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# Performance of Laser Glazed ZrO<sub>2</sub> TBCs in Cyclic Oxidation and Corrosion Burner Rig Tests

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PERFORMANCE OF LASER GLAZED  $ZrO_2$  TBCs  
IN CYCLIC OXIDATION AND CORROSION BURNER RIG TESTS

by

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ABSTRACT

E-1195

The performance of laser glazed zirconia (containing 8 w/o  $Y_2O_3$ ) TBCs was evaluated in cyclic oxidation and cyclic corrosion tests. Plasma sprayed zirconia coatings of two thicknesses (0.02 and 0.04 cm) were partially melted with a  $CO_2$  laser. The power density of the focused laser beam was varied from 35 to 75  $W/mm^2$ , while the scanning speed was about 80 cm per minute. In cyclic oxidation tests, the specimens were heated in a burner rig for 6 minutes and cooled for 3 minutes. The results obtained indicated that the laser treated samples had the same life as the untreated ones. However, in corrosion tests, in which the burner rig flame contained 100 PPM sodium fuel equivalent, the laser treated samples exhibited nearly a fourfold life improvement over that of the reference samples. In both tests, the lives of the samples varied inversely with the thickness of the laser melted layer of zirconia.

INTRODUCTION

The development and the potential benefits of ceramic thermal barrier coatings (TBCs) for gas turbine high temperature components have been well documented in the technical literature. Some of the key early work from the 1950's to early 1970's was conducted at NASA Lewis Research Center (refs. 1 to 6). In the mid-1970's Stecura and Liebert (refs. 7-9) developed a successful zirconia

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based TBC. This work stimulated further development and testing of TBCs for potential aircraft and non-aircraft applications (ref. 10).

The duplex TBCs identified by NASA consist of an inner layer of oxidation resistant plasma sprayed NiCrAlY bond coat and an outer, plasma sprayed yttria-stabilized zirconia layer. The bond coat is about 0.01 cm thick while the ceramic layer can vary in thickness from 0.01 cm to 0.04 cm or more.

Although TBCs performed well in the clean combustion gases resulting from burning Jet A fuel or natural gas, preliminary studies (refs. 11 to 15) revealed that their durability was reduced when they were tested in combustion gases containing gaseous impurities such as sodium, vanadium, sulfur, etc. in general. The failures were observed to occur in the ceramic layer in a manner similar to TBC failures in clean fuel tests (refs. 13 and 16). The cracks initiated in the ceramic layer parallel and close to the ceramic/bond coat interface. This is in agreement with the findings of Levine (ref. 17) who measured the tensile strength of  $ZrO_2 - 12Y_2O_3/NiCrAlY$  TBCs and determined that this was weakest part of the coating system. The failures of TBC's tested in combustion gases doped with sodium and vanadium were correlated by Miller (ref. 18) in terms of dew points (ref. 19) melting points of the condensates and the temperature distribution within the coatings. Palko (ref. 15) observed that liquid  $Na_2SO_4$  was absorbed into the open porosity of the ceramic coating and speculated that the difference in thermal expansion between the coating and solidified salt was the cause of spallation. To alleviate the problems caused by dirty fuels, two approaches were undertaken. The first approach (ref. 13) was to develop new thermal barrier coating systems, e.g. calcium silicate, that would resist corrosive environments. The

second approach is represented by the present work. The idea behind this approach is to improve the performance of zirconia-based TBCs through partial densification of the ceramic layer by treatment with a laser. Densification of the surface of a ceramic coating was expected to reduce the coating penetration by salts contained in the combustion gases, and their attack on the bond coat. To test this idea, two separate experiments were performed. One experiment was a cyclic oxidation test of laser glazed TBCs in a Mach 0.3 burner rig fired with a Jet A fuel to determine the effect of glazing on coating performance in a clean environment. The other experiment was a cyclic corrosion test in a similar burner rig fired with Jet A fuel, but with the combustion gases doped with sodium to determine if glazing reduced penetration of  $\text{Na}_2\text{SO}_4$  into the ceramic coat. The laser glazing approach is not unique in the sense that the laser surface fusion technique was used by others (refs. 20, 21, 22) to "segment" ceramic coatings and ceramic turbine shrouds to increase strain tolerance.

## EXPERIMENTAL PROCEDURE

### Materials

The composition of the NiCrAlZr powder used to form the bond coat is listed in Table 1. The same table shows the composition of yttria-stabilized zirconia powder. The particle size of both powders was in the range between 200 and 325 mesh. Waspalloy tubing (1.27 cm diameter and 0.124 cm wall thickness) was used as the substrate material for the cyclic oxidation tests. Its nominal composition is shown in the third column of Table 1. Hollow MAR M-509 erosion bars, cast to the configuration described by Hodge et al. (ref. 13) was used as the substrate for the cyclic corrosion test. The composition of the alloy is shown in the column 4 of Table 1.

