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**NASA TM X-3410**

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**DURABILITY OF ZIRCONIA  
THERMAL-BARRIER CERAMIC COATINGS  
ON AIR-COOLED TURBINE BLADES  
IN CYCLIC JET ENGINE OPERATION**

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**NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • SEPTEMBER 1976**

1. Report No. <b>NASA TM X-3410</b>	2. Government Accession No.	3. Recipient's Catalog No.	
4. Title and Subtitle <b>DURABILITY OF ZIRCONIA THERMAL- BARRIER CERAMIC COATINGS ON AIR-COOLED TURBINE BLADES IN CYCLIC JET ENGINE OPERATION</b>		5. Report Date <b>September 1976</b>	6. Performing Organization Code
		8. Performing Organization Report No. <b>E-8700</b>	10. Work Unit No. <b>505-04</b>
7. Author(s) <b>Curt H. Liebert, Richard E. Jacobs, Stephan Stecura, and C. Robert Morse</b>		11. Contract or Grant No.	
9. Performing Organization Name and Address <b>Lewis Research Center National Aeronautics and Space Administration Cleveland, Ohio 44135</b>		13. Type of Report and Period Covered <b>Technical Memorandum</b>	
		14. Sponsoring Agency Code	
12. Sponsoring Agency Name and Address <b>National Aeronautics and Space Administration Washington, D.C. 20546</b>		15. Supplementary Notes	
16. Abstract <p><b>Thermal barrier ceramic coatings of stabilized zirconia over a bond coat of Ni-Cr-Al-Y were tested for durability on air-cooled turbine rotor blades in a research turbojet engine. Zirconia stabilized with either yttria, magnesia, or calcia was investigated. On the basis of durability and processing cost, the yttria stabilized zirconia was considered the best of the three coatings investigated.</b></p>			
17. Key Words (Suggested by Author(s)) <b>Jet engines Ceramics</b>		18. Distribution Statement <b>Unclassified - unlimited STAR Category 07</b>	
19. Security Classif. (of this report) <b>Unclassified</b>	20. Security Classif. (of this page) <b>Unclassified</b>	21. No. of Pages <b>16</b>	22. Price* <b>\$3.50</b>

\* For sale by the National Technical Information Service, Springfield, Virginia 22161

# DURABILITY OF ZIRCONIA THERMAL-BARRIER CERAMIC COATINGS ON AIR-COOLED TURBINE BLADES IN CYCLIC JET ENGINE OPERATION

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## SUMMARY

Thermal barrier ceramic coatings of stabilized zirconia over a bond coat of a nickel-chromium-aluminum-yttrium alloy were tested for durability on air-cooled turbine rotor blades in a research turbojet engine under cyclic operating conditions. Zirconia stabilized with either yttria, magnesia, or calcia was investigated.

The coated blades were in very good condition after 500 2-minute cycles of testing between full power and flameout. At full power the turbine-inlet temperature, pressure, and rotor speed were 1640 K, 304 kilopascals (3 atm), and 8300 rpm, respectively. The blade metal temperature, coating surface temperature, and temperature drop through the coating were as high as 1200, 1350, and 135 K, respectively. At flameout the values of turbine-inlet temperature, pressure, and speed were about 1000 K, 101 kilopascals (1 atm), and 3300 rpm. The blade average metal wall temperature was about 800 K.

On the basis of durability and processing cost, the yttria stabilized zirconia coating was considered the best of the three coatings investigated.

## INTRODUCTION

Stabilized, plasma sprayed zirconia is a promising thermal barrier between the hot gas and cooled jet engine parts. The analysis and initial tests of reference 1 showed that the use of these ceramic coatings can reduce metal temperatures and coolant flow requirements or increase allowable turbine-inlet temperatures. The tests of reference 1 also demonstrated the durability of a coating of calcia stabilized zirconia over a bond coat of Nichrome. This coating satisfactorily completed 150 hours of steady-state engine operation at a turbine-inlet gas temperature and pressure of about 1640 K and 304 kilopascals (3 atm). And it withstood 35 engine start-stop cycles. The use of only one stabilizer for the ceramic and the limited cyclic testing suggested that more research was needed.

The purpose of this study, then, was to evaluate the durability of several stabilized zirconia coatings by subjecting them to the conditions of cyclic engine operation. Blades of an air-cooled turbine wheel were coated with zirconia stabilized with either yttria, magnesia, or calcia over a bond coat of a nickel-chromium-aluminum-yttrium alloy (Ni-16Cr-6Al-0.5Y) hereinafter called Ni-Cr-Al-Y. The blades were tested in a re-search turbojet engine which was cycled between full power and flameout. The 2-minute cycles produced maximum and minimum temperatures of 1640 and 1000 K, maximum and minimum speeds of 8300 and 3300 rpm, and maximum and minimum pressures of 304 and 101 kilopascals (3 and 1 atm). The cooling-air flow was set to limit the average blade metal wall temperature to 1200 K.

