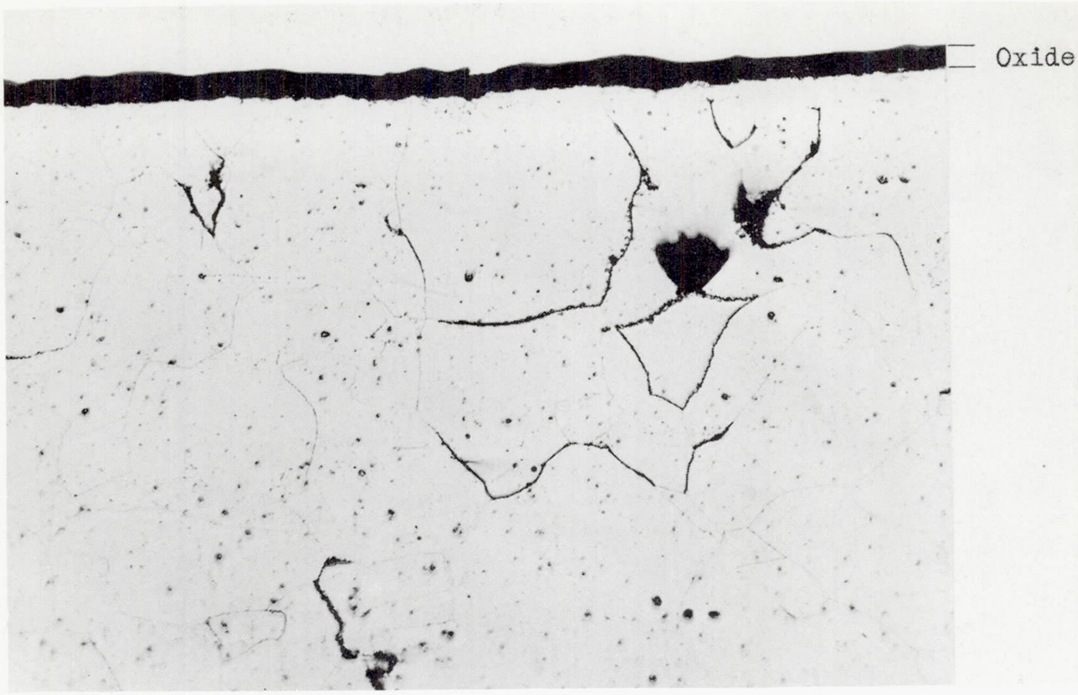


(a) Fired twice; operated 106.4 hours. Electrolytically etched in 50-percent hydrochloric acid. X100.