## Coating Applications

The Waspalloy tubing was cut into 18 cm long pieces. Each piece was provided with fittings to permit internal cooling during plasma spraying. The procedure described in Reference 8 was used in sample preparation and plasma spraying. The coatings were manually sprayed in air to nominal thicknesses of 0.012 cm for the bond coat, 0.020 and 0.040 cm for the ceramic coat. The thickness of the bond coat varied from the nominal value by about  $\pm 20$  percent in extreme cases and of the ceramic coat by  $\pm 15$  percent. After plasma spraying, each tube was cut to about 9 cm lengths, appropriate for burner rig testing. The hollow erosion bars were plasma sprayed in the same manner as oxidation samples but without internal air cooling. The nominal thicknesses for bond coat and ceramic coats were as before 0.012, 0.020, and 0.040 cm. For any single erosion bar, the bond coat thickness varied by as much as  $\pm 40$  percent and thickness of the ceramic coat by  $\pm 25$  percent from the nominal values. These significant variations in thickness were due to the difficulty of manually spraying and in measuring a body without cylindrical symmetry.

## Laser Glazing

A  $\text{CO}_2$  laser was used in this investigation to melt the surface of the ceramic coatings. The laser beam was focused with a ZnSe (zinc selenide) lens (focal distance 30.5 cm) to cover a circular area of approximately 0.11 cm in diameter. Preliminary experimentation had shown that the laser beam with a power density of  $50\text{W}/\text{mm}^2$  could melt plasma sprayed zirconia to a depth of 0.01 cm at a scanning speed of about 80 cm per minute. Burner rig samples were glazed in an apparatus that allowed controlled rotation of the specimen about its axis and controlled translation along the same axis. By adjusting the speed of rotation and translation, the cylindrical surface of the sample

could be scanned in spiral fashion. Allowance was made for 50 percent overlap of the scans. Figure 1 shows the surface of a zirconia coating laser glazed with a  $50 \text{ W/mm}^2$  beam. The small area on the left side shows the as-plasma sprayed condition. The glazed surface is smooth and shiny. Thus glazing offers an aerodynamic benefit. Furthermore the laser glazed surface is segmented. Figure 2 shows the surface of a glazed zirconia coat on the flat portion of a corrosion sample at higher magnification. In evidence are the striations and the segmentation or mud-flat cracking produced by the laser melting. The average diameter of the segments is about 0.05 cm. The width of the bands represents one half of the laser beam diameter. Due to lack of cylindrical symmetry, the scanning speed for the corrosion samples was not constant. The average speed was about 85 cm per minute. Laser beams with power density of 35, 50, and  $75 \text{ W/mm}^2$  were used.

#### Burner Rig Testing

The Mach 0.3 burner rig used in these tests is shown in Figure 3 and has been described in reference 23. Eight specimens were placed in a holder which was rotated at 630 rpm in front of the burner nozzle. The same type of burner was used for cyclic oxidation and cyclic corrosion tests. In the cyclic oxidation test, the samples were heated for six minutes and then cooled with a stream of compressed air for three minutes. The burner rig was fired at a fuel-to-air ration of about 0.049 using Jet A fuel. The test temperature was  $1050 \pm 20^\circ \text{ C}$  as determined with a calibrated (for emissivity and window absorption), disappearing filament optical pyrometer when focusing on the center of the sample surfaces facing the nozzle of the burner. About three minutes in the flame were required for the hot zone of the samples to reach test temperature. At the end of the three minutes of forced air cooling, the temperature was about  $80^\circ \text{ C}$ . All of the TBC failures occurred or apparently

started on the back surfaces of the samples where the temperature was determined to be about  $100^{\circ}$  C higher (ref. 24). In this test, the samples were visually examined and the temperature monitored at least once every day. Samples were tested until TBC failure (cracking and/or spallation) was observed. Figure 4 illustrates typical samples after test. All coatings were run in triplicate. After test, the specimens were photographed and mounted in epoxy for metallographic examination.