The results of the coating durability tests are presented in terms of the coating condition as determined by visual and metallographic examinations. The results also include current relative material and processing costs and the selection of the most desirable coating.

## APPARATUS AND PROCEDURE

### Test Blades

The turbine blade walls were made of cast B-1900 (ref. 2) and had a commercially applied aluminide anti-erosion-corrosion coating. This coating diffused about 0.010 centimeter into the blade wall. All but six of the blades used in the present cyclic tests had previously been operated in the engine for 200 to 500 hours and were well oxidized. About 10 percent of them were dented at the leading edge tip because of foreign object damage. These used blades were then given the thermal barrier coating described in the next section. Figure 1 is a photograph of a thermal barrier coated blade, and figure 2 shows a cross section of such a blade. The thermal barrier coatings were applied to 83 blades. Further details of the blade are given in reference 3.

### Coating Description and Application Process

Each of the three thermal barrier coating composites investigated consisted of a bond coat of Ni-Cr-Al-Y of a thickness of  $0.010 \pm 0.005$  centimeter, covered with either yttria, magnesia, or calcia stabilized zirconia applied to a thickness of  $0.038 \pm 0.008$  centimeter. Before the bond coating, all airfoil surfaces and base platforms were first grit blasted with commercial, pure (white) alumina. Use of the white alumina minimized contamination that might occur with less pure grit. The inlet air supply pressure to the equipment was 700 kilopascals. Grit blasting with impingement nearly normal to the surfaces cleaned and roughened the surfaces by removing about 0.0013 centimeter of oxidized aluminide coating. The alumina grit size was 250 micrometers, and the sur-

face roughness after grit blasting was measured with a surface roughness meter as about 6 micrometers, rms.

Within 30 minutes after grit blasting, a bond coat of Ni-Cr-Al-Y was plasma sprayed onto the blade wall to a roughness of about 5 micrometers, rms. The particle size of the Ni-Cr-Al-Y powder fed into the plasma spray gun was 74 to 44 micrometers.

Within 30 minutes after bond coat application, stabilized zirconia was plasma sprayed over the bond coat. Thirty-one blades were prepared with nominal 12 weight percent of yttria stabilized zirconia, 13 with 3.2 weight percent of magnesia stabilized zirconia, and 39 with 5 weight percent calcia stabilized zirconia. The yttria and magnesia stabilized zirconia particle size was 74 to 44 micrometers and the calcia stabilized zirconia powder size was 105 to 10 micrometers. The roughness of the applied ceramic coatings was 8 to 10 micrometers, rms. The substrate temperature did not exceed 420 K during the plasma spray operations.

The bond and ceramic coatings were built up to the desired thickness by a succession of spray passes in the spanwise and chordwise directions on the airfoils. The coatings were first applied to the blade leading edge, then to the trailing edge, and finally to the pressure and suction surfaces. In this way overlapping coating seams were joined on the flatter surfaces. This was important because furnace tests of the coating have shown that seams along small radii such as the leading and trailing edges can lead to coating cracking.

The coated surface area on each blade was 110 square centimeters. The coating thickness was measured during the coating process by checking the overall thickness of the airfoil at points at the midspan and midchord. The measurements were made with micrometer calipers. The powder needed to coat a blade was 113 grams for the yttria stabilized zirconia, 255 grams for the magnesia stabilized zirconia, and 56 grams for the calcia stabilized zirconia. The plasma spray gun was held nearly perpendicular to the surface at distances of 15 and 10 centimeters for bond and ceramic coat applications, respectively. The processing time for a blade with yttria, magnesia, or calcia stabilized zirconia was about 20, 35, or 15 minutes, respectively.

### Coating Equipment

Commercial grit blasting equipment was used to clean and roughen the blade surfaces. A hand held plasma spray gun such as that described in reference 4 was used to apply powders of bond and ceramic materials. In the gun an electric arc is contained within a water cooled nozzle. Argon gas passes through the arc and is excited to temperatures of about 17 000 K. The bond and ceramic powders were mechanically fed into the nozzle and were almost instantaneously melted.

## Test Equipment and Procedure

An existing research turbojet engine modified to investigate air-cooled turbine blade configurations was used to evaluate the durability of the coating. The turbine wheel held 74 coated blades and 2 uncoated blades. The turbine wheel diameter was 81.8 centimeters, and the blade was 10.5 centimeters long. Instrumentation was provided for measurements of turbine-inlet gas temperature and pressure, fuel-air ratio, blade cooling-air inlet temperature and flow rate, blade metal wall temperature, and trailing-edge ceramic coating temperature. Three of the coated blades were instrumented with Chromel-Alumel thermocouples at the midspan as shown in figure 2. The thermocouples were mounted in radial grooves in the metal to measure the average metal wall temperature. Details of the thermocouple installation are described in reference 5. The gas-side ceramic coating temperature of the rotating blades at the midspan trailing edge region were measured with an infrared pyrometer of the type described in reference 6. This pyrometer can measure local blade gas-side surface temperatures to an accuracy of about 2 percent. Two uncoated blades instrumented with thermocouples were used to check pyrometer accuracy.