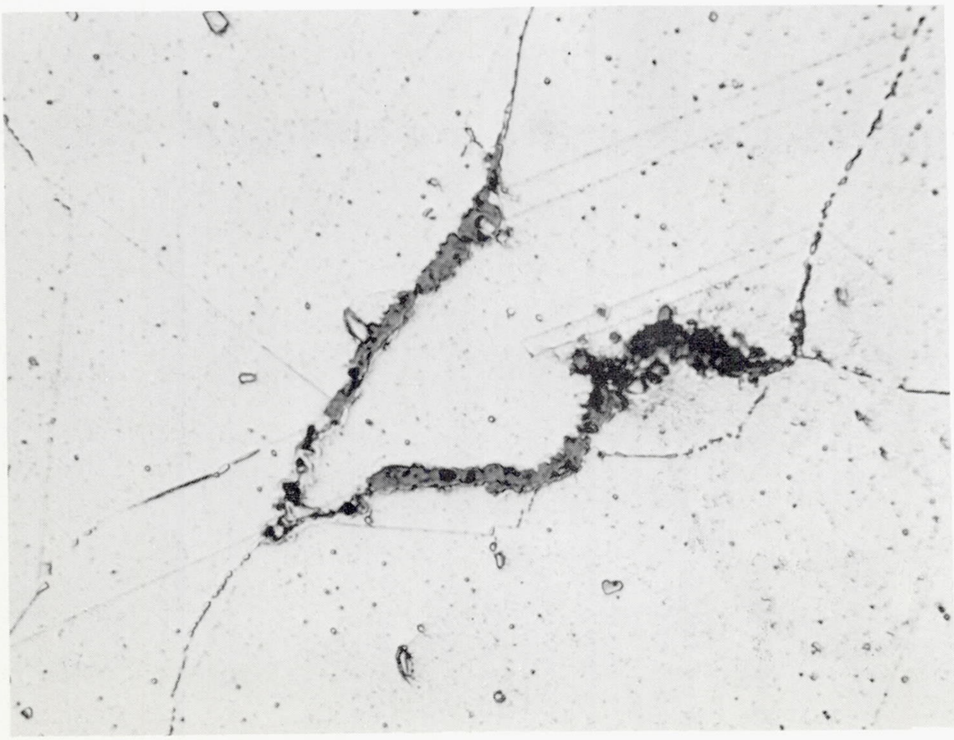


(b) Same specimen as in figure 4(a) at X250.

Figure 4. - Extensive grain boundary corrosion of uncoated Refractaloy 26 operated in a turbojet engine.



(c) Fired once; operated 142.9 hours. Electrolytically etched in 50 percent hydrochloric acid. X100.



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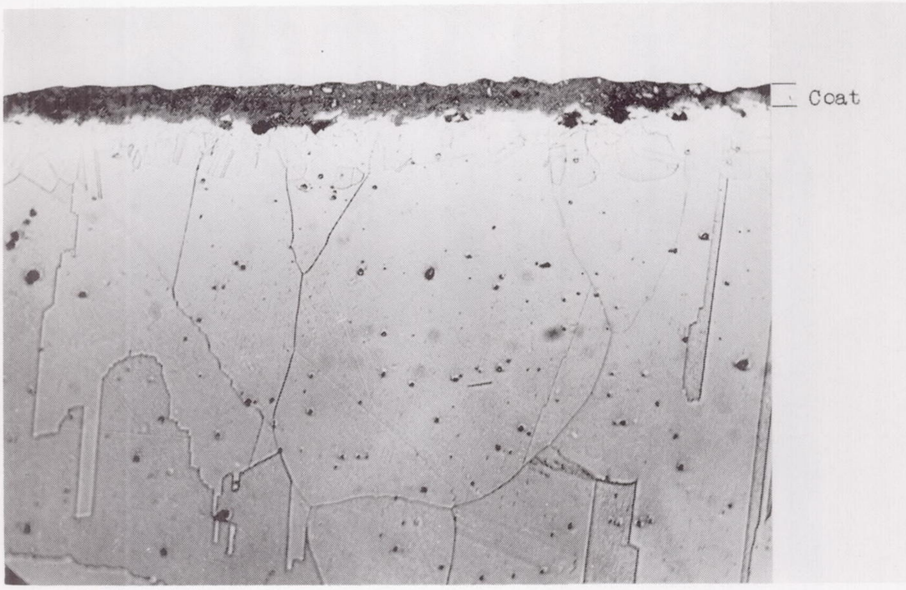
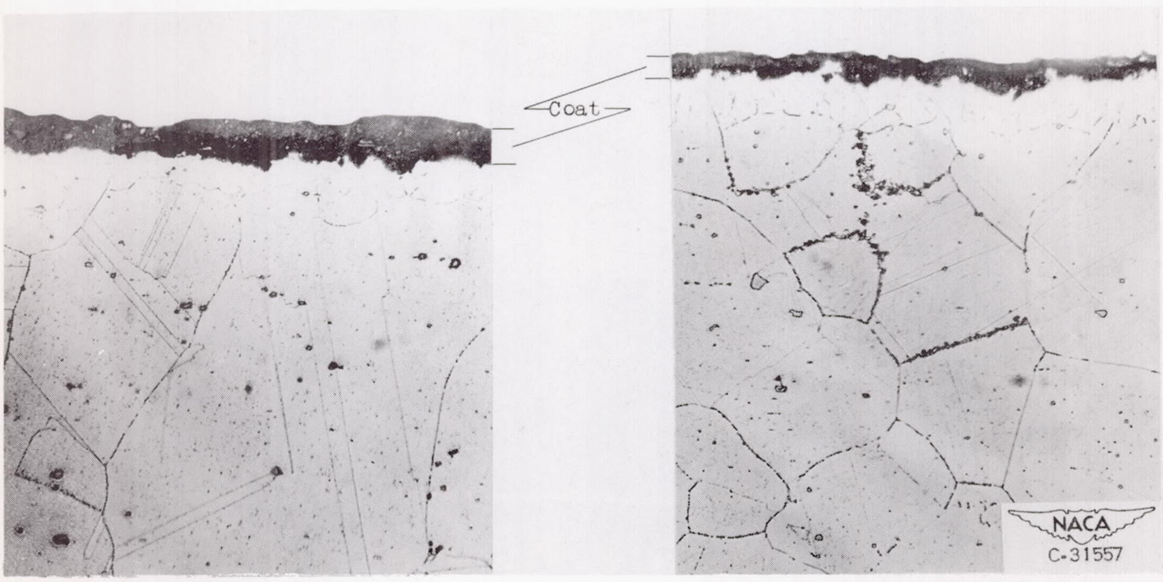
(d) Same specimen as in figure 4(c) at X750.