The corrosion test was run at a substrate temperature of  $843^{\circ}$  C. The level of sodium, introduced to the combustor as an aqueous solution of NaCl, was 100 PPM in the fuel equivalent. The sulphur content of the Jet A fuel was approximately 0.05 weight percent. The operation and calibration of the equipment was as described in reference 13. The samples were exposed to thermal cycles consisting of 55 minutes at temperature, followed by five minutes out of the flame, with the internal and external cooling air on. The samples were examined for failure after ten cycle intervals. Testing was stopped when TBC showed evidence of spallation. The reference non-glazed samples were run in duplicate and the laser glazed samples in triplicate. After the test, representative samples were photographed, plasma sprayed with copper (to facilitate electron probe analysis) mounted in epoxy and sectioned very slowly through the hot zone with a diamond wheel. No liquid was used during cutting and polishing in order to retain all of the sodium sulfate that penetrated the zirconia coating during test. These cross-sections were subjected to electron microprobe analysis and metallographic examination. Figure 5 illustrates typical corrosion samples after test.

## RESULTS AND DISCUSSION

### A. Cyclic Oxidation Test

The definition of failure in these types of experiments is rather arbitrary. In this investigation, the appearance of a crack or spallation of a small

piece of ceramic coating were the criterion for removal of the sample from test as illustrated in figures 4 and 5. In other investigations (ref. 21) a failure was considered to have occurred when the ceramic coat spalled from 50 percent or more of the test zone. Therefore, it is difficult to compare the results obtained in two different investigations. In this investigation, the failures occurred on the back surface of the samples, where the temperature was about  $100^{\circ}\text{C}$  higher than the test temperature of  $1050^{\circ}\text{C}$ . Figure 6 summarizes the results obtained in the cyclic oxidation test. Two sets of data are shown. One set pertains to a group of samples in which the thickness of the ceramic coating was 0.020 cm and the other set represents samples with 0.040 cm thick ceramic coatings. In the first set, the group of samples glazed with the  $50\text{ W/mm}^2$  laser beam did not perform as well as the group glazed with the  $35\text{ W/mm}^2$  beam. Also in the case of 0.040 cm thick, ceramic coatings the power density of the laser beam, and consequently the thickness of the glazed layer, had small but negative effect on the performance of the TBC. There is a slight improvement in TBC life with increasing thickness of the ceramic coat. Expressing these observations in statistical terms one can say that within the 90% confidence level, laser power density has negative effect and that within the 95% confidence level. The thickness of  $\text{ZrO}_2$  coating has a positive effect on life of TBC's. The interaction term was found to be positive and significant at 90% confidence level. That means with lower thickness, the negative effect of power density is more pronounced. Examination of the figure 7, which shows the microstructure of a representative sample, reveals that the mode of failure was cracking in the ceramic coat, near and parallel to the bond coat/ceramic coat interface. This type of failure has been observed in all investigations dealing with cyclic oxidation tests of TBCs. Also, one can observe the amount of porosity in the plasma



sprayed ceramic coat and the complete densification of the laser glazed surface layer. The bond coat seems to be partly oxidized. Levine (ref. 25) performed cyclic oxidation tests on similarly prepared samples, which were not laser glazed, and obtained similar results (within 95% confidence level). Therefore, it appears that glazing did not negatively affect the performance of the TBC. In references 20, 21, and 22 it is claimed that a "segmented" ceramic structure, formed by precracking the ceramic in a direction perpendicular to the plane of the coating as a result of laser glazing may significantly increase its strain tolerance. Improved TBC performance, as a result of laser induced segmentation, was not observed in the present study.

#### B. Cyclic Corrosion Test

The experimental results obtained in the cyclic corrosion test are summarized in graphic form in Figure 8. There are two sets of data. One set represents data obtained with 0.020 cm thick ceramic coatings and the other set pertains to 0.040 thick coatings. It is evident that the glazed TBCs performed significantly better than the reference non-glazed ones. At the 50 W/mm<sup>2</sup> power density level the improvement in life is nearly four-fold. Similar improvement can be observed when the thickness of the ceramic coat is doubled. As can be seen in figure 5 the location of the failures is random. Figure 9 shows the microstructure of TBC near the leading edge. The samples failed in the usual manner, that is, cracking occurred in the ceramic coat near and parallel to the bond coat/ceramic coat interface. On several occasions an unusual type of failure was observed namely separation of the bond coat from the substrate. The degree of attack of the bond coat appears to be insignificant. Further examination of this microstructure reveals the loss of thickness of ceramic coating as the glazed layer is nearly gone. Since yttria stabilized zirconia does not react with sodium sulfate (ref. 26) this loss may be attributed to erosion. Spallation was probably not

responsible for this loss since the surface remains smooth. Figures 10 and 11 show electron microprobe traverses of non-glazed and glazed TBCs, respectively. Although determinations for several elements were made, only the traces for Na, S, and O are shown. The non-glazed sample (Fig. 10) which failed in 66 cycles exhibits a significant degree of bond coat oxidation/sulfidation along grain boundaries as revealed by the oxygen and sulfur traces. It appears that every pore in the ceramic contains sodium sulfate. It can be seen that the glazed sample (Fig. 11) is considerably less affected despite the fact that its exposure to the corrosive environment was 5 times longer. The oxygen and sulfur traces in the bond coat indicate less oxidation/sulfidation along the grain boundaries. Also the ceramic coat shows less permeation by sodium sulfate. In both cases there is a concentration of sodium sulfate at the ceramic coat/bond coat interface.