Figure 3 presents a schematic of the system used to control and provide the desired cyclic test conditions. This was accomplished primarily by controlling the combustor fuel (ASTM A-1) supply. Adjustments were made for this control system so that maximum turbine-inlet temperature and pressure were maintained at about 1640 K and 304 kilopascals (3 atm). This maximum temperature and pressure condition will be called "full power." At full power, the rotor speed was 8300 rpm. After about 70 seconds at full power, the turbine-inlet temperature, speed, and pressure were reduced to about 1000 K, 3300 rpm, and 101 kilopascals (1 atm). These reductions were made in about 20 seconds by reducing and then shutting off the fuel supply. This condition is designated "flameout." When fuel was supplied and the engine reignited, the engine reached full power in about 30 seconds. The cooling-air flow was adjusted to limit the leading- and trailing-edge metal wall temperatures to 1200 K at full power. The coolant-to-gas-flow ratio at full power was about 1.9 percent, and the cooling-air temperature was 420 K. During flameout the metal wall temperatures reached about 800 K. The transient and steady-state values of the turbine-inlet temperature and speed during one of the cycles are shown in figure 4. These values were repeatable within 20 K and 200 rpm throughout the 500 cycles of testing. The blade metal wall temperatures and the leading and trailing edges during cyclic operation are presented in figure 5. Other details of the procedure for automatic cycling and fuel flow control are described in reference 7.

A total of 500 2-minute cycles were run. The engine was stopped for visual inspection of the coating at 100, 300, and 500 cycles. Seventy-four blades were tested at a time. After 100 cycles eight blades were removed from the wheel for detailed examination and reference purposes. After termination of tests at 500 cycles, one of each of the three

types of stabilized coated blades that had been run for the full duration of the tests was sectioned, and the coating and blade microstructure was examined at the midspan and leading edge region with light optical photomicrographs at a magnification of 150. The microstructure of one untested yttria stabilized zirconia coated blade was also examined. The ceramic coating thickness and roughness were also obtained on one of each of the three types of stabilized coated blades that had been tested for 500 cycles. The ceramic coating thickness was measured after it was purposely spalled from the blade metal wall. This blade was heated in a furnace to 1600 K, well above the allowable bond-ceramic interface temperature (ref. 1) and metal melting temperature (ref. 2). Then, this blade was instantly cooled by plunging it into a 300 K water bath.

## RESULTS AND DISCUSSION

### Durability

Visual inspection of the coating after 100 cycles of testing in the research turbojet engine showed that about 90 percent of the blades had a metallic colored scuff mark at the tip of the leading edge. Also about 40 percent of the blades had a 1-square-centimeter chipped area of ceramic in the vicinity of the scuff mark. The cause of the chipping was the impingement of metallic pieces of thermocouple probes that had broken during engine transient overheating and excessive vibration. (During accidental overheating the turbine-inlet temperature and coated blade metal wall temperature instantaneously reached 1900 and 1300 K, respectively.) About half of the yttria and half of the calcia stabilized zirconia coated blades were chipped. But only one of the 13 magnesia stabilized zirconia coatings showed this damage. The reason for this apparent greater resistance to chipping (foreign object damage) is not known.

The inspection after 100 cycles showed that the yttria stabilized zirconia was completely removed from three blades. The cause for this was the procedure in processing the first group of 10 blades. The roughening and cleaning procedure was probably not adequate for the hard, dented, and oxidized surfaces. Also during application of the bond coat to the first group of blades, particles spurted intermittently from the plasma gun. This anomaly could also have contributed to the poor coating adherence. The blades processed after the first group were more thoroughly cleaned and roughened; fresh, clean grit was used and more attention was given to grit impingement normal to the surface and keeping the air pressure at or above 700 kilopascals. Better control was also maintained on the performance of the plasma spray feed apparatus.

At the end of 100 cycles, eight blades (including the three blades in the first group discussed above) were removed from the wheel and replaced by blades with calcia stabilized zirconia coatings processed using the more consistent and carefully controlled

coating procedure. Cyclic testing was then continued for another 200 cycles. Inspection with the unaided eye disclosed no change in coating appearance. The tests were then continued for another 200 cycles and then terminated. The thermal barrier coatings on 66 blades (24 with yttria, 12 with magnesia, and 30 with calcia stabilized zirconia) completed 500, 2-minute cycles between full power and flameout without external evidence of deterioration except for the foreign object damage incurred during the first 100 cycles. The other eight calcia stabilized zirconia coatings completed 400 cycles of testing.

A trailing edge view of the rotor assembly of the coated blades after conclusion of the tests is shown in figure 6. The two uncoated blades in the figure were used as reference blades for infrared measurements (ref. 6) of ceramic coating trailing-edge surface temperatures. The black spots on the blade tips in figure 6 are soot deposits that occurred during engine shutdown. The black lines along the span near the root on the suction surface were also caused by soot deposition.

An overall view of the coated surface of a typical blade after completion of 500 cycles is shown in figure 7. The soot was burned from this blade after it had been removed from the wheel but rust particles deposited during the test still remain.

