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Part II. Effect of Spray Parameters on the Performance of Several Hafnia-Yttria and Zirconia-Yttria Coatings

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Summary

This is the second of two reports which discuss initial experiments on thermal barrier coatings prepared and tested in newly upgraded plasma spray and burner rig test facilities at Lewis Research Center. The first report, Part I, describes experiments designed to establish the spray parameters for the baseline zirconia-yttria coating. Coating quality was judged primarily by the response to burner rig exposure, together with a variety of other characterization approaches including thermal diffusivity measurements. That portion of the study showed that the performance of the baseline NASA coating was not strongly sensitive to processing parameters. In this second part of the study, new hafnia-yttria coatings were evaluated with respect to both baseline and alternate zirconia-yttria coatings. The hafnia-yttria and the alternate zirconia-yttria coatings were very sensitive to plasma-spray parameters in that high-quality coatings were obtained only when specific parameters were used. The reasons for this important observation are not understood.

Introduction

This report discusses experiments to investigate the performance of hafnia-yttria thermal barrier coatings using the procedure described in Part I (Miller, Leissler, and Jobe, 1992). The hafnia-yttria system was chosen because of its chemical similarity to the zirconia-yttria system, which has been successfully developed for use as a thermal barrier coating. The primary driving force is a desire to identify new materials that may be more stable at higher temperatures.

Experiment

Four powder lots of hafnia-yttria and one powder lot of zirconia-yttria, denoted as lots B1 to B5, respectively, were used in this study. They were prepared by a different vendor than the one used for Part I. A second lot of zirconia-yttria, identified here and in Part I as lot A2, was also included in this study. The bond coat compositions were either Ni-35%Cr-5%Al-1%Y as in Part I, or they were Ni-35%Cr-5%Al-1%Yb. Coating layer thicknesses, substrates, spray torches, and test rigs were identical with those described in Part I. The only difference was that a chiller was installed in the cooling water line for the plasma-spray torches. Thermal diffusivity measurements were not made on these specimens.

Results and Discussion

Characterization of Powder Lots

All the hafnia-yttria and zirconia-yttria powder lots were prepared by agglomerating and sintering. The hafnia-yttria lots were prepared by vendor "B" according to NASA specifications and differ primarily in the percentage of yttria. Lot B5 is an off-the-shelf zirconia-yttria prepared by the vendor to the specifications of an engine company. The goal of this portion of the study was to investigate the response of these new materials as a function of changes in processing parameters.

Chemistries, particle size distributions, and x-ray analysis for the powder lots. – Table I shows the levels of yttria and hafnia, and up to six trace impurities as measured by NASA and by the vendor. NASA and vendor results agreed for the yttria constituent. The compositions, based on the NASA analyses, were

B1: HfO_2-8.4%Y_2O_3
B2: HfO_2-11.4%Y_2O_3
B3: HfO_2-15.0%Y_2O_3
B4: HfO_2-27.2%Y_2O_3
B5: ZrO_2-7.8%Y_2O_3

The NASA analyses indicated relatively high levels of silica and iron oxide impurities in these powders. However, since the analyses were done on the same date that corresponded to the higher values for iron oxide and silica in table I of Part I, the high levels reported for these two impurities are questionable.

Particle size distribution was determined by sieve analysis. The sieve analyses for the four hafnia-yttria lots B1 to B4 and one zirconia-yttria lot B5 are given in table II, together with the analysis for the reference lot A2. The analyses show that the four hafnia-yttria lots had a more narrow particle size distribution than the reference lot A2, with between 5.2 and

TABLE I.–CHEMICAL ANALYSES BY X-RAY FLUORESCENCE SPECTROMETRY FOR LOTS B1 TO B5

Lot	Analysis	Composition, wt %								
		Zirconia	Yttria	Alumina	Calcia	Iron Oxide	Hafnia	Silica	Titania	Magnesia
B1	Vendor	2.4	8.4	– – –	– – –	– – –	Bal	– – –	– –	– – –
	NASA	2.16	8.42	1.14	0.06	0.52	Bal	0.57	– –	– – –
	(6/89)									
B2	Vendor	2.5	11.0	– – –	– – –	– – –	Bal	– – –	– –	– – –
	NASA	2.65	11.35	.14	.05	.54	Bal	.59	– –	– – –
	(6/89)									
B3	Vendor	2.3	15.6	– – –	– – –	– – –	Bal	– – –	– –	– – –
	NASA	2.33	15.03	.14	.05	.48	Bal	.54	– –	– – –
	(6/89)									
B4	Vendor	1.0	27.3	– – –	– – –	– – –	Bal	– – –	– –	– – –
	NASA	.82	27.24	.13	.06	.56	Bal	.74	0.1	0.04
	(6/89)									
B5	Vendor	Bal	7.71	.12	.03	.02	1.86	.19	.10	.06
	NASA	Bal	7.84	.17	.06	.55	1.78	.23	– –	– – –
	(1/91)									

TABLE II.–CUMULATIVE PARTICLE SIZE DISTRIBUTIONS FOR LOTS B1 TO B5 AND REFERENCE LOT A2

Particle size range		Cumulative particle size distribution, percent					
		Lot					
Sieve size	μm	B1	B2	B3	B4	B5	A2
–325	–44	5.2	9.4	7.6	12.2	48.5	18.1
–270/+325	–53/+44	41.1	40.0	34.2	34.9	66.9	36.9
–230/+270	–62/+53	77.3	72.6	65.8	70.5	83.7	61.2
–200/+230	–74/+62	97.7	96.1	96.7	98.9	94.3	87.3
–170/+200	–88/+74	99.7	99.7	99.6	99.9	98.6	99.5
–140/+170	–105/+88	99.9	100.0	99.8	99.95	99.9	99.9

12.2 percent below 325 mesh (44 μm) and between 0.1 and 0.4 percent above 200 mesh (74 μm). This narrow distribution is in accordance with NASA specifications. Zirconia-yttria lot B5 had a broad distribution, with 48.5 percent finer than 325 mesh and 1.4 percent more coarse than 200 mesh.

