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Work performed for
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REVIEW OF NASA PROGRESS IN THERMAL BARRIER COATINGS FOR
STATIONARY GAS TURBINES

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SUMMARY

Ceramic thermal barrier coatings for industrial/utility gas turbines have been investigated at NASA Lewis under the DOE-sponsored Critical Research and Advanced Technology Support Project (CRT).

In early burner rig tests of a zirconia-yttria/nickel-chromium-aluminum-yttrium ($ZrO_2-12w/oY_2O_3/NiCrAlY$) coating system on air-cooled superalloy specimens, ceramic coating life (spallation) was found to be sensitive to Na and V concentration in the fuel. The locations of coating spallation corresponded to areas where combustion products were predicted to condense.

Three new thermal barrier coating systems were identified. These are based on calcium silicate, ZrO_2-8w/oY_2O_3 , and a MgO-NiCrAlY cermet. Furthermore, it was found that the spall resistance can be increased by reducing the ceramic layer thickness from 0.038 to 0.013 cm and by the use of more oxidation/corrosion resistant bond coats.

INTRODUCTION

The Critical Research and Advanced Technology Support Project (CRT) started at NASA Lewis in late 1977 under DOE Division of Fossil Fuel Utilization (FFU) sponsorship. The purpose of this project was to provide federal technical support in critical technology areas for the FFU Advanced Power Systems Program. The status of CRT through September, 1978 is given in the FY 1978 Annual Report (1). Work on ceramic/thermal barrier coatings was performed as part of the Long-Life Materials and Technology evaluation tasks.

The basis for the current interest in the use of insulating ceramic thermal barrier coatings (TBCs) on cooled hot section components of gas turbines was brought about by the good performance of such coatings in clean-fuel combustion gas exposures (2, 3, 4). These coatings are presently being developed for both clean-fuel aircraft gas turbines and the potential dirty-fuel environments of industrial/utility gas turbines. Through the use of such coatings, turbine inlet temperatures could be increased, cooling air flow rates could be decreased, or cooling schemes could be simplified. Alternatively, existing gas temperatures and cooling schemes could be maintained and the components could be operated at lower metal temperatures. In this latter instance, increased component durability and reliability would result. These benefits have been examined analytically for utility gas turbines and important fuel and capital cost savings have been identified (5, 6, 7).

Initial results with the $ZrO_2-12w/oY_2O_3/NiCrAlY$ system in the presence of the usual heavy fuel impurities (sodium (Na), vanadium (V), etc.) were less than encouraging (8). As a consequence of these early test results, a comprehensive program within the CRT project was undertaken to improve spall resistance of thermal barrier coatings exposed to the combustion products of potential coal-derived fuels. This program consisted of separate studies involving: (1) evaluation of the behavior of thermal barrier coatings exposed to Na and/or V doped combustion gases; (2) reaction of thermal barrier materials with potential air, fuel and bond coat impurities; (3) optimization of bond coat composition, thickness and deposition parameters; and (4) evaluation and endurance testing of advanced thermal barrier coating systems. The purpose of this paper is to review and discuss the results of this program.

RESULTS AND DISCUSSION

Behavior of Thermal Barrier Coatings in Corrosive Environments

The response of single $ZrO_2-12w/oY_2O_3/Ni-16Cr-6Al-0.6Y$ coated air-cooled hollow erosion bar specimens to Mach 0.3 burner rig exposure with various levels of sodium (Na) and vanadium (V) fuel contamination is summarized in Fig. 1 (8). This coating system was developed for clean fuel, aircraft gas turbines applications (2).

At the 0.5 ppm Na fuel equivalent level, the coating system survived 1300 one-hour cycles without spallation as shown in Fig. 2. Appreciable erosion, presumably due to carbon particles, was noted. At the 5 ppm Na fuel equivalent level, the coating system failed after 92 one-hour cycles at a location outside the hot zone. At the 0.2 and 2.0 ppm V levels, the coating system failed in about 200 and 25 one-hour cycles, respectively. With vanadium in the combustion gases, failures occurred in the hot zone as shown in Fig. 3. When a fuel equivalent dopant level of 5 ppm Na + 2 ppm V was used, the coating failed in 43 one-hour cycles. The microstructure of the coating system on this specimen after exposure is shown in Fig. 4 along with the microstructure of the as-deposited coating. Here, as well as in the other tests, no bond coat corrosion was detected. In addition, coating failure in these tests occurred within the ceramic near the bond coat/oxide interface. This mode of failure is similar to tensile failures reported by Levine (9) and to the furnace and burner rig oxidation failures observed by Stecura (2, 3).

The locations of coating spallation corresponded to areas where combustion products were predicted to condense based on the calculated dew points of the condensates and the temperature profile of the specimens. A summary of the calculated dew points and melting points for the various fuel impurity conditions is given in Table 1 (10).