Despite foreign object damage, which caused chipping at the blade leading edges, the coatings were in very good condition. The chipped areas did not deteriorate further, and the exposed bond coat in the chipped regions remained intact for at least 400 cycles of testing. Ceramic coating roughness measurements showed no roughness change during the tests. In actual usage the roughness should be reduced by polishing the ceramic to a roughness of about 3 micrometers rms (ref. 8). This will improve the durability and aerodynamic performance.

### Coating and Metal Wall Temperatures

During the full power portion of the cyclic tests (fig. 4) the highest surface temperatures and temperature drop through the coating occurred at the blade leading edges. Calculations based on the method of reference 1 predicted a leading-edge surface temperature of about 1350 K, a temperature drop through the ceramic coating of 135 K, and an average metal wall temperature of 1204 K. The measured average metal wall temperature was between 1180 and 1200 K. The measured coating surface temperature and average metal wall temperatures at the trailing edge were about 1225 and 1200 K, respectively. The average metal temperatures measured on the blade pressure and suction surfaces (fig. 2) ranged between 900 to 1100 K. All measured blade metal temperatures were repeatable within 50 K during the 500 cycles of testing.

At flameout conditions the measured metal temperatures of the coated blade ranged from 700 to 850 K. No ceramic coating temperatures were measured because the tem-



peratures were below the range of the pyrometer.

### Coating Microstructure

The microstructure of the bond coat and ceramic was metallographically examined on several blades at the leading-edge region where durability problems are most likely to occur. A cross section of an yttria stabilized zirconia composite which was not engine tested is shown in the light optical photomicrograph of figure 8(a). This type of microstructure was encountered in all three of the ceramic coatings investigated. This microstructure corresponds to that of calcia and yttria stabilized zirconia coatings used for thermal insulation on rocket nozzles (ref. 9). The ceramic consists of solid material connected with a network of fine voids interspersed with larger voids. The porosity gives the coating a lower thermal conductivity and a higher reflectivity than solid zirconia.

Figure 8(b) is a photomicrograph of an yttria stabilized zirconia composite on a blade after it was tested for 500 engine cycles. From these photomicrographs it is apparent that the aluminide coat that was originally present on all of the blades was not removed by grit blasting. The Ni-Cr-Al-Y bond appears to have adhered well to this aluminide coat. The Ni-Cr-Al-Y, however, is not uniform in thickness. Furthermore, measurements of the thicknesses of the Ni-Cr-Al-Y and stabilized zirconia indicated variations as large as 0.005 and 0.008 centimeter, respectively. Automated deposition procedures could provide more uniform coating thicknesses.

Figure 9(a) shows a typical photomicrograph of a calcia stabilized zirconia composite after 500 cycles of testing. The microstructure is very similar to that of the yttria stabilized zirconia. Cracks in the calcia stabilized zirconia coating were observed on some of the micrographs (fig. 9(b)). These cracks generally were located parallel and adjacent to the bond coat. In some cases these cracks penetrated to the outer surface of the coating. The formation of such cracks shown in figure 9(b) can weaken the coating adherence. These cracks, however, did not cause spalling of the coating during testing. Although not shown, the microstructure of the magnesia stabilized zirconia composite was similar to the other composites, and the Ni-Cr-Al-Y bond coat adhered well to the aluminide coat.

### Other Considerations

Of the three ceramic coatings studied, control of the coating thickness during deposition was most difficult with the magnesia stabilized zirconia. Also more of the magnesia stabilized zirconia powder was used to coat a blade to the desired thickness: two

times more than the yttria stabilized powder and 4.5 times more than the calcia stabilized powder. The total processing time for a yttria or calcia stabilized zirconia coated blade was about 20 and 15 minutes, respectively. In contrast, the time for a magnesia stabilized coated blade was about 35 minutes. In addition, the current cost of the magnesia and yttria stabilized zirconia powders is about twice that of the calcia stabilized zirconia. The cost per blade for the magnesia is therefore highest of the three. Based on these considerations and the results of the cyclic tests, which produced microscopic cracks in the calcia stabilized coating, the yttria stabilized zirconia is the most desirable.

### SUMMARY OF RESULTS

The following are the results of durability tests made on thermal barrier coatings of stabilized zirconia in turbojet engine cyclic operation.

1. The coatings on 66 blades (24 with yttria, 12 with magnesia, and 30 with calcia stabilized zirconia) completed 500, 2-minute cycles between full power and flameout in a research turbojet engine. The measured metal temperatures at full power conditions were as high as 1200 K, and the coating surface temperature was 1350 K. The temperature drop through the coating was calculated as high as 135 K, respectively.

2. After 100 cycles of operation three blades with yttria stabilized zirconia were removed from the engine because the ceramic coating came off the surface. This coating failure was traced to initial coating application difficulties. Another five blades were removed for detailed examination and reference purposes. These eight blades were replaced with calcia stabilized zirconia coated blades which then successfully completed 400 cycles of testing.