X-ray diffraction analysis of the (111) region of the diffraction pattern gave the following percent monoclinic peak intensities for the hafnia-yttria powder lots: lot B1, 19.6 percent; lot B2, 5.9 percent; lot B3, 3.4 percent; and lot B4, 5.6 percent (however, the cubic/tetragonal $(111)_{f,t}$ peak was very broad). For the zirconia-yttria powder lot B5, the percent peak monoclinic intensity was only 1.3 percent. The scan rate used for these measurements was 2 sec/0.02°.

Preparation and x-ray analysis of plasma-sprayed specimens. –The test specimens were cylindrical superalloy substrates coated with a layer of low-pressure plasma-sprayed bond coat, as in Part I. The bond coat was Ni-35%Cr-5%Al-1%Yb in some cases and Ni-35%Cr-5%Al-1%Y in others. Both compositions were cut to below 325 mesh. The coatings were prepared using Electro-Plasma Inc. (EPI) plasma generators for both the atmospheric-pressure plasma-sprayed ceramic and low-pressure plasma-sprayed bond coat and ancillary equipment as described in Part I. One important difference was that a chiller was added to the plasma torch water-cooling circuit. Also, the flow rate of the Ar-3.8%H auxiliary gas that was used for low-pressure plasma

spraying of the bond coat was increased to 95 standard liters per minute (SLPM), or 202 standard cubic feet per hour (SCFH), and the power level was increased to 82 kW at 1500 A.

The ceramic-layer spray parameters were initially based on the experiments described in Part I. However, the companion density specimens were not prepared immediately before or immediately after preparation of the durability test specimens. Attempts to prepare companion specimens at a later date failed to produce meaningful information because by that time the plasma torch electrodes had degraded, causing higher coating porosity. As a result, the densities of the durability specimens could not be determined.

Also, as will be discussed further, the initial set of hafnia-yttria specimens that had been prepared from parameters selected from Part I did not perform well. New specimens prepared using parameters that were selected to yield lower densities gave greatly enhanced lives. Unfortunately, the second group of specimens had to use a different bond coat than the first because supplies of the first bond coat were no longer available. Two cylindrical specimens were prepared for each parameter set.

One specimen from each of the five lots B1 to B5 was selected for x-ray diffraction analysis. In each case the specimens had been sprayed using a 35/40/2 parameter set. (According to this shorthand notation the first number refers to the power level in kilowatts, the second to the percent helium in argon in the arc gas, and the third to the powder carrier gas flow rate in standard liters per minute.) The (111) region of the pattern for the four hafnia-yttria specimens, lots B1 to B4, is shown in figure 1(a). The scan rate for these patterns was 2 sec/0.02°. Each pattern in the figure shows a strong (111) cubic/tetragonal peak, although the peak for lot B4 is shifted to a lower angle. The (111) and (111) monoclinic peaks are visible on either side of the patterns for lots B1 and B2. Figure 1(b) shows a 4 sec/0.02° scan of the same region for lot B3. The upper trace in that figure is at a 45 times more sensitive scale than the lower trace. The weak monoclinic peaks are visible in the upper trace. The percent monoclinic intensity (based on peak intensities) and the corresponding scan rates for all five B lots were as follows:

B1: 9.4 percent (2 sec/0.02°)
B2: 7.2 percent (2 sec/0.02°)
B3: 0.6 percent (4 sec/0.02°)
B4: 0 percent (32 sec/0.02°)
B5: 0 percent (2 sec/0.02°)

The percent monoclinic intensities for two of the lot A2 specimens that had been sprayed using the 36I/20/1.3 parameter set were 1.3 and 1.6 percent. (The "I" after the 36 denotes internal powder injection.) These values compared well with the lot A2 specimens from Part I.

Figure 1(c) shows the (400) region of lots B1 to B4. There is no distinct evidence of the t'-tetragonal phase that was observed with zirconia-yttrias tested in Part I. The pattern for zirconia-yttria lot B5 also did not show the t' peaks. Thus, the phase composition of the plasma-sprayed zirconia-yttria material from lot B5 differed from lot A2, though the percentages of yttria were very similar. The reasons for these differences are not well understood.

Burner Rig Durability Study

Burner rig test conditions. — The burner rig test conditions used were the same as those reported in Part I (Miller, Leissler, and Jobe, 1992). A four-specimen rotating carousel of specimens was exposed to the flame of a Mach 0.3 burner rig for 6 min to a maximum temperature of 1150 °C (2100 °F) followed by 4 min of forced-air cooling to room temperature. The effective time at temperature was estimated to be 4.0 min/cycle. A calibrated disappearing-filament optical pyrometer was used to measure temperature. The calibration experiment yielded the same pyrometer correction factor as for zirconia-yttria. This was fortunate because the two types of coatings were generally tested together in the same carousel. However, much more careful work is required to confirm that the correction factors (i.e., the emissivities) of each material are the same. Failure was taken as the first indication of spalling or rupture of the delaminated ceramic.

Durability test results. — Durability test results for the hafnia-yttria specimens from lots B1 to B4 and the zirconia-yttria specimens from lots A2 and B5 are given in table III. The initial portions of these specimens were tested in the burner rig labeled rig 1 in Part I. After a breakdown of rig 1, the remaining specimens were tested in rig 2. This shift was made before the rig-to-rig effect discussed in Part I was noted. Inspection of table III again shows such an effect. Of 11 specimens tested in both rigs, nine lasted significantly longer in rig 1 than in rig 2 while three of the shorter-lived specimens lasted as long or longer in rig 2. However, there was no evidence of a spray order effect. (It is possible that the installation of the chiller between the time that the specimens from Part I and Part II were prepared allowed the power lines and torch to run cooler and that this eliminated the spray order effect by preventing overheating.)