The experimental data obtained in this investigation provide strong evidence, that laser glazing of zirconia based TBCs will improve their life in a corrosive environment as a result of reduced permeability of the surface.

#### CONCLUSIONS

As a result of this preliminary study of the effect of laser glazing on life of zirconia based TBCs in cyclic oxidation and cyclic corrosion tests, the following conclusions have been drawn:

1. The laser glazed zirconia based TBCs show at least a four-fold improvement over non-glazed TBCs in cyclic corrosion tests.
2. Laser glazing of zirconia based TBCs to give an aerodynamically smooth surface has no apparent effect on their life in cyclic oxidation tests. Glazed and non-glazed TBCs endured similar numbers of cycles in a Jet A fuel fired burner rig.
3. The concept of increasing strain tolerance of TBCs by "segmenting" the ceramic layer through exposure to a high intensity heat source such as a laser was not borne out by this study.

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TABLE 1. - CHEMICAL COMPOSITION OF MATERIALS USED IN THIS INVESTIGATION

Element	Content, w/o			
	Bond coat <sup>a</sup>	Zirconia	Waspalloy <sup>a</sup>	MM-509
Al	14	0.011	1.4	ND <sup>b</sup>
B	----	ND	0.006	0.004
C	----	----	0.07	0.56
Ca	----	0.078	----	ND
Co	----	ND	13.5	Balance
Cr	14	ND	----	23.26
Fe	----	0.03	2.0 max	0.32
Hf	----	2.10	----	ND
Mg	----	0.014	----	ND
Mn	----	ND	0.75 max	ND
Mo	----	ND	4.3	ND
Ni	Balance	ND	Balance	10.95
S	----	ND	----	0.011
Si	0.08	0.05	----	<0.10
Ta	----	ND	----	3.66
Ti	----	0.054	3.0	0.30
V	----	ND	----	ND
W	----	ND	----	6.50
Y	----	6.28	----	ND
Zr	0.1	Balance	0.009	0.32

<sup>a</sup>Nominal compositions.

<sup>b</sup>Not determined.

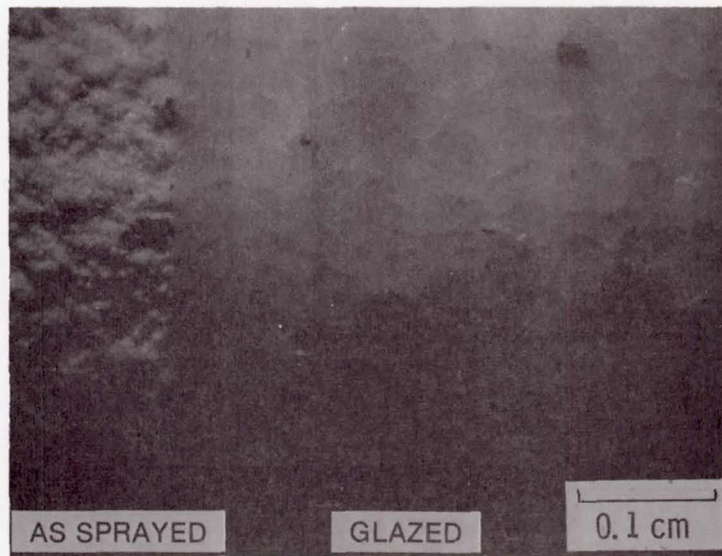


Figure 1.—Photograph of a 0.020 cm thick, laser glazed ceramic coat of a cyclic oxidation sample. Power density: 50 W/mm<sup>2</sup>.

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Figure 2.—Photomicrograph of a 0.020 cm thick, laser glazed ceramic coating. Power density: 35 W/mm<sup>2</sup>.