The most severe conditions for a porous plasma spray deposited ceramic coating such as ZrO_2-12w/oY_2O_3 occur when a corrosive liquid such as vanadium oxide (V_2O_5) or sodium vanadate ($Na_2V_2O_6$) can condense at the surface and completely permeate the coating. That is, the dew point, T_{dp} , is above the surface temperature, T_s , of the ceramic and the melting point, T_{mp} , is below the bond coat temperature, T_{bc} . This was the

case with V_2O_5 formed in the 0.2 ppm and 2 ppm V tests and with $Na_2V_2O_6$ formed in the 5 ppm Na + 2 ppm V test. The problem with fuel or air impurities should not be as severe if the condensed combustion product does not react with the ceramic coating. If the melting point of the condensate is above the dew point (i.e., the condensate is a solid), or if the dew point is below the bond coat temperature, the fuel contaminant may be harmless. The fuel contaminant may also be harmless if the dew point is less than the surface temperature. With 5 ppm Na in the fuel the dew point of the non-reactive sodium sulfate (Na_2SO_4) condensate was below the surface temperature in the hot zone. Failure occurred out of the hot zone where the dew point was above the surface temperature -- as illustrated in Fig. 5. With 0.5 ppm Na in the fuel the dew point was below the bond coat temperature in the hot zone and below the surface temperature, but above the bond coat temperature elsewhere. Thus, this test was no more severe than an oxidation test.

In a second series of Mach 0.3 burner rig tests, three yttria-stabilized zirconias were tested at three fuel-to-air mass ratios and at a fuel equivalent impurity level of 5 ppm Na plus 2 ppm V. The conditions were similar to the conditions of previous tests except eight specimens were tested simultaneously and the substrates were 1.27 cm O.D. Waspaloy cylinders. The results of this test are summarized graphically in Fig. 6. The data indicated that the spall behavior of yttria-stabilized zirconia thermal barrier coatings was sensitive to changes in the fuel-to-air mass ratio and that ZrO_2 -8w/o Y_2O_3 coatings are more spall resistant in this type of combustion environment.

As the fuel-to-air mass ratio was increased from 0.039 to 0.049, the following occurred: the amount of impurities also increased 25 percent; the surface temperature of the ceramic increased; and the temperature gradient through the coating increased. Based on the responses of single coated ZrO_2 -12w/o Y_2O_3 specimens to changes in the fuel impurity concentration, the difference in spall behavior as a consequence of increasing the fuel-to-air mass ratio was attributed primarily to an increase in fuel impurity flux.

Reaction Studies

In support of the burner rig tests, basic studies were conducted to determine how powdered zirconia-yttria, calcium silicate (actual composition was 1.8CaO.SiO₂) and the candidate coating material, barium zirconate ($BaZrO_3$), react with powders of the potential combustion product compounds of major fuel and air impurities and with bond coating elements (11, 12). Equivalent amounts of each compound and of each thermal barrier powder were mixed and reacted in crimped platinum tubes, and the reactions were monitored as a function of time by X-ray diffraction.

Based on the reactions at 1200° and 1400° C, the chemical compounds which were used to react with zirconia-yttria (ZrO_2 -8w/o Y_2O_3) could be divided into four distinct groups:

1. Chemical compounds that did not react with zirconia-yttria:
 Na_2SO_4 , K_2SO_4 , Cr_2O_3 , Al_2O_3 , and NiO
2. Chemical compounds that reacted completely with zirconia-yttria:
 $CaCO_3$ (CaO), $BaCO_3$ (BaO), and $BaSO_4$

3. Chemical compounds that reacted preferentially with the monoclinic phase: Na_2CO_3 (Na_2O), K_2CO_3 , CoCO_3 (CoO), Fe_2O_3 , MgO , SiO_2 , and ZnO
4. Chemical compounds that reacted preferentially with the cubic phase: V_2O_5 , $(\text{NH}_4)_2\text{HPO}_4$ (P_2O_5)

The results for Na_2SO_4 , V_2O_5 and P_2O_5 are in agreement with prior investigations (6, 8).

The results of reactions of calcium orthosilicate and barium zirconate with essentially the same set of compounds are summarized as follows:

1. The impurities that reacted with $2\text{CaO}\cdot\text{SiO}_2$ are Na_2O , BaO , MgO , CoO , Al_2O_3 , Cr_2O_3 , P_2O_5 and V_2O_5 .
2. The impurities that did not react with $2\text{CaO}\cdot\text{SiO}_2$ are Na_2SO_4 , K_2O , K_2SO_4 , BaSO_4 , NiO , ZnO and Fe_2O_3 .
3. The impurities that reacted with BaZrO_3 are Al_2O_3 , Fe_2O_3 , Cr_2O_3 , SiO_2 , P_2O_5 and V_2O_5 .
4. The impurities that did not react with BaZrO_3 are Na_2O , Na_2SO_4 , K_2O , K_2SO_4 , MgO , CaO , CoO and ZnO .

It is noteworthy that no monovalent or divalent oxides and sulfates reacted with barium zirconate. Similarly, calcium orthosilicate was not affected by sulfates; however, it was attacked by sodium, barium and cobalt oxides. Vanadium and phosphorous are impurities generally contained in industrial fuels, and their pentoxides reacted readily with zirconia-yttria barium zirconate and calcium silicate.

Bond Coat Optimization

The work in this study was aimed at improving the oxidation and corrosion resistance of the bond coatings and the adherence of the thermal barrier coatings (15). Three testing methods were used to evaluate bond coatings. Cyclic furnace tests were used to determine the durability of bond coatings alone (oxidation) and to determine their performance as bond coatings in a thermal barrier coating system (endurance). The third method of evaluation was to test thermal barrier coating systems on solid superalloy pin specimens in a Mach 0.3 burner rig.