The initial test specimens were prepared using three sets of spray parameters, 40/40/4.5, 55/20/4.5, and 45/20/4.5, with units of kilowatts, percent helium in argon, and standard liters per minute of argon powder carrier gas, respectively. (Note: the latter parameter set was inadvertently omitted for lot B2, the reference zirconia-yttria lot A2 was initially sprayed using only the 40/40/4.5 parameter set, and the zirconia-yttria lot B5 was not initially included but had been previously evaluated in an unpublished study). All these coatings used the same lot of low-pressure plasma-sprayed Ni-35%Cr-5%Al-1Y bond coat.

TABLE III.–RESPONSE OF HAFNIA-YTTRIA AND REFERENCE ZIRCONIA-YTTRIA SPECIMENS TO BURNER RIG EXPOSURE

Lot	Coating System	Parameter[a] Set	Spray Order	Test Life, cycles		
				Rig 1	Rigs 1 and 2	Rig 2
B1	HfO_2–$8Y_2O_3$/NiCrAlYb	36I/20/1.3	1	– – –	– – – – – – –	55
			2	– – –	– – – – – – –	31
		35/40/2	1	50	– – – – – – –	– – –
			2	64	– – – – – – –	– – –
	HfO_2–$8Y_2O_3$/NiCrAlY	45/20/4.5	1	– – –	– – – – – – –	6
			2	8	– – – – – – –	– – –
		55/20/4.5	1	11	– – – – – – –	– – –
			2	– – –	– – – – – – –	7
		40/40/4.5	1	– – –	– – – – – – –	5
			2	– – –	– – – – – – –	4
B2	HfO_2–$11Y_2O_3$/NiCrAlYb	36I/20/1.3	1	37	– – – – – – –	– – –
			2	36	– – – – – – –	– – –
		35/40/2	1	70	– – – – – – –	– – –
			2	96	– – – – – – –	– – –
	HfO_2–$11Y_2O_3$/NiCrAlY	55/20/4.5	1	123	– – – – – – –	– – –
			2	– – –	– – – – – – –	81
		40/40/4.5	1	– – –	– – – – – – –	12
			2	20	– – – – – – –	– – –
B3	HfO_2–$15Y_2O_3$/NiCrAlYb	36I/20/1.3	1	– – –	– – – – – – –	278
			2	– – –	1177 (28%)[b]	– – –
		35/40/2	1	1470	– – – – – – –	– – –
			2	– – –	894 (24%)[b]	– – –
	HfO_2–$15Y_2O_3$/NiCrAlY	45/20/4.5	1	24	– – – – – – –	– – –
			2	14	– – – – – – –	– – –
		55/20/4.5	1	– – –	– – – – – – –	12
			2	18	– – – – – – –	– – –
		40/40/4.5	1	10	– – – – – – –	– – –
			2	– – –	– – – – – – –	10
B4	HfO_2–$27Y_2O_3$/NiCrAlYb	36I/20/1.3	– –	807	– – – – – – –	– – –
			– –	1072	– – – – – – –	– – –
		35/40/2	1	1848	– – – – – – –	– – –
			2	– – –	1648 (82%)[b]	– – –
	HfO_2–$27Y_2O_3$/NiCrAlY	45/20/4.5	1	– – –	– – – – – – –	30
			2	27	– – – – – – –	– – –
		55/20/4.5	1	– – –	– – – – – – –	14
			2	23	– – – – – – –	– – –
		40/40/4.5	1	– – –	– – – – – – –	12
			2	14	– – – – – – –	– – –
A2	ZrO_2–$8Y_2O_3$/NiCrAlYb	36I/20/1.3	1	– – –	– – – – – – –	994
			2	– – –	– – – – – – –	1176
		35/40/2	1	– – –	– – – – – – –	827
			2	– – –	– – – – – – –	932
		40/40/4.5	1	– – –	– – – – – – –	1139
			2	2448	– – – – – – –	– – –
	HfO_2–$27Y_2O_3$/NiCrAlY	40/40/4.5	– –	– – –	– – – – – – –	692
			– –	1155	– – – – – – –	– – –
B5	ZrO_2–$7Y_2O_3$/NiCrAlYb	36I/20/1.3	1	– – –	– – – – – – –	581
			2	– – –	– – – – – – –	665
		35/40/2	1	– – –	– – – – – – –	539
			2	– – –	– – – – – – –	90
		55/20/4.5	1	– – –	– – – – – – –	15
			2	– – –	– – – – – – –	51
		40/40/4.5	1	– – –	– – – – – – –	13
			2	– – –	– – – – – – –	15

[a]According to this shorthand notation, the first number refers to the power level in kilowatts, the second to the percent helium in argon in the arc gas, and the third number to the powder carrier gas flow rate in standard liters per minute.

[b]Number in parentheses indicates percentage of testing done in rig 1.

All the initial coatings from B powder lots performed very poorly, with most coatings failing well under 100 cycles. Therefore, another batch of test specimens was prepared using two different sets of parameters. The first set, 35/40/2, used low feed-gas flow rate, which was expected to yield lower ceramic density. The other set used internal injection of the ceramic and it is listed in table III as 36I/20/1.3, where the "I" stands for internal injection. Unfortunately, the bond coat for this second batch of specimens had to be switched to a Ni-35%Cr-5%Al-1%Yb from the Ni-35%Cr-5%Al-1%Y.

The test results are plotted in figure 2 for the portion of the data from rig 1 for which results for most of the parameter sets are available. As mentioned previously, the density could not be measured for these specimens because the companion specimens were not sprayed until after the electrodes had degraded. For the parameter sets involving external injection, porosity is expected to increase from 40/40/4.5 to 55/20/4.5 to 45/20/4.5. The porosity of the 35/40/2 parameter set was roughly comparable to that of the 45/20/4.5 set in subsequent unpublished experiments, although this may or may not have been so at the time the test specimens were prepared. The porosity of the specimens prepared using the internally injected 36I/20/4.5 parameter set may also be relatively high, based on the improved performance and on density measurements performed on specimens sprayed at a later date. Micrographs of selected specimens are discussed in the subsection Metallography.