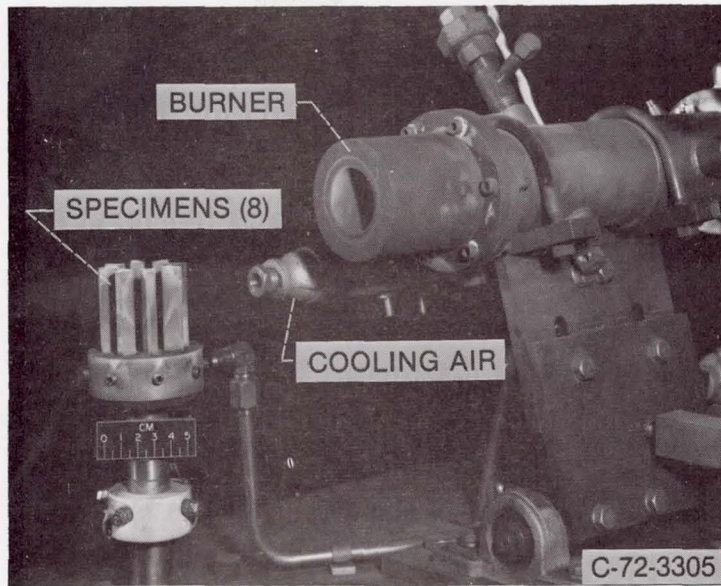


Figure 3.—Mach 0.3 burner rig assembly.

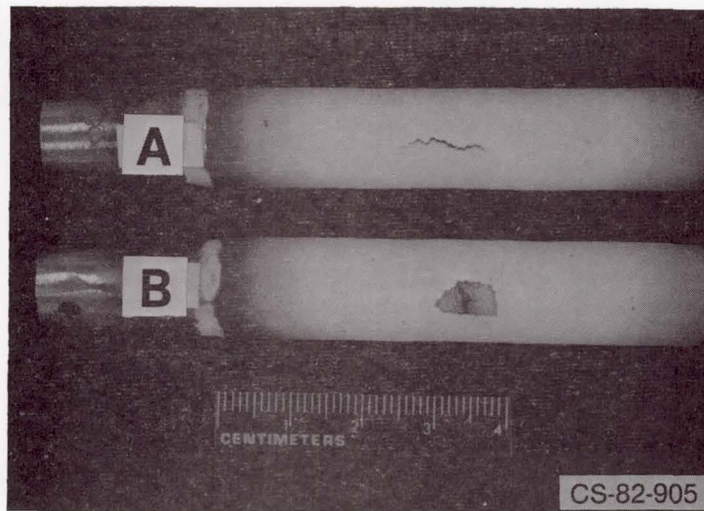


Figure 4.—Photographs of samples with 0.040 cm thick ceramic coat, after cyclic oxidation test at 1050° C (6 min heating, 3 min cooling per cycle).

A. Glazed with 50 W/mm<sup>2</sup> laser beam  
Life: 2800 cycles

B. Glazed with 50 W/mm<sup>2</sup> laser beam  
Life: 2500 cycles.



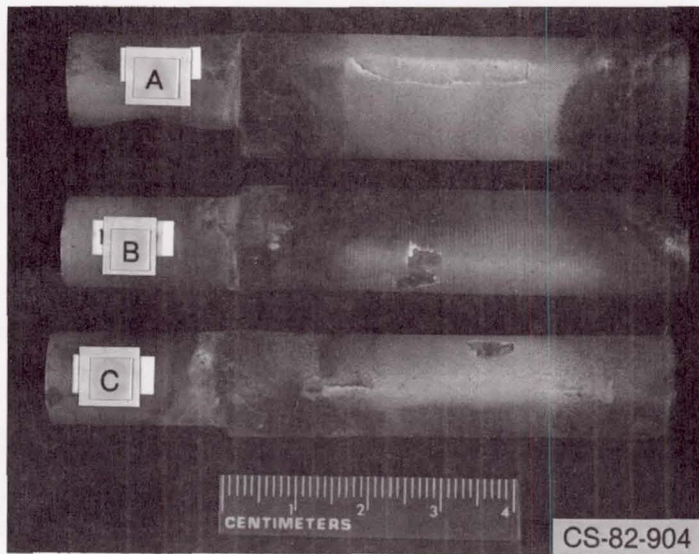


Figure 5.—Photographs of samples with 0.040 cm thick ceramic coat after cyclic corrosion test at 843° C substrate temperature, 100 ppm Na doped Jet A fuel, 55 minutes hot, 5 minutes cooling per cycle.