3. Metallographic investigation of the coatings showed that the Ni-Cr-Al-Y bond adhered well to the metal surfaces. However, microscopic cracks were detected in the calcia stabilized zirconia coating. These cracks did not cause the coating to spall.

4. Control of the deposition of the magnesia stabilized coating was more difficult, and the current cost of processing a blade is higher than for the other coatings.

5. On the basis of durability and processing cost, the yttria stabilized coating was considered the best of the three coatings investigated.

Lewis Research Center,

National Aeronautics and Space Administration,

Cleveland, Ohio, May 27, 1976,

505-04.

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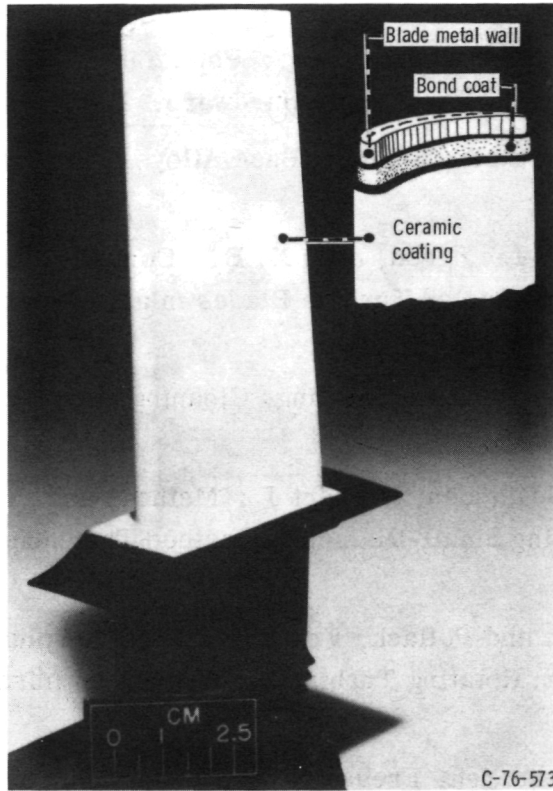


Figure 1. - Ceramic coated turbine blade.

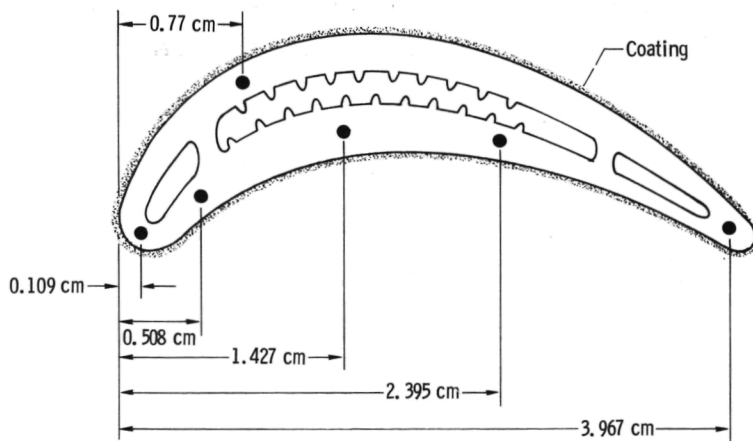


Figure 2. - Test blade midspan cross section and composite of thermocouple locations on three coated blades.

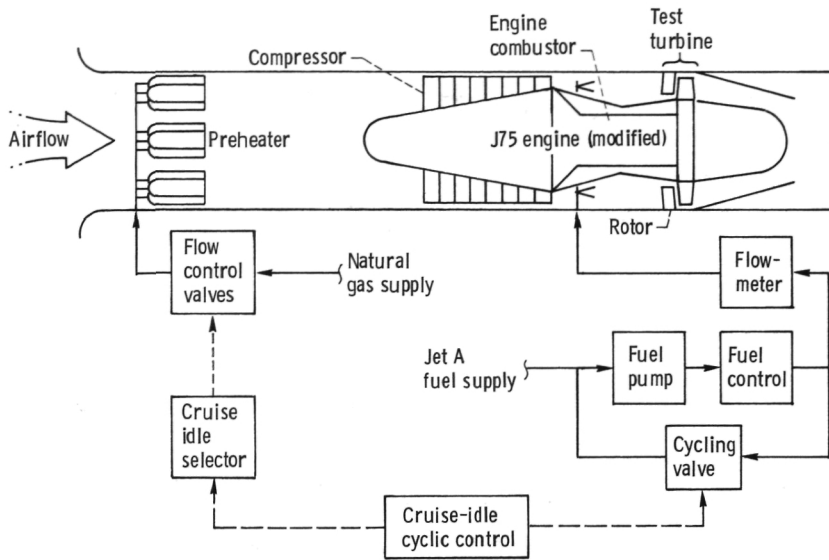


Figure 3. - Research engine-preheater fuel systems.