Several initial observations can be made from inspection of table III and figure 2:

(1) Lives in rig 1 tended to exceed those in rig 2.

(2) All the specimens from reference zirconia-yttria lot A2 performed well regardless of spray parameters.

(3) The hafnia-yttria specimens, as well as the zirconia-yttria specimens from lot B5, all appear to show a strong parameter/life effect.

(4) The HfO_2-16%Y_2O_3 and HfO_2-27%Y_2O_3 lots B3 and B4, respectively, outperformed the compositions having lower levels of yttria.

(5) The best performing hafnia-yttria compositions were comparable in life to the reference A2 zirconia-yttria specimens.

(6) The best zirconia-yttria coatings from lot B5 had shorter lives than the best zirconia-yttria coatings from Part I.

A statistical analysis was used to help to quantify these impressions.

Statistical analysis of the hafnia-yttria and reference zirconia-yttria test burner rig data. — A statistical analysis was performed on a portion of the data in table III. The data were handled as a randomized block plan with the independent variables being rig and "treatment." Each of the various combinations of composition, lot, and spray parameters (density) was considered to be a treatment. This differs from the approach in Part I, which used rig and lot as the independent variables, with density and density squared as covariates. Density was not included explicitly in the present analysis because the density values were unknown. The analysis was mostly limited to the better performing specimens from the second batch sprayed, and only those specimens tested solely in one of the two rigs were included. The bond coats for these specimens consisted of Ni-35%Cr-5%Al-1%Yb, and the ceramic layers, in terms of lot and spray parameters, were B3 (35/40/2), B4 (36I/20/1.3), B4 (35/40/2), A2 (36I/20/1.3), A2 (35/40/2), A2 (40/40/4.5), B5 (36I/20/1.3), and B5 (35/40/2). The one set of specimens taken from the first batch had a Ni-35%Cr-5%Al-1%Y bond coat and a ceramic from lot A2 (parameter set 40/40/4.5). Comparisons between the latter specimen and the others are confounded by possible batch effects. The dependent variable was the log of life (which produced a better fit than life), and the one specimen from treatment B5 (35/40/2) which failed in only 90 cycles was removed from the analysis.

High F-ratios and low probabilities suggest that there is a statistically significant rig effect ($F = 23.5$, $p = 0.005$) and that there are differences among the specimens ($F = 9.9$, $p = 0.011$). The difference between mean log of life values is 0.2774, with a 95-percent least significant interval of 0.1470. The ratio corresponding to this difference of logs was 1.9, which is equal to the value obtained in Part I. Because the F-test indicates that there are significant differences among the treatments, one may construct a least significant interval plot (Mason, Gunst, and Hess, 1989), as shown in figure 3. This figure supports the observations discussed in the section Durability Test Results.

An additional observation is that the NiCrAlYb bond coat outperformed the NiCrAlY bond coat; however, that conclusion is only tentative because of the batch effect described here.

Metallography. — Photomicrographs of selected specimens are shown in figure 4. The figure includes photomicrographs from each of the five B lots at the 35/40/2 parameter set and from lot A2 at 40/40/4.5 The top photos in the figures are from sections near the base of the test specimen and are taken as the as-sprayed microstructure. The bottom photos are from the hot zone region after failure. The general features of these photographs are similar to those shown in Part I (ref. 1) and in Brindley and Miller (1990) and DiMasi, Sheffler, and Ortiz (1989). In the as-sprayed photographs, fine-scale porosity is present in the bond coat layer. After testing, the bond coats have the same features as in Part I; that is, the porosity coarsens and is observed primarily at the interface with the ceramic. Short-lived specimens (figures 4(a) and (b), bottom photos) show this process at an intermediate stage. As in Part I, an alumina scale forms on the bond coat with very occasional stringers, and α-Cr is observed within the bond coat but is depleted near the interface with the substrate. No difference is observed between NiCrAlY and NiCrAlYb bond coats. In the ceramic layer, all six figures are rather similar,

especially in the as-sprayed structure. One of these, the top photo in figure 4(d), appears to be more porous than the others, although more work would be required to confirm that a difference actually exists. Failure occurs, as expected, in the ceramic layer near the interface, and there is no obvious difference between the appearance of the ceramic in long-lived versus short-lived specimens and in zirconia-yttria versus hafnia-yttria specimens.

Table IV lists the oxide scale thickness for selected specimens. The table also indicates whether the ceramic was zirconia- or hafnia-based, whether the bond coat contained yttrium or ytterbium, a qualitative estimate of ceramic layer density based on spray parameters, in which rig the specimen was tested, the cycles to failure, the total number of cycles in the test rig, and the average of six measurements of scale thickness. The pooled standard deviation for these measurements (based on the mean variance) is 0.73, and the standard error of the mean is 0.03.

Figure 5 is a plot of the log of the measured scale thickness versus the log of cycles in the burner rig for zirconia-yttria and hafnia-yttria specimens tested in rigs 1 and 2. The plot shows that there is a strong correlation between time in the rig and scale thickness but little or no correlation between scale thickness and rig or ceramic. The absence of a high correlation between thickness and rig suggests that the specimens in each rig must be at essentially the same temperature, and that the rig effect must have been due to some other phenomenon such as a difference associated with the cooling air.

For the best coating systems tested, the scale thicknesses were about 6 μm shortly after failure. If any of the specimens had been cycled much less frequently (such as once per day), they would have survived a greater number of hours at temperature, and therefore the scale thickness (or weight gain) at failure would have been greater. For very infrequent cycling, the scale thickness (or weight gain) at failure can rise by perhaps a factor of two. (Miller, Agarwal, and Duderstadt, 1984; DiMasi, Sheffler, and Ortiz, 1989). This observation leads to the concept of critical scale thickness (or the analogous concept of critical weight gain). The critical scale thickness is the scale thickness observed after single cycle failure, that is, the thickness observed after that amount of time at temperature which is just sufficient to cause failure on first cooling. The concept, which applies only to a specific coating system in a specific application, may be incorporated into life prediction models. The term is occasionally misinterpreted; for example, Wu et al.(1989) have criticized this concept based on the incorrect assumption that all coatings must fail at the same weight gain regardless of composition or cycle. This is a misreading of the definition.