In cyclic oxidation tests at 1100°C fourteen bond coats were evaluated. Based on specific weight change data and post-test metallography results, the most oxidation resistant bond coatings identified for B-1900 + Hf were Ni-14.1Cr-13.4Al-0.10Zr, Ni-14.3Cr-14.4Al-0.16Y, and Ni-15.8Cr-12.8Al-0.36Y. A typical set of oxidation curves is given in Fig. 7 for cyclic oxidation of bond coats on B1900+Hf at 1010° and 1100°C . For MAR-M509 the most oxidation resistant bond coating found was Ni-30.9Cr-11.1Al-0.48Y.

In endurance testing at 1010°C , twenty-two bond coats were evaluated on B 1900+Hf and eight bond coats were evaluated on Mar-M509. The main criterion used for evaluating thermal barrier coating system performance (life) in endurance testing was the time to crack initiation in the thermal barrier coating.

On B-1900+Hf the 0.010-cm-thick Ni-15.8Cr-12.8Al-0.36Y and Ni-14.1Cr-13.4Al-0.10Zr bond coatings (plasma deposited at 11 kW with argon) appeared to significantly improve the life of the TBC relative to the reference Ni-16.2Cr-5.6Al-0.61Y bond coating. Although Ni-30.9Cr-11.1Al-0.48Y was characterized as a good oxidation resistant bond coating on MAR-M509, thermal barrier coating life was shorter with this bond coating than life of a coating system with the reference bond coating.

In addition to the effect observed for composition changes of the bond coatings, it became apparent during the course of this work that coating deposition parameters affect the oxidation behavior of bond coats. It was found that the oxidation resistance of the bond coats was significantly improved by increasing the coating thickness (0.010 to 0.015 cm), arc power (11 to 20 kW) and hydrogen concentration in the arc gas (0 to 3.5 v/o). The effects of plasma arc power and hydrogen additions to the arc gas on the 1100° C oxidation behavior of a 0.015-cm-thick Ni-15.8Cr-12.8Al-0.36Y bond coating on B-1900+Hf are shown in Fig. 8. Burner rig testing results paralleled results of furnace oxidation and endurance testing. Good bond coatings identified in oxidation and endurance testing gave superior results for thermal barrier coating systems evaluated on solid superalloy pin specimens in burner rig tests.

Advanced Thermal Barrier Coating Systems

The results of early tests of thermal barrier coatings developed for clean fuel aircraft gas turbine service indicated a need to identify ceramic coatings having improved resistance to fuel and air impurities (8, 14). An initial step toward this goal was taken when a series of coating systems was tested in a Mach 0.3 burner rig with the flame doped to the fuel equivalent impurity level of 5 ppm Na + 2 ppm V (8). Ceramic coating thickness was maintained at 0.04 cm and bond coat thickness was maintained at 0.013 cm. The results of these tests are summarized in Fig. 9. Various thermal and chemical treatments of the ZrO₂-12w/oY₂O₃/Ni-16Cr-6Al-0.6Y system resulted in little or no improvement. A more oxidation/hot corrosion resistant bond coat (Ni-21Co-19Cr-13Al-0.6Y) and a dense Y₂O₃ top-coat with the baseline coating offered some improvement. The ZrO₂-8w/oY₂O₃/Ni-16Cr-5Al-0.15Y coating offered an even greater improvement. The most promising of these coating identified was 1.8CaO.SiO₂/Ni-16Cr-6Al-0.6Y which survived over 600 one-hour cycles before spalling compared to about 60 for ZrO₂-12w/oY₂O₃/Ni-16Cr-6Al-0.6Y system. A higher thermal conductivity cermet coating consisting of 50 volume percent MgO - 50 volume percent Ni-20-Cr-7Al-1.0Y over the standard bond coating survived 1000 one-hour cycles without spalling. However, coating thickness was reduced about 50 percent by erosion or microspalling.

In a second series of Mach 0.3 burner rig tests the lives of 1.8 CaO.SiO₂ and ZrO₂-8w/oY₂O₃ thermal barrier coatings exposed to Mach 0.3, Na plus V doped combustion gases were about four times longer when the ceramic thickness was reduced from 0.038 to 0.013 cm (15). High Cr + Al, Ni-31Cr-11Al-0.5Y and Ni-16Cr-13Al-0.4Y bond coatings improved the durability of calcium silicate and ZrO₂-8w/oY₂O₃ TBCs by approximately 2 to 3 times. Similarly, Co-22Cr-13Al-0.3Y and Co-20Cr-9Al-0.4Y bond coatings improved the durability of calcium silicate TBCs by factors of approximately 3 and 2 times, respectively. The results of these tests are summarized in Fig. 10.

Chemical and electron microprobe analyses supported the predictions of condensate compositions and their role in inducing spalling of ZrO_2-8w/oY_2O_3 (Fig. 11). The calcium silicate coating failure involved condensation and penetration of Na, V, and Co (cobalt) containing condensates, calcium sulfate ($CaSO_4$) formation as a result of reaction with sulfur dioxide (SO_2), and the phase change from $\beta-Ca_2SiO_4$ (larnite) to $CaSiO_3$ (wollastonite).