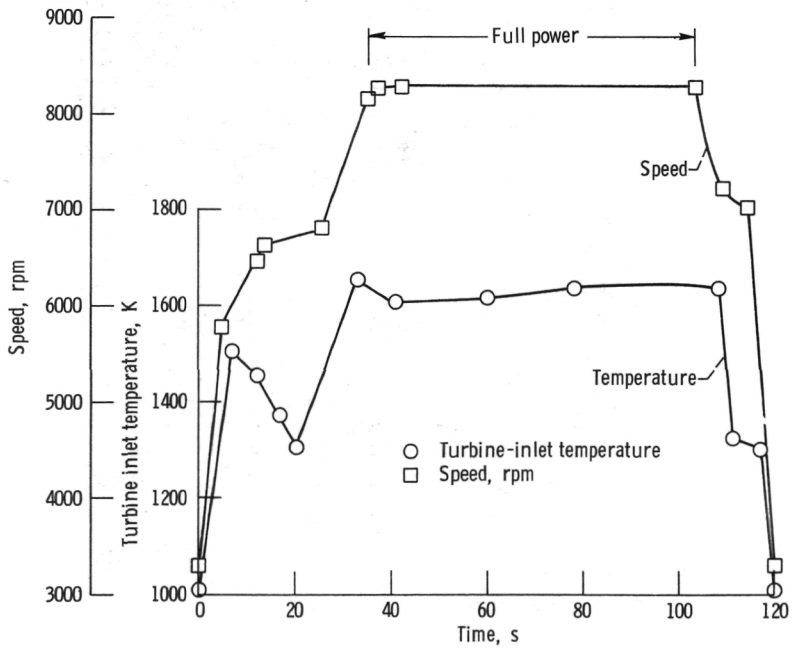


Figure 4. - Turbine-inlet temperature and speed during cyclic tests.

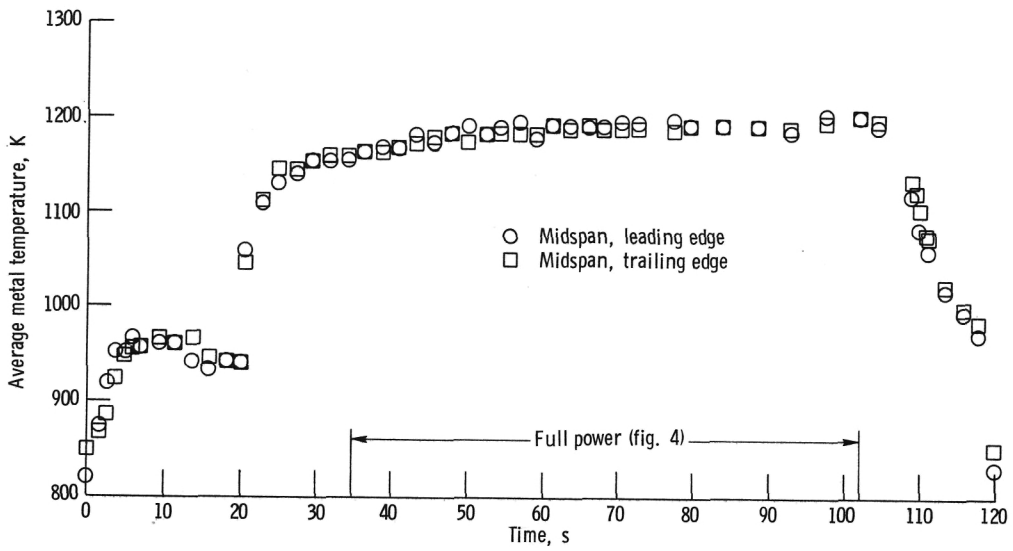


Figure 5. - Typical coated blade metal temperatures during cyclic tests.