TABLE IV.–OXIDE SCALE THICKNESSES FOR SELECTED SPECIMENS

Ceramic case	Estimated density	Rig	Bond coat	Cycles to failure	Total cycles	Scale thickness, μm
Zirconia	Low	2	NiCrAlYb	581	635	4.7
	Low	2	NiCrAlYb	90	143	2.3
	Low	2	NiCrAlYb	932	932	5.2
	High	2	NiCrAlYb	1139	1243	4.5
	High	1	NiCrAlYb	2448	2448	4.8
	High	2	NiCrAlY	692	692	4.5
	High	1	NiCrAlY	1155	1195	4.0
Hafnia	Low	1	NiCrAlYb	50	53	1.9
	Low	1	NiCrAlYb	126	126	2.0
	Low	1	NiCrAlYb	1270	1270	5.8
	Low	1	NiCrAlYb	894	357	3.7
	High	1	NiCrAlY	10	10	1.2
	Low	1	NiCrAlYb	807	912	4.2
	Low	2	NiCrAlYb	1648	1782	4.7
	Low	2	NiCrAlY	14	14	1.2
	Low	1	NiCrAlYb	1470	1578	5.5

Summary of Results

While it was shown in Part I that high-precision (i.e., low random error) density measurements may be made using an Archimedes approach, this study has shown that the measurements must be made on coupons that are prepared at the same time as the test specimen. Otherwise the densities may change because of such factors as electrode degradation.

As in Part I, considerable rig-to-rig variability was observed, but, unlike Part I, the first specimen prepared did not tend to outperform the second. The installation of a chiller in the plasma torch water cooling system between preparation of the first and second batches may have helped to prevent the recurrence of the spray-order effect.

While the zirconia-yttria coatings from Part I were relatively insensitive to spray parameter variations, the effect of spray parameters in this study was very strong for the zirconia-yttria prepared to engine company specifications and for the hafnia-yttria prepared by vendor "B." The reason for this is not apparent, and there is no obvious correlation to particle size distribution or chemistry.

The better hafnia-yttria compositions, when sprayed with certain parameter sets, performed about as well as the baseline zirconia-yttria coatings. However in contrast to prior experience with zirconia-yttria compositions, the hafnia-yttria compositions from the cubic portion of the phase field outperformed the partially stabilized compositions.

Conclusions

The strong sensitivity of some lots of zirconia-yttria and hafnia-yttria to spray parameters, while other lots are relatively

insensitive, suggests that optimization studies should always investigate a range of spray parameters. For example, future investigations could use three sets of parameters such as the 40/40/4.5, 40/20/4.5, and 40/40/1.5 sets (this notation was defined in the text). Furthermore, the fines could be sieved out of the latter set to promote lower bulk density.

The success of certain compositions combined with certain spray parameter sets suggests that the hafnia-yttria system deserves further investigation. Since the best hafnia-yttria compositions were taken from the fully stabilized cubic phase field, it is possible that the fully stabilized hafnia-yttria would be more stable at higher temperatures (above about 1200 °C (2190 °F)) than the partially stabilized zirconia-yttria compositions that are in use today. However, the strong sensitivity of the current lots of hafnia-yttria to processing parameters makes it difficult to confirm or deny this prediction. Further research into the factors which cause some powders to be sensitive to spray parameters while others are relatively insensitive would be desirable.

Lewis Research Center
National Aeronautics and Space Administration
Cleveland, Ohio, November 12, 1992

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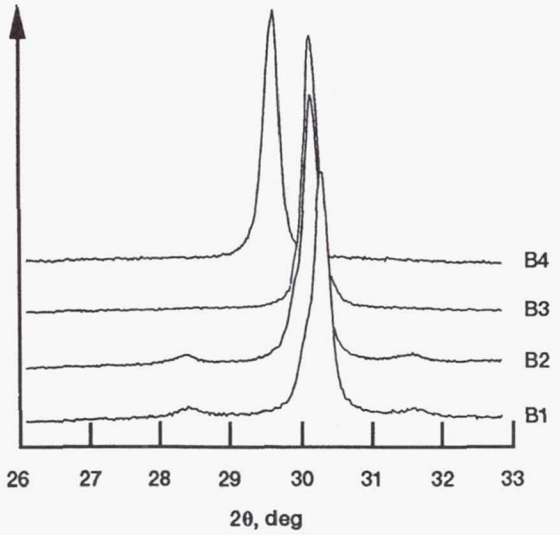
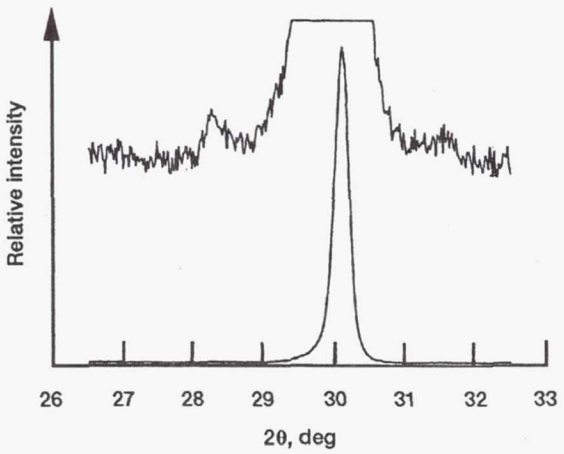
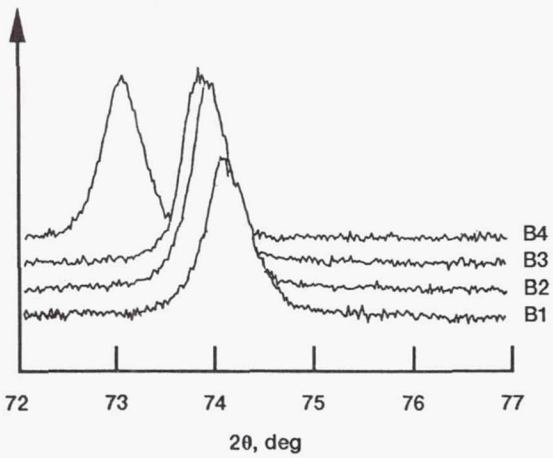


(a) (111) region, lots B1 to B4, 2 sec/0.02° scan.