CONCLUDING REMARKS

As a part of this DOE-sponsored Critical Research and Advanced Technology Project substantial progress has been made in understanding the behavior of thermal barrier coatings in environments resulting from firing potential coal-derived fuels and in improving the durability of TBCs for this application. Specifically, the responses of $ZrO_2-12w/oY_2O_3/NiCrAlY$ coatings to Na- and V-doped fuel have been determined. Thermochemical calculations of condensate dew points and melting points combined with the temperature profile of the specimens were found to give a good explanation of observed coating failure locations. Additional understanding of how and why coatings fail (spall) was gained from reaction studies between ceramic coating materials and potential air, fuel, and bond coat impurities.

In the area of advanced ceramic coating development, two coatings (calcium silicate and ZrO_2-8w/oY_2O_3) were identified which significantly extended coating life in Na and/or V doped fuel combustion products. Recent testing involving these two coatings show that coating life can be extended about 4 times when the ceramic coating thickness is reduced from 0.038 to 0.013 cm. Also ceramic coating life was increased about 3 times when some of the high Cr and Al bond coats identified in the bond-coat optimization study were used. Similarly Co-based bond coats improved the durability of calcium silicate-based thermal barrier coating systems by approximately 2 to 3 times.

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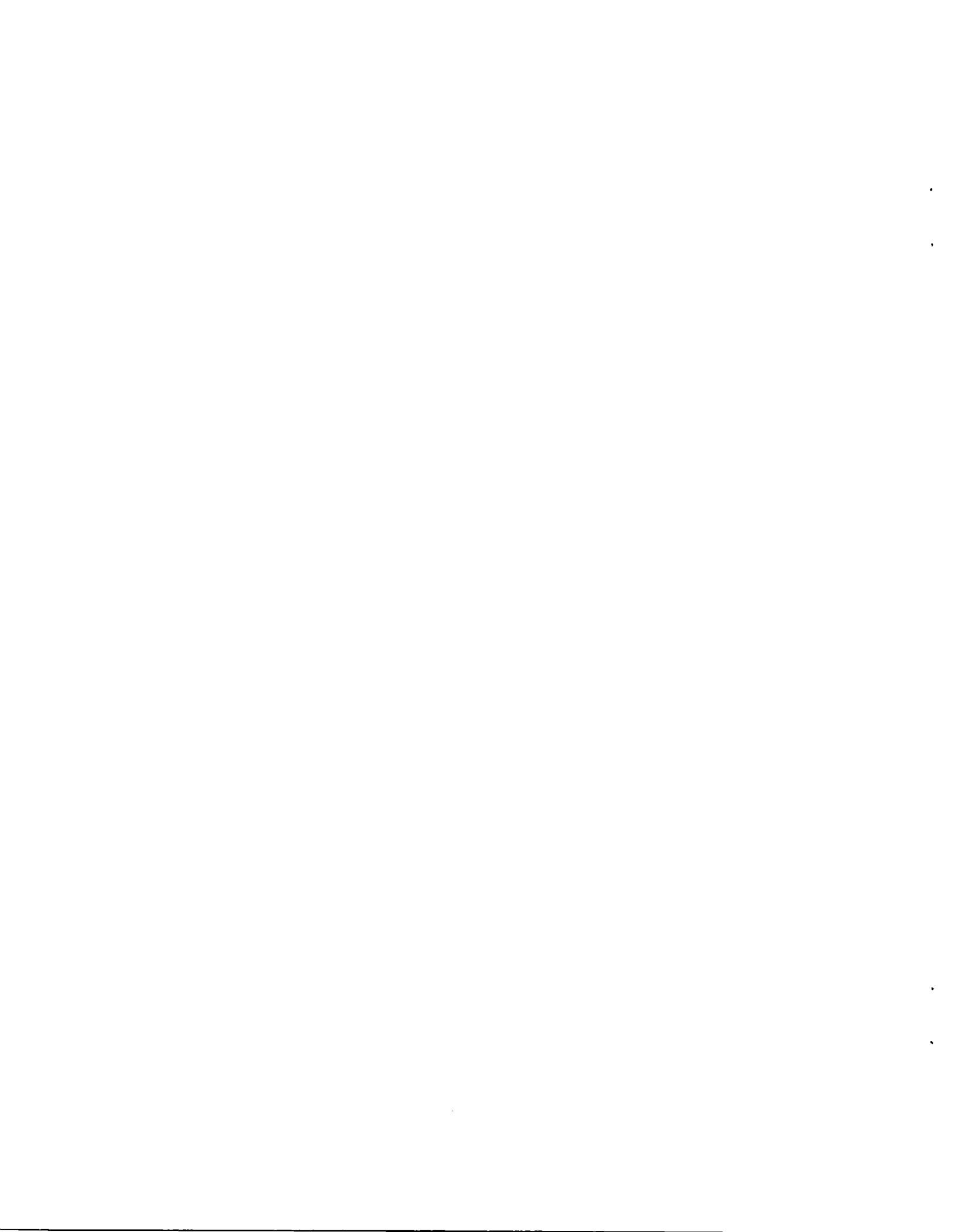
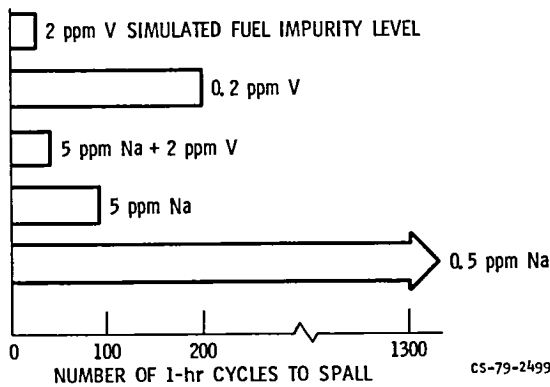