Figure 4. - Concluded. Extensive grain boundary corrosion of uncoated Refractaloy 26 operated in a turbojet engine.



(a) Fired once; unoperated. Electrolytically etched in 50-percent hydrochloric acid.



(b) Fired once; operated 99.2 hours. Electrolytically etched in 50 percent hydrochloric acid.

(c Fired twice; operated 149.5 hours. Extensive precipitation in grain boundaries. Electrolytically etched in 50-percent hydrochloric acid.

Figure 5. - Ceramic-metal interfaces of coated Refractaloy 26 before and after operation in a turbojet engine. Photographed at X250; reduced 23 percent in reproduction.

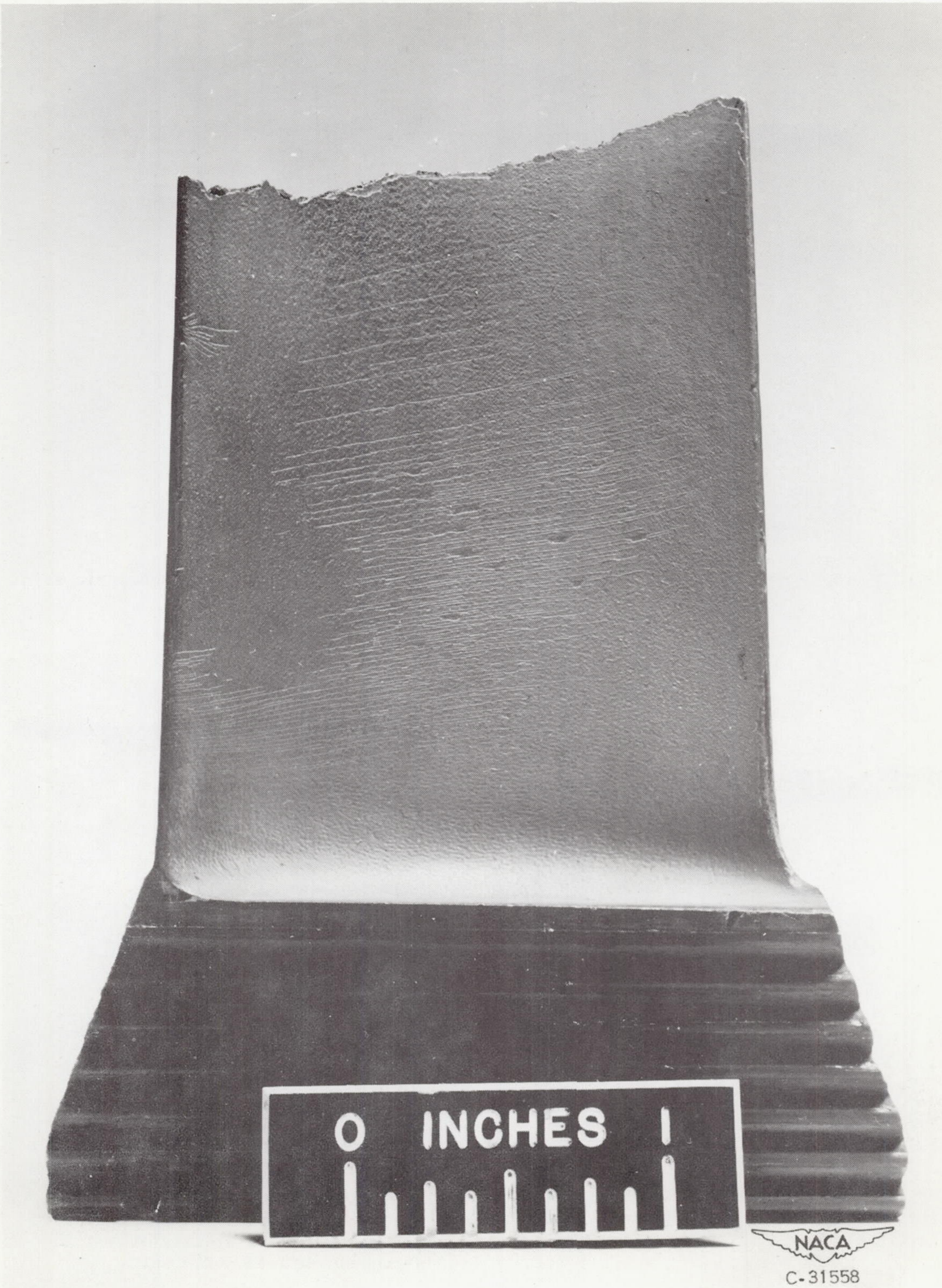
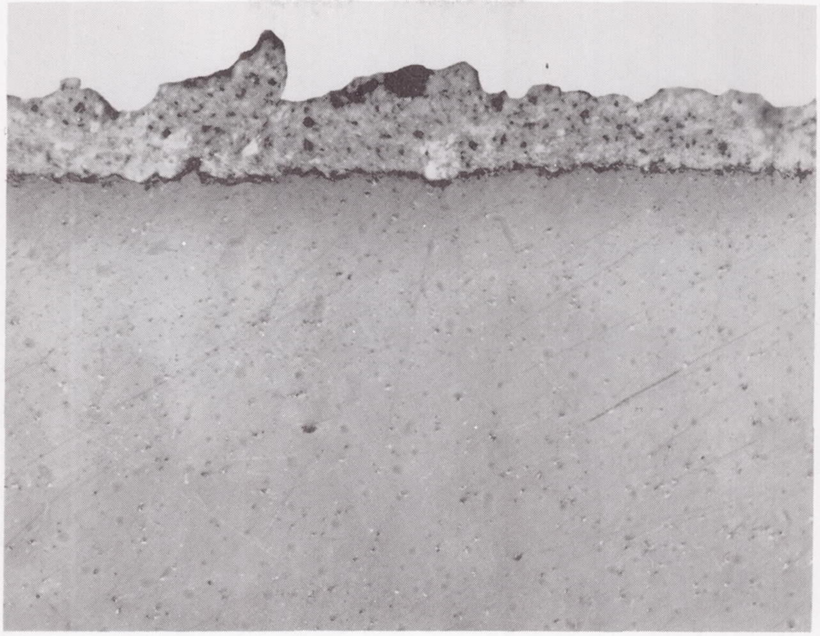
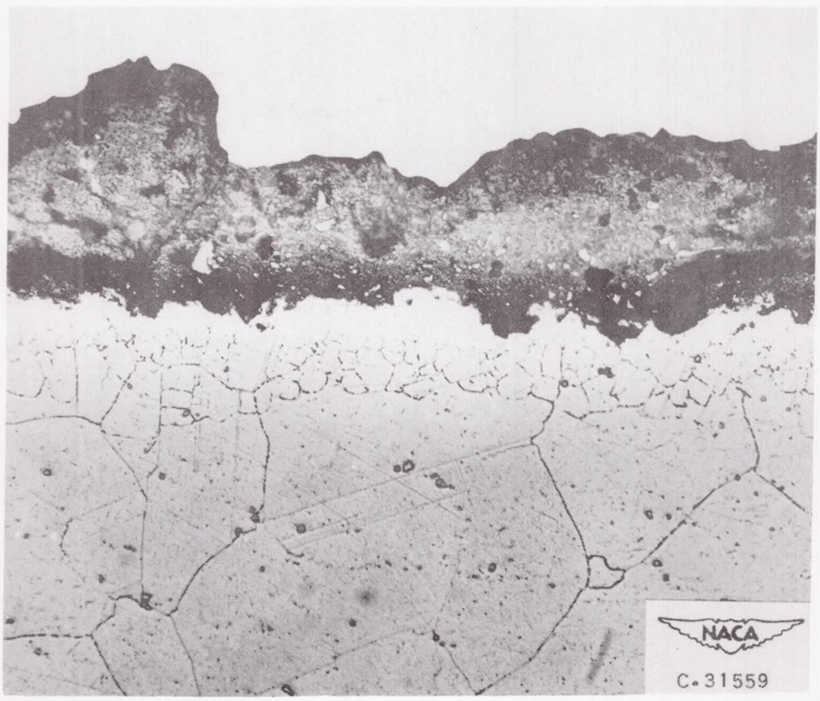