(b) (111) region, lot B3, 4 sec/0.02° scan.



(c) (400) region, lots B1 to B4.

Figure 1.—X-ray diffraction patterns in the (111) and (400) regions for hafnia-yttria lots B1 to B4.

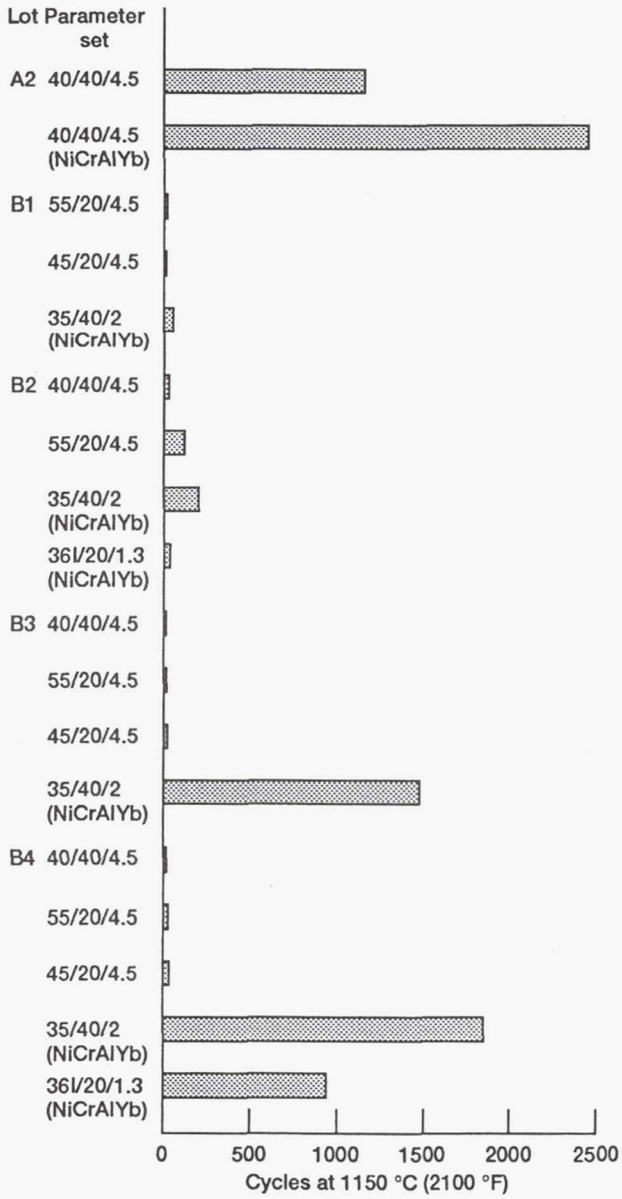


Figure 2.—Burner rig life in rig 1 versus lot and spray parameters for hafnia-yttria and reference zirconia-yttria test specimens (NiCrAlY bond coat except where noted otherwise).