TABLE 1. - RELATION BETWEEN CONDENSATE DEW AND MELTING POINTS AND $ZrO_2 \cdot 12 Y_2O_3/NiCrAlY$ PERFORMANCE

DOPANT LEVEL (REFERRED TO FUEL)	PREDICTED CONDENSATE	DEW POINT T_{dp} , °C	MELTING POINT, T_{mp} , °C	CONDENSATE LOCATION	CYCLES TO COATING FAILURE AND FAILURE LOCATION
5 ppm Na	$Na_2SO_4(l)$	920	884	$T_s > T_{dp} > T_{mp} > T_{bc}$	92, OUT OF HOT ZONE
0.5 ppm Na	$Na_2SO_4(s)$	845	884	$T_s > T_{mp} > T_{bc} > T_{dp}$	1300
2 ppm V	$V_2O_5(l)$	1210	670	$T_{dp} > T_s > T_{bc} > T_{mp}$	25, HOT ZONE
0.2 ppm V	$V_2O_5(l)$	1125	670	$T_{dp} > T_s > T_{bc} > T_{mp}$	200, HOT ZONE
5 ppm Na	$V_2O_5(l)$	1210	670	-----	-----
+ 2 ppm V	$Na_2V_2O_6(l)$	~1155	627	$T_{dp} > T_s > T_{bc} > T_{mp}$	43, HOT & COLD ZONE
	$Na_2SO_4(l)$	~910	884	$T_s > T_{dp} > T_{mp} > T_{bc}$	
	$V_2O_5(s)$	575	670	-----	

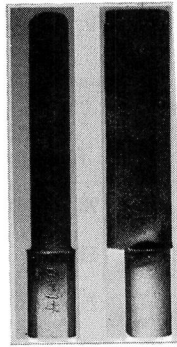
MACH 0.3 BURNER RIG, FUEL/AIR RATIO 0.042; SPECIMEN TEMPS: 980° C SURFACE, (T_{SURF}), 850° C BOND COAT/CERAMIC INTERFACE (T_{bc}) IN THE HOT ZONE; 890° C SURFACE, 760° C BOND COAT/CERAMIC INTERFACE AT TIP & ROOT OF TEST SPECIMEN

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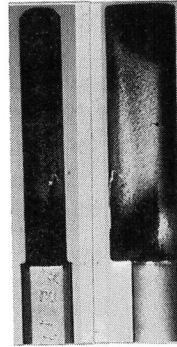


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Figure 1. - Results of $ZrO_2 \cdot 12 w/o Y_2O_3$ TBC fuel impurity sensitivity tests. Mach 0.3 burner rig, 0.05 w/o S in fuel; surface temperature, 980° C; bond coat temperature, 850° C.

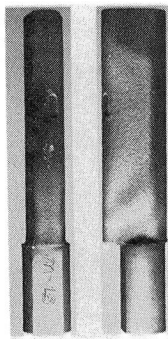


1300 ONE-hr CYCLES
0.5 ppm Na

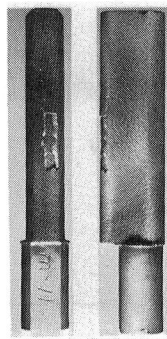


92 ONE-hr CYCLES
5 ppm Na CS-79-2817

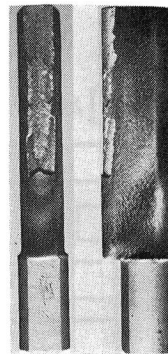
Figure 2. - Effects of sodium on $ZrO_2 \cdot 12$ w/o Y_2O_3 /Ni-16Cr-6Al-0.6Y. Mach 0.3 burner rig, 0.05 w/o S in fuel; surface temperature, $980^\circ C$; bond coat temperature, $850^\circ C$.