Figure 6. - Striations present in thick coating (1.0 to 2.0 mil) on Refractaloy 26 after 153.6 hours of engine operation.



(a) Unetched. X100. Polarized light.



(b) Same specimens above electrolytically etched in 50-percent hydrochloric acid. X250. Recrystallization zone produced during heat treatments.

Figure 7. - Ridges produced in thick ceramic coating on Refractaloy 26 during engine operation.

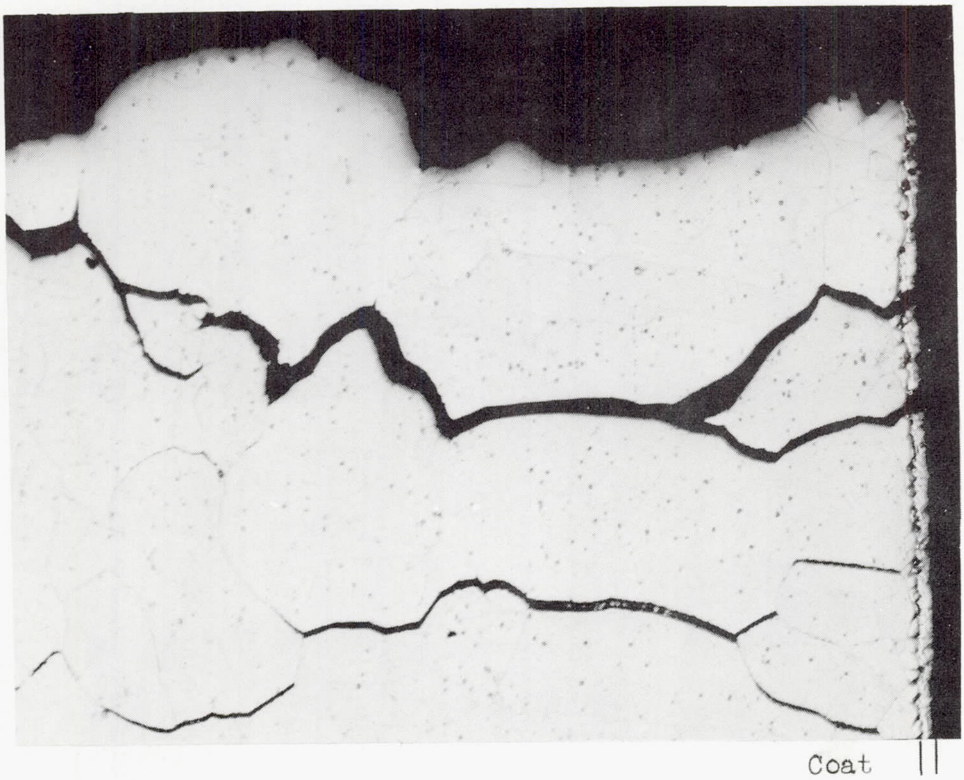
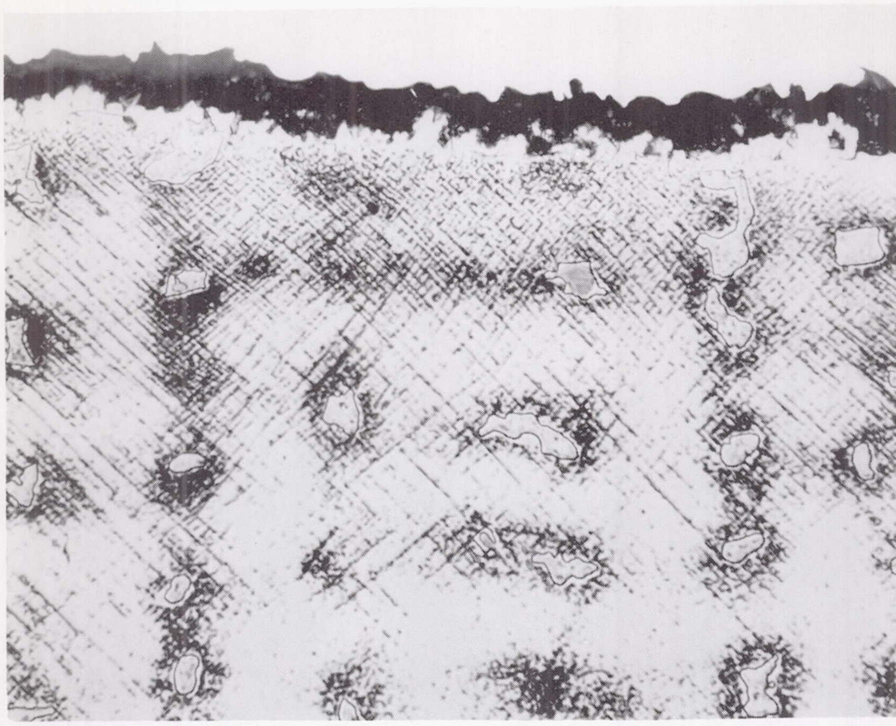