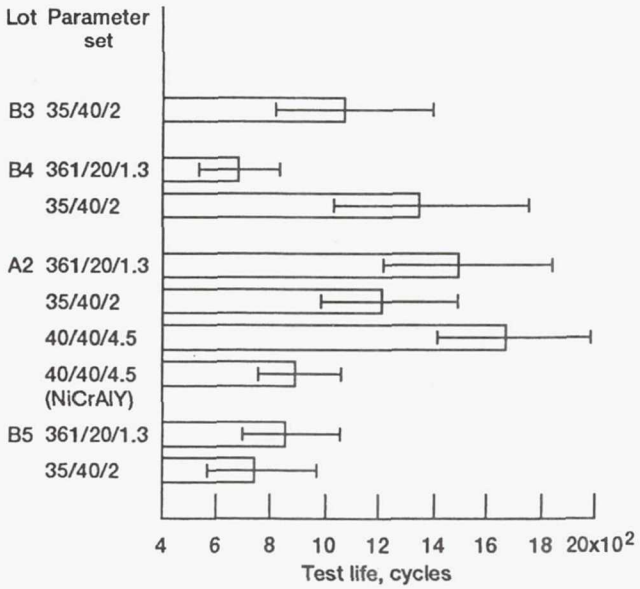
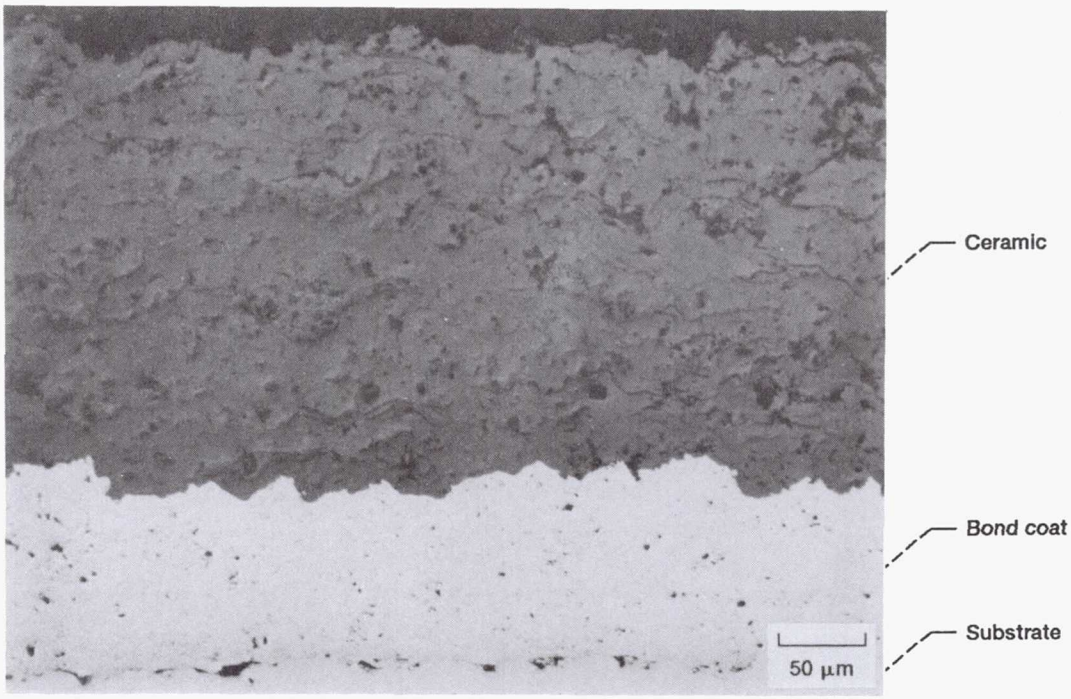
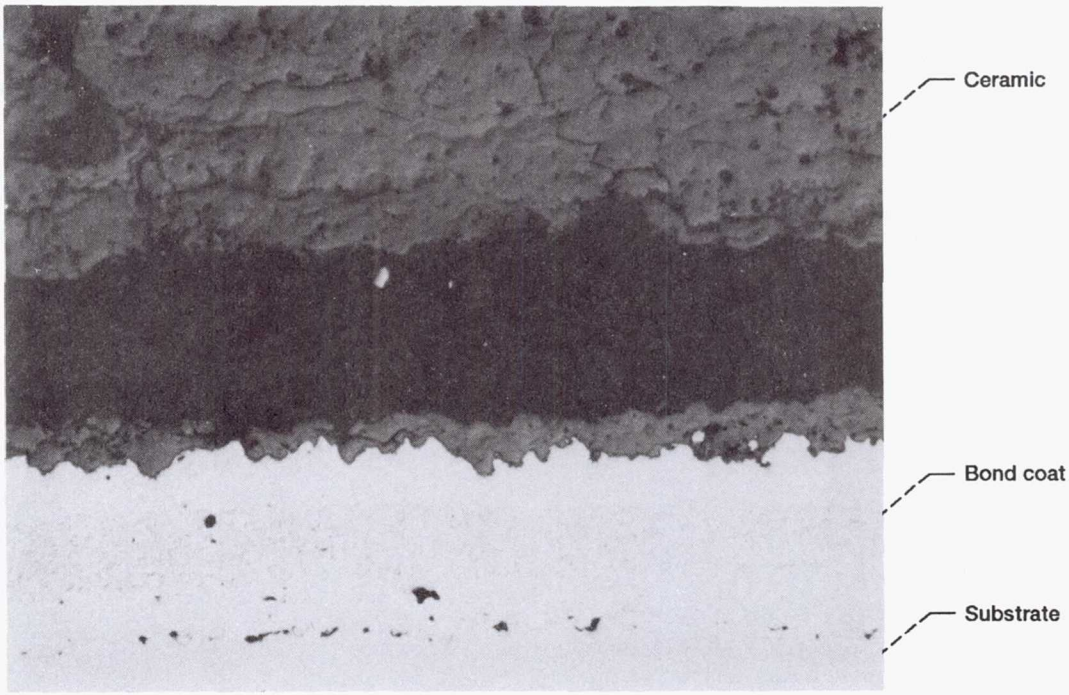


Figure 3.—Plot of means and least significant intervals for hafnia-yttria and reference zirconia-yttria burner rig data (NiCrAlYb bond coat except where noted otherwise).