200 ONE-hr CYCLES
0.2 ppm V CS-79-2818

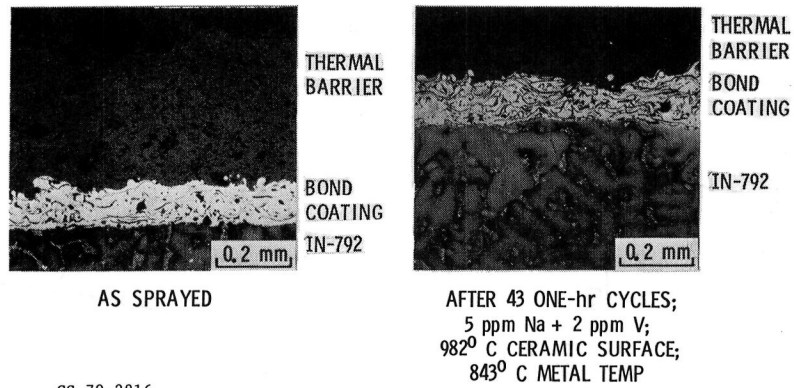


25 ONE-hr CYCLES
2 ppm V



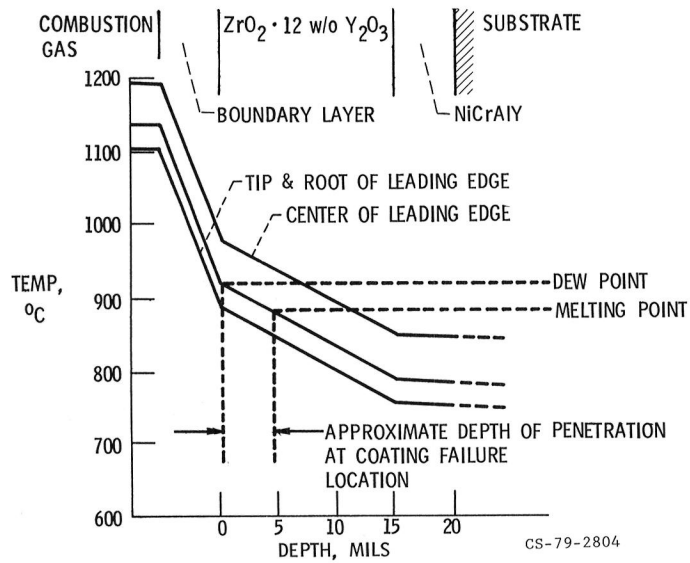
43 ONE-hr CYCLES
5 ppm Na + 2 ppm V

Figure 3. - Effect of vanadium and sodium plus vanadium on $ZrO_2 \cdot 12$ w/o Y_2O_3 /Ni - 16Cr-6Al-0.6Y. Mach 0.3 burner rig, 0.05 w/o S in fuel; surface temperature, $980^\circ C$; bond coat temperature, $850^\circ C$.



CS-79-2816

Figure 4. - Microstructures of $ZrO_2 \cdot 12 \text{ w/o } Y_2O_3/Ni-16Cr-6Al-0.6Y$ on IN-792 (a) as-sprayed (b) after 43 one-hour cycles in combustion products of 5 ppm Na+2 ppmV doped fuel, 980° C ceramic surface and 850° C metal temperature.



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Figure 5. - Penetration of $ZrO_2 \cdot 12 \text{ w/o } Y_2O_3$ by Na_2SO_4 when 5 ppm of Na is present in the fuel.

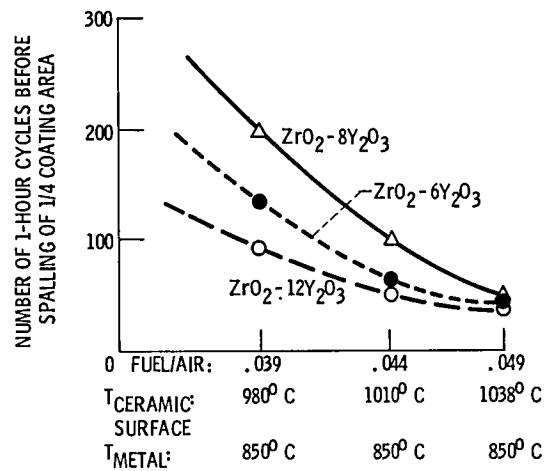
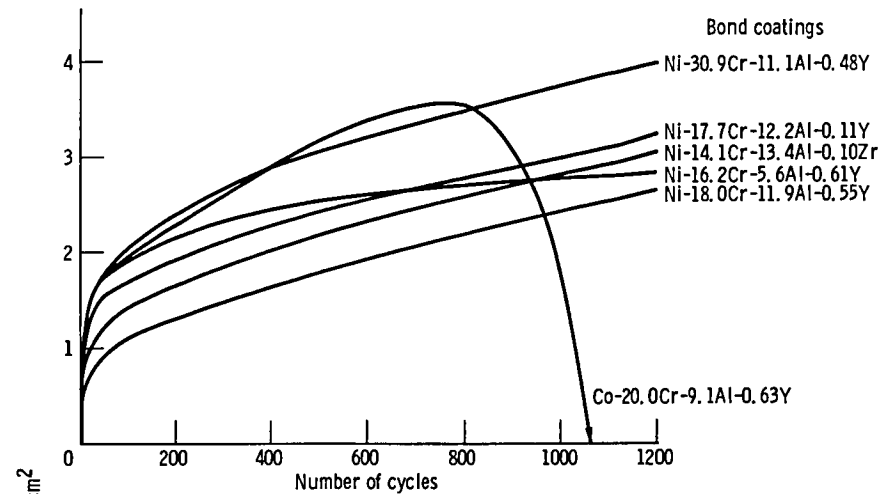
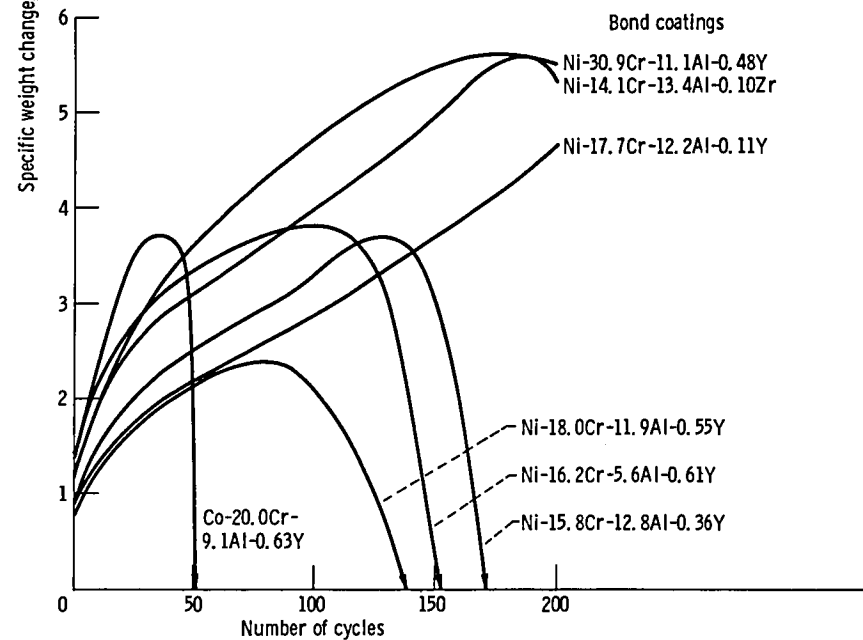