- A. Non-glazed  
Life: 122 cycles
- B. Glazed with 50 W/mm<sup>2</sup> laser beam  
Life: 355 cycles
- C. Glazed with 35 W/mm<sup>2</sup> laser beam  
Life: 366 cycles

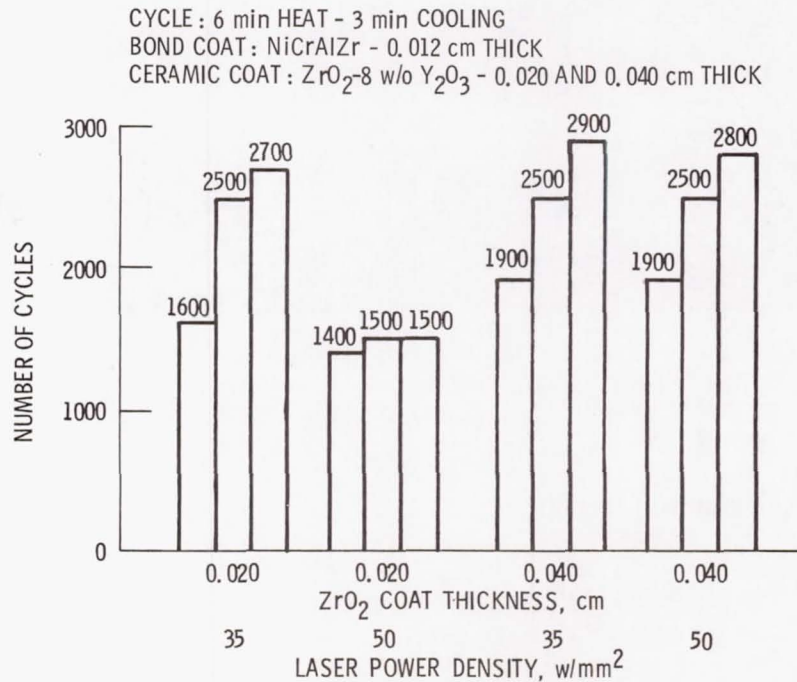


Figure 6. - Life of ZrO<sub>2</sub> TBC's in burner rig cyclic oxidation test at 1050° C.

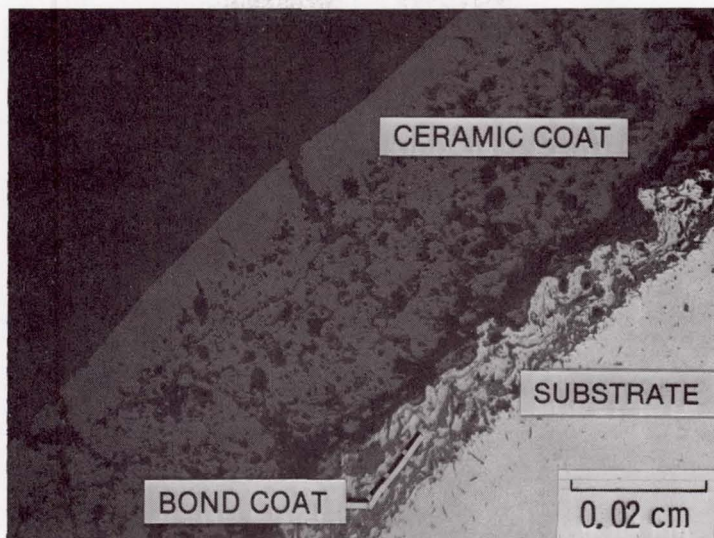


Figure 7.—Microstructure of hot zone of cyclic oxidation sample after 2800 cycles to 1050° C. Ceramic coat thickness: 0.040 cm; Laser beam: 50 W/mm<sup>2</sup>.

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CYCLE: 55 min HEAT - 5 min COOLING  
 SALT CONCENTRATION IN THE FUEL : 100 ppm  
 BOND COAT : NiCrAlZr - 0.012 cm THICK  
 CERAMIC COAT : ZrO<sub>2</sub>-8 w/o Y<sub>2</sub>O<sub>3</sub> - 0.020 AND 0.040 cm THICK