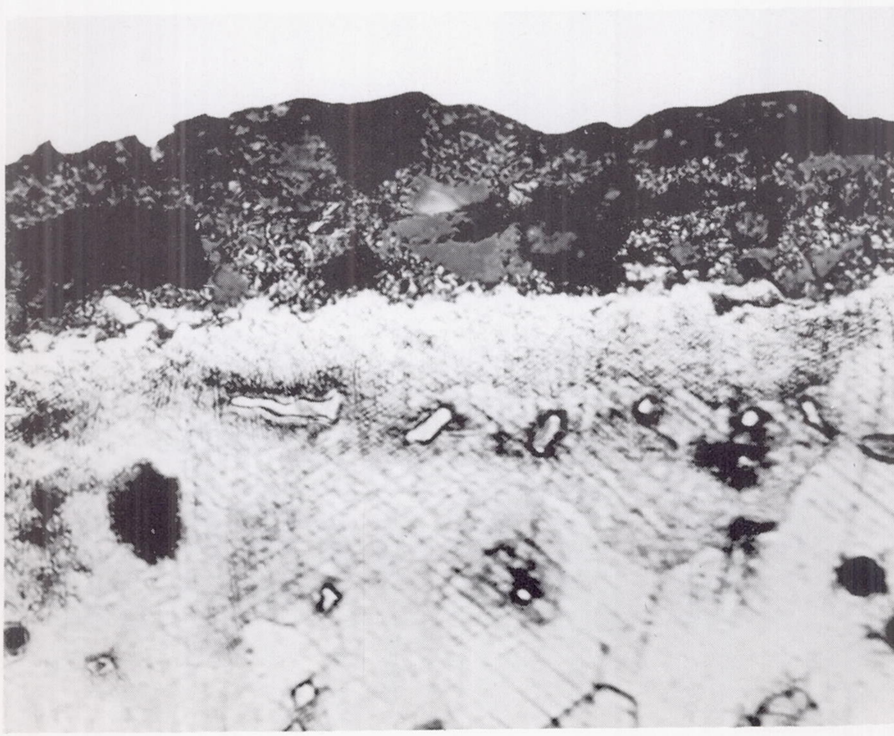


Figure 8. - Typical intercrystalline failure in coated Refractaloy 26 occurring during operation in a turbojet engine. Electrolytically etched 50-percent hydrochloric acid. X100.



(a) Uncoated; fired once; operated 66.5 hours. Electrolytically etched in 5-percent aqua regia. X750.



(b) Coated; fired once; operated 73.6 hours. Electrolytically etched in 5-percent aqua regia. X750.

Figure 9. - Comparison of uncoated and coated Stellite 21 after engine operation.