Figure 6. - Fuel-to-air mass ratio burner rig study. Fuel impurity level: 5 ppmNa + 2 ppmV; substrate: 1/2 in. O. D. Waspaloy; bond coat: Ni-16Cr-6Al-0.31Y.



(a) 1010° C oxidation.



(b) 1100° C oxidation.

Figure 7. - Effect of oxidation temperature on weight-change behavior of 0.010-cm-thick plasma-deposited bond coatings on B-1900 + Hf in cyclic furnace oxidation at 1000° and 1100° C in static air. Cycles: 1 hr at test temperature and 20 min or more cooling. Coatings applied at 11 kW with argon arc gas.

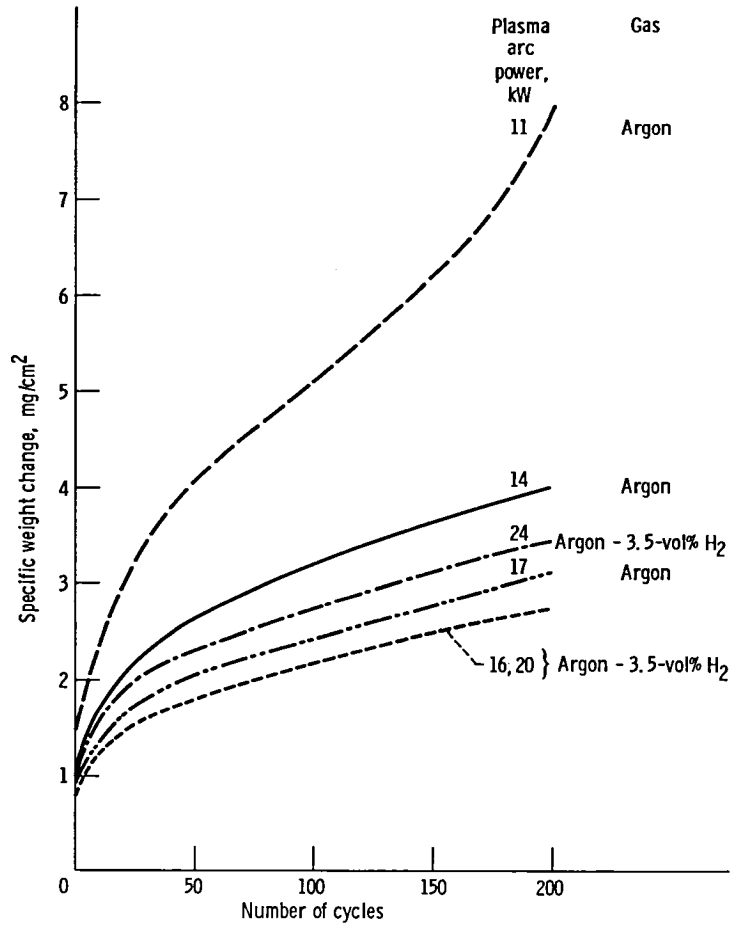


Figure 8. - Effect of plasma-deposition parameters on weight-change behavior of Ni-15. 8Cr-12. 8Al-0. 36Y bond coating on B-1900 + Hf in cyclic furnace oxidation at 1100° C in static air. Nominal coating thickness, 0. 015 cm.