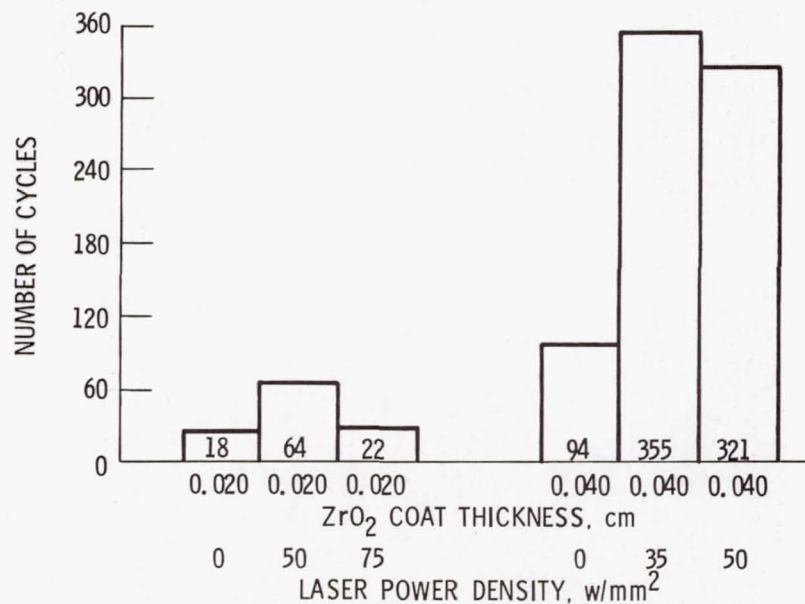


Figure 8. - Life of ZrO<sub>2</sub> TBC's in burner rig corrosion test at 843° C (substrate temperature).

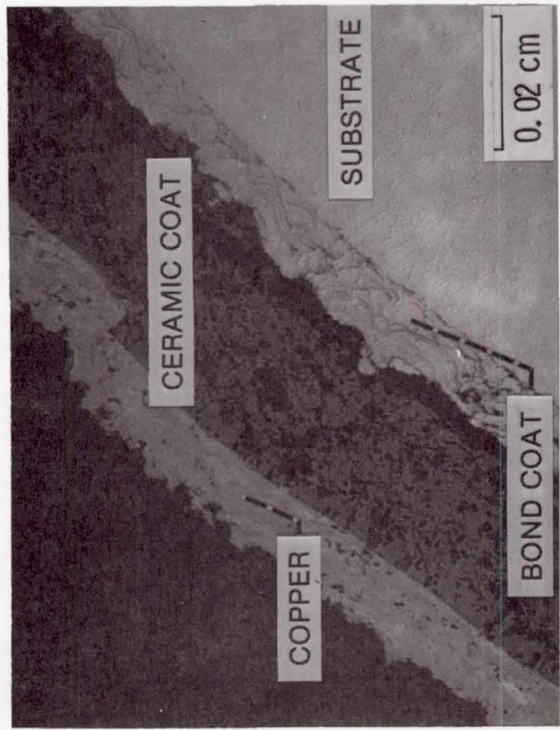


Figure 9.—Microstructure near the leading edge of a corrosion sample after exposure to 408 cycles at 843° C. Zirconia coating thickness: 0.040 cm; Laser beam: 50 W/mm<sup>2</sup>. 100X

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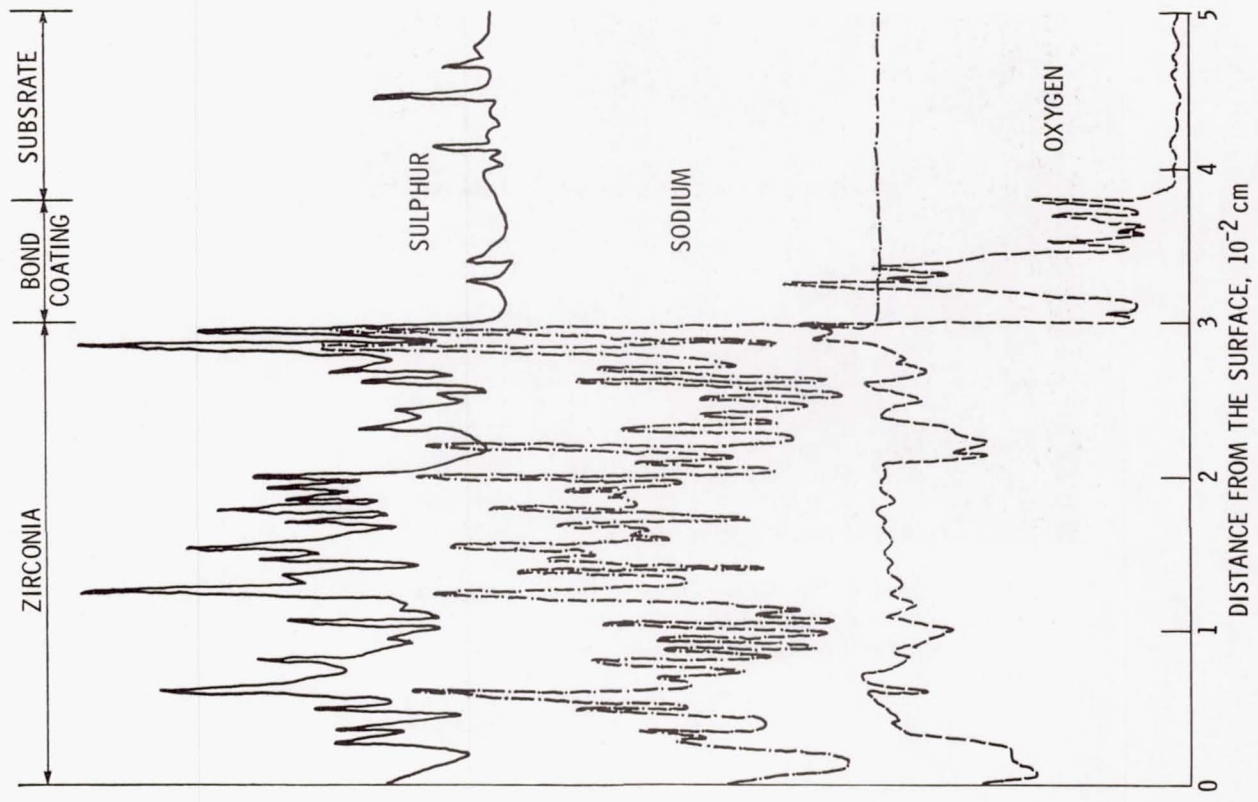