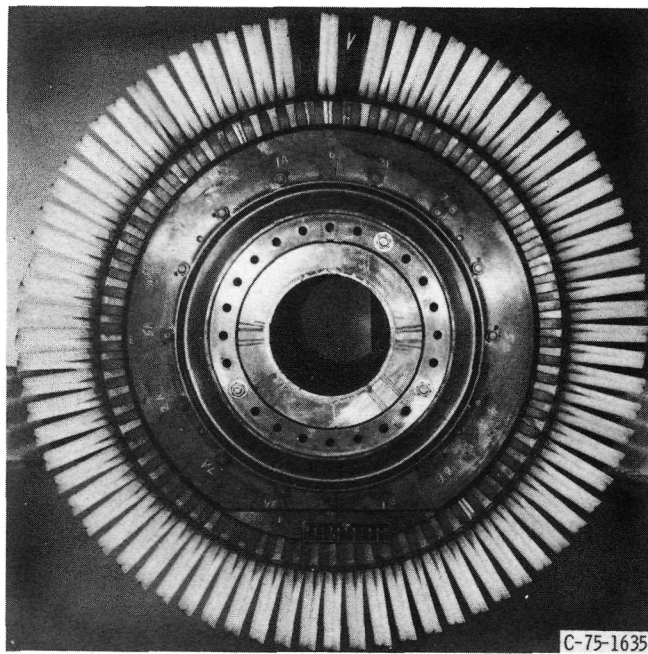
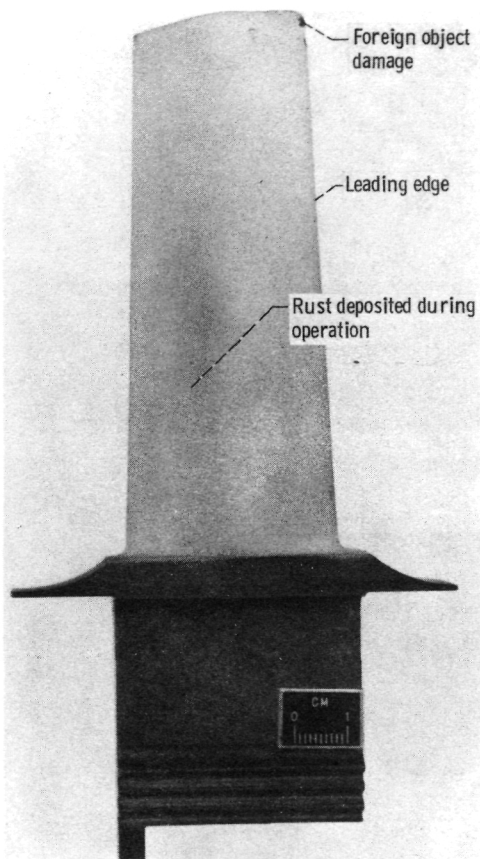
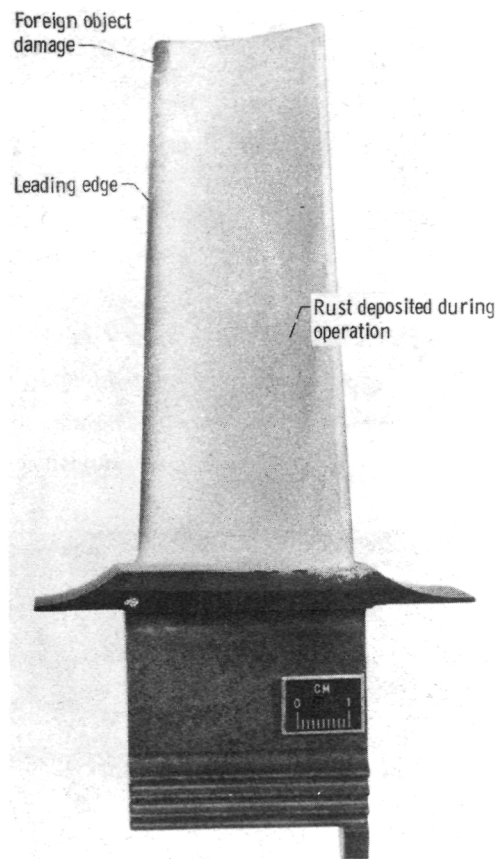


Figure 6. - Ceramic coated turbine blades after 500 cycles of testing.

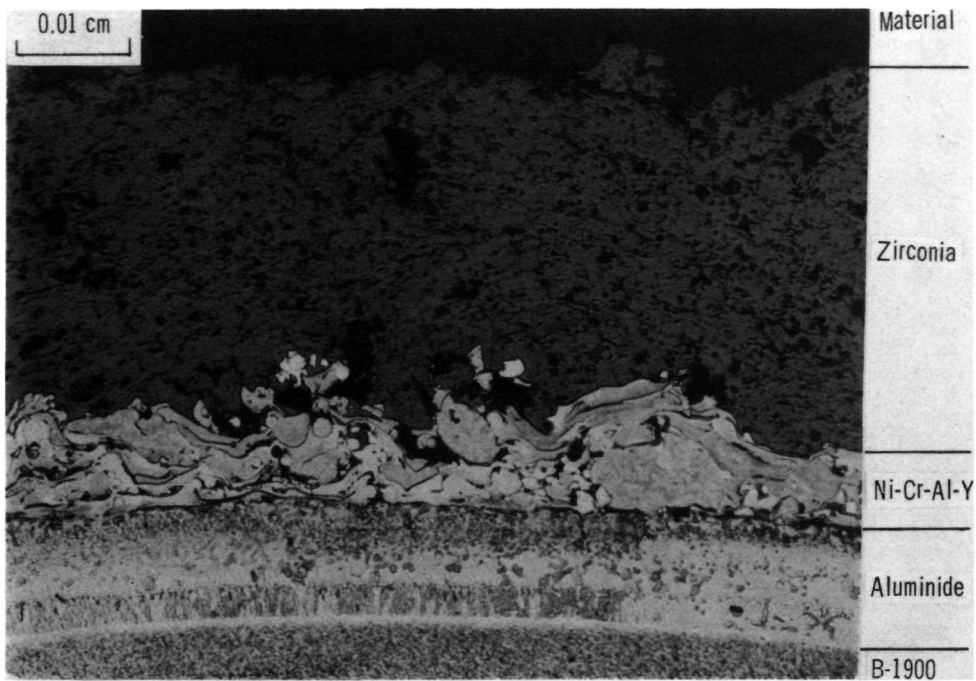


(a) Suction surface.