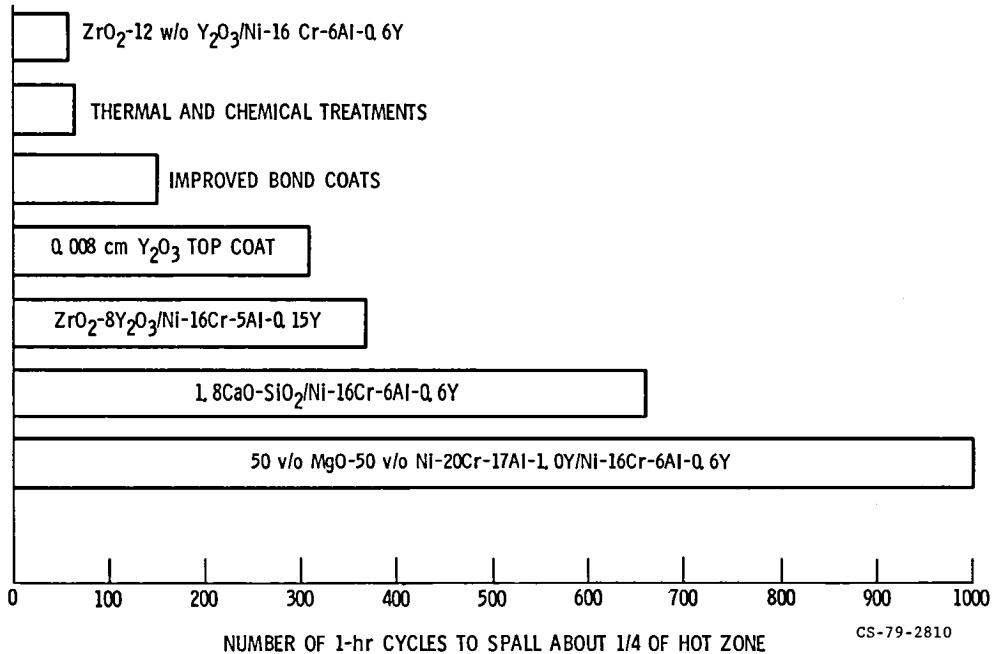
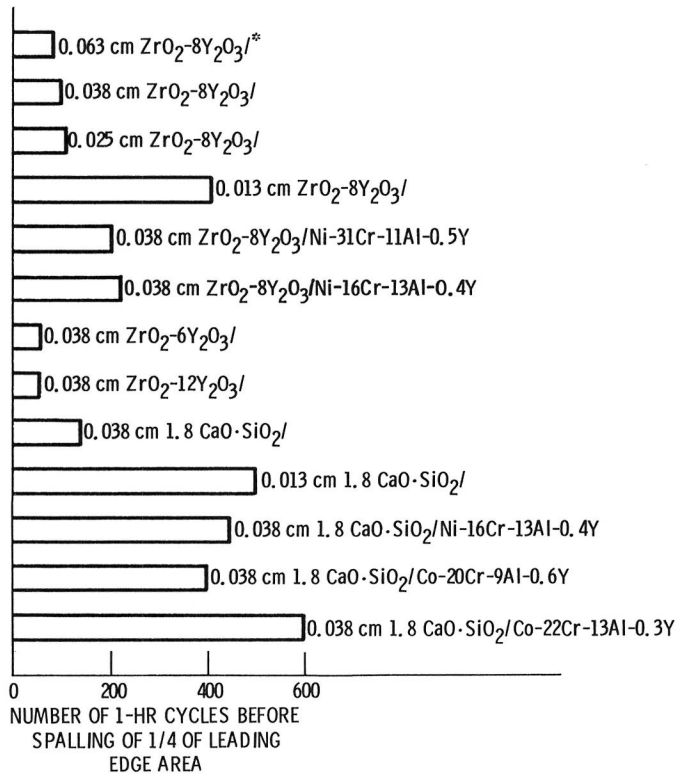


Figure 9. - Improved thermal barrier coatings. Mach 0. 3 burner rig, fuel impurities, 5 ppm Na, 2 ppm V, 0. 05% S; flame temperature, 1380° C; ceramic surface temperature, 980° C; metal temperature, 850° C.



*BOND COATING IS 0.013 cm Ni-16Cr-6Al-0.3Y UNLESS OTHERWISE SPECIFIED.

Figure 10. - Mach 0.3 burner rig corrosion test; fuel impurity level, 5 ppm Na + 2 ppmV; fuel/air, 0.046; ceramic surface temperature, 980^o C; bond coat temperature, 850^o C.

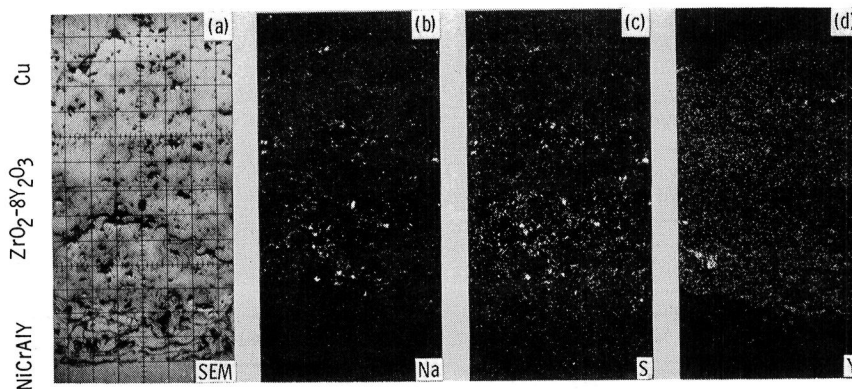


Figure 11. - Electron microprobe maps of $ZrO_2-8Y_2O_3/Ni-16Cr-6Al-0.31Y$ coating system after 120 1-hour cycles.



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16. Abstract Ceramic thermal barrier coatings for industrial/utility gas turbines have been investigated at NASA Lewis under the DOE-sponsored Critical Research and Advanced Technology Support Project (CRT). In early burner rig tests of a zirconia-yttria/nickel-chromium-aluminum-yttrium ($ZrO_2-12w/oY_2O_3/NiCrAlY$) coating system on air-cooled superalloy specimens, ceramic coating life (spallation) was found to be sensitive to Na and V concentration in the fuel. The locations of coating spallation correspond to areas where combustion products were predicted to condense. Three new thermal barrier coating systems were identified. These are based on calcium silicate, ZrO_2-8w/oY_2O_3 , and a MgO-NiCrAlY cermet. Furthermore, it was found that the spall resistance can be increased by reducing the ceramic layer thickness from 0.038 to 0.013 cm and by the use of more oxidation/corrosion resistant bond coats.			
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