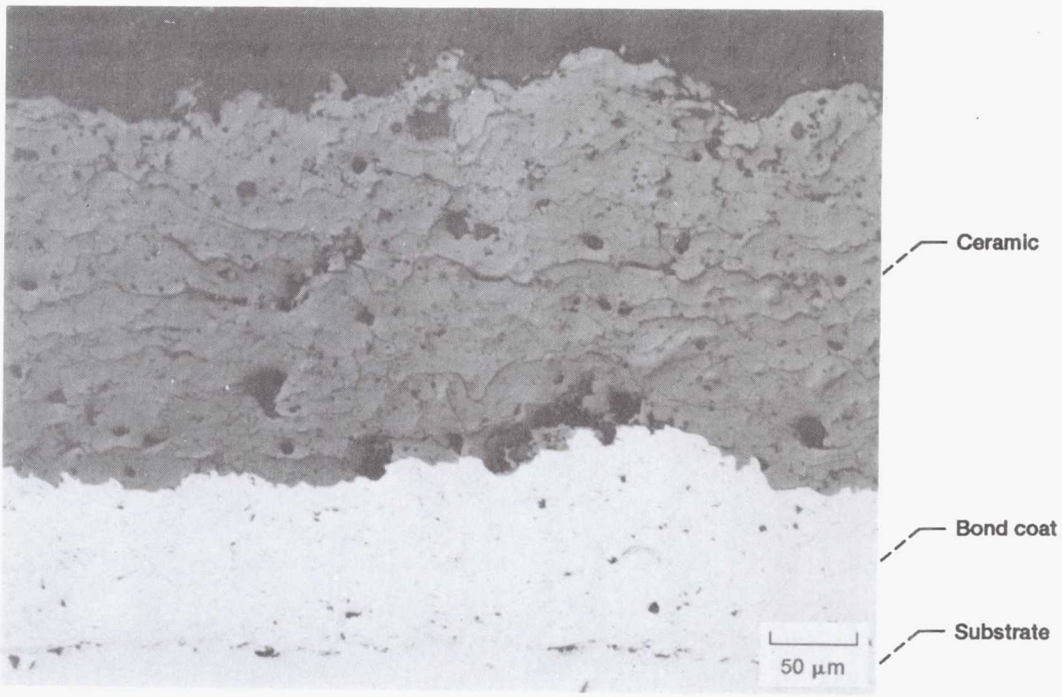
As-sprayed microstructure



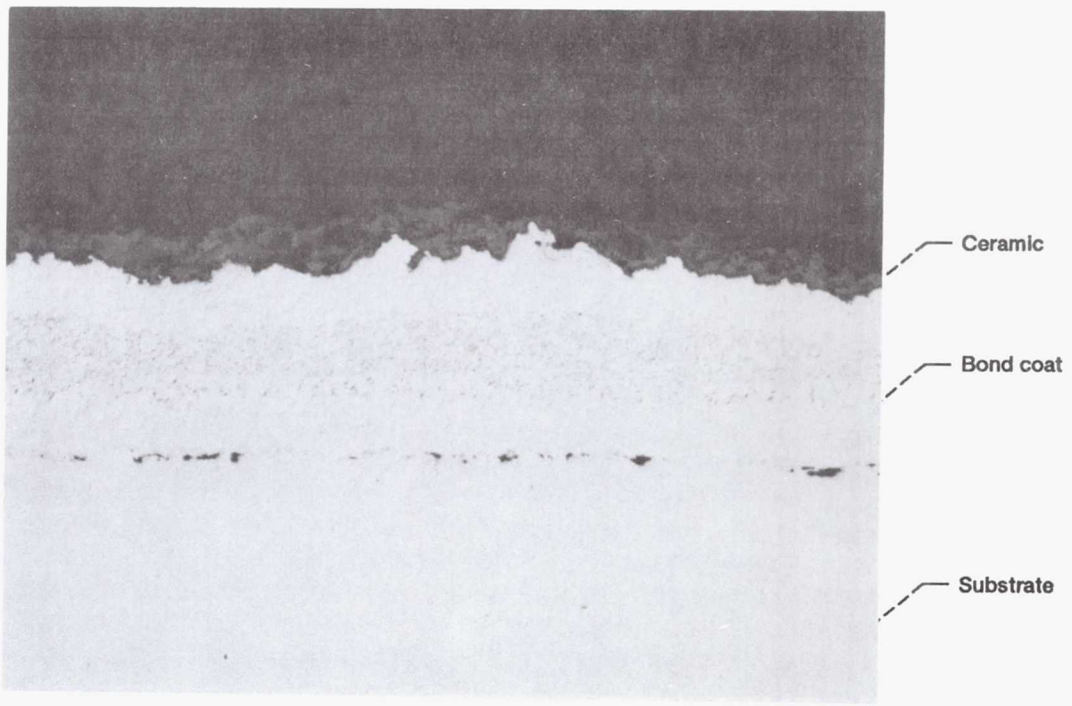
Hot zone after failure

(a) Lot B1, parameter set (35/40/2), order 1.

Figure 4.—Photomicrographs of selected specimens taken near base of test specimen for as-sprayed microstructure and in hot zone after failure. Bond coats are low-pressure plasma-sprayed NiCrAlYb.



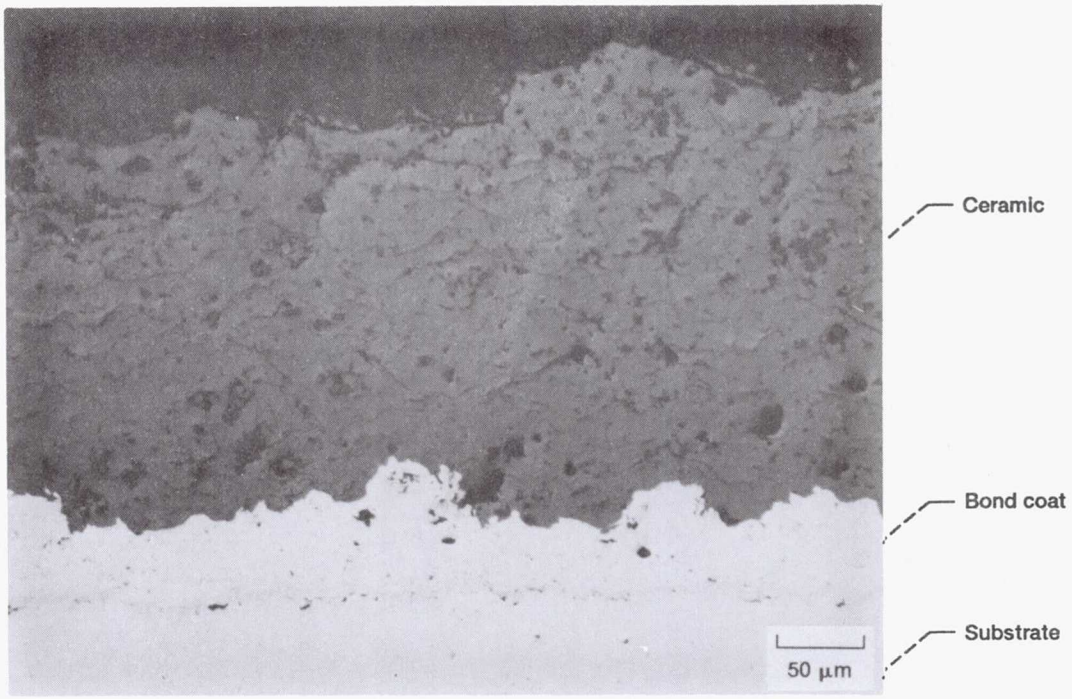
As-sprayed microstructure