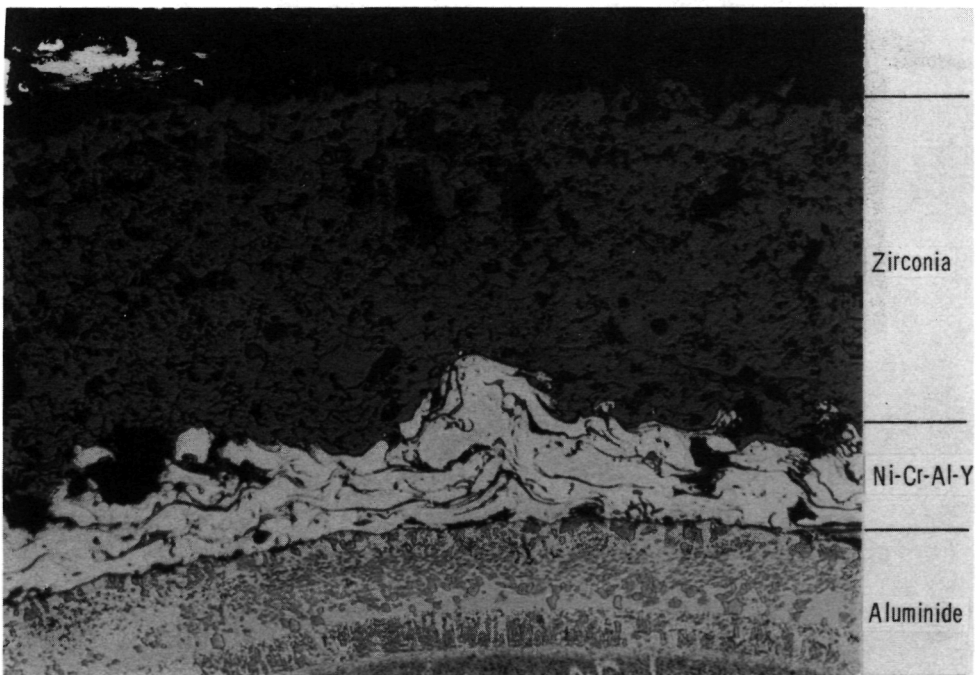


(b) Pressure surface.

Figure 7. - Ceramic-coated blade after testing in engine.



(a) Before cyclic tests.



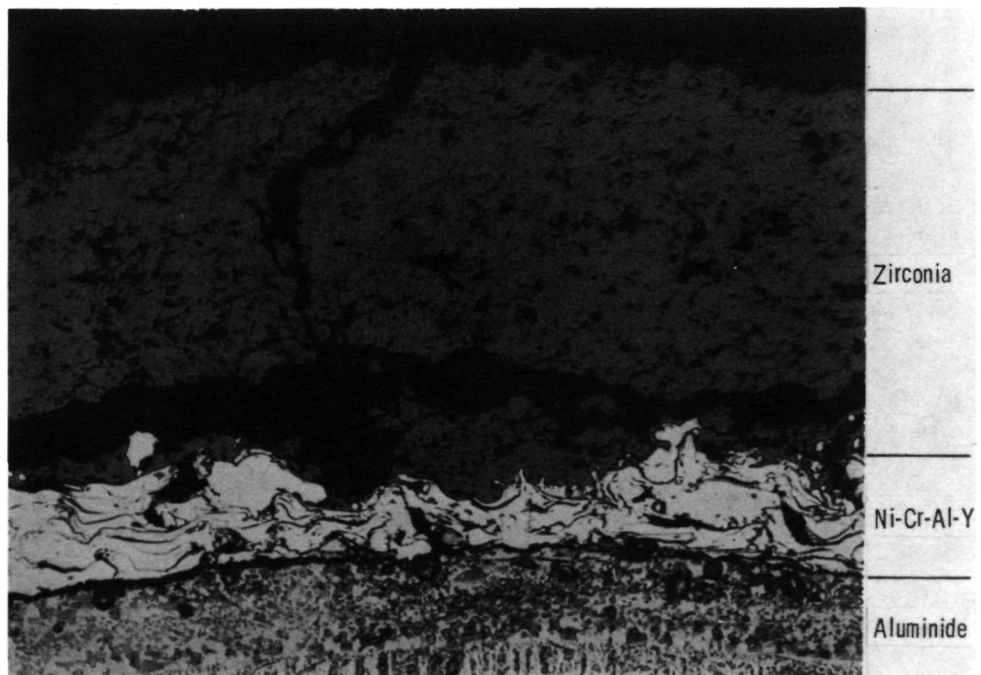
(b) After cyclic tests.

Figure 8. - Microstructure of yttria stabilized zirconia on turbine blade leading-edge at midspan.





(a) Uncracked.



(b) Cracked.

Figure 9. - Microstructure of calcia stabilized zirconia on turbine blade leading-edge at midspan.



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