Figure 10. - Electron microprobe continuous - ribbon traverse of TBC system consisting of MAR-509 substrate, NiCrAlZr bond coat and non-glazed 0.040 cm thick zirconia coating after corrosion test. Life: 66 cycles.

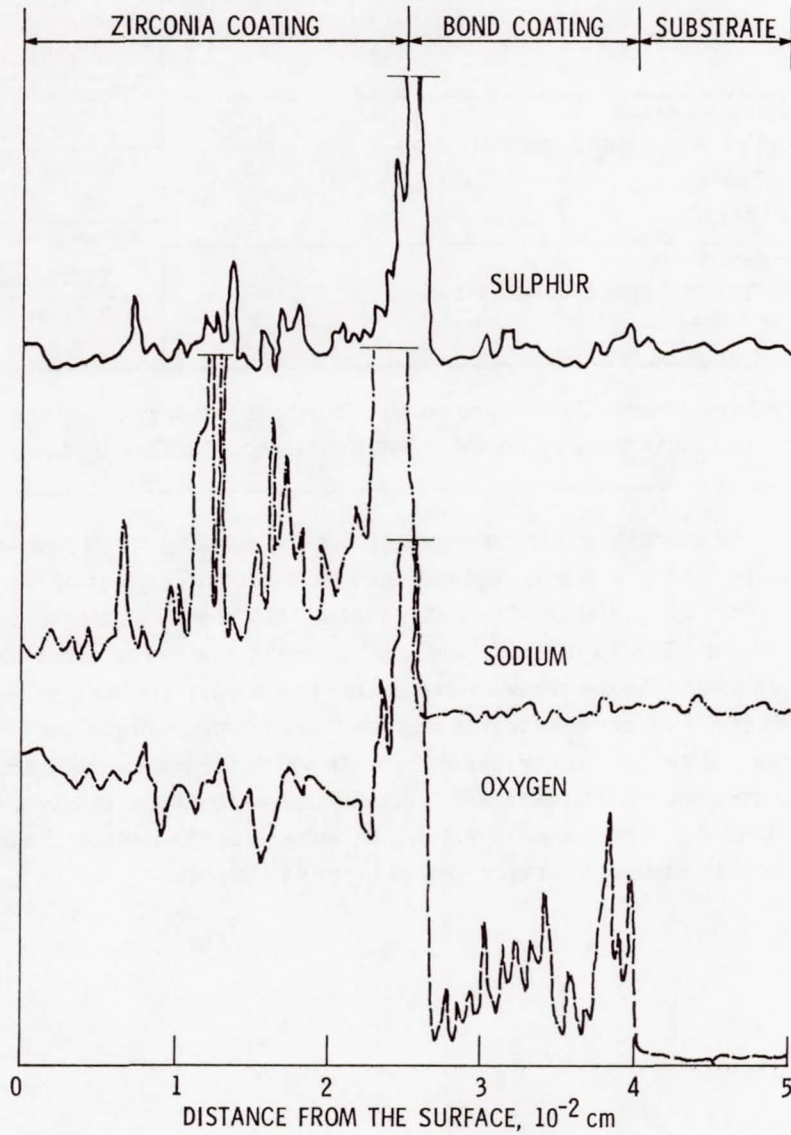


Figure 11. - Electron microprobe continuous-ribbon traverse of TBC system consisting of MAR-509 substrate, NiCrAlZr bond coat and laser glazed 0.040 cm thick zirconia coating after corrosion test. Life: 315 cycles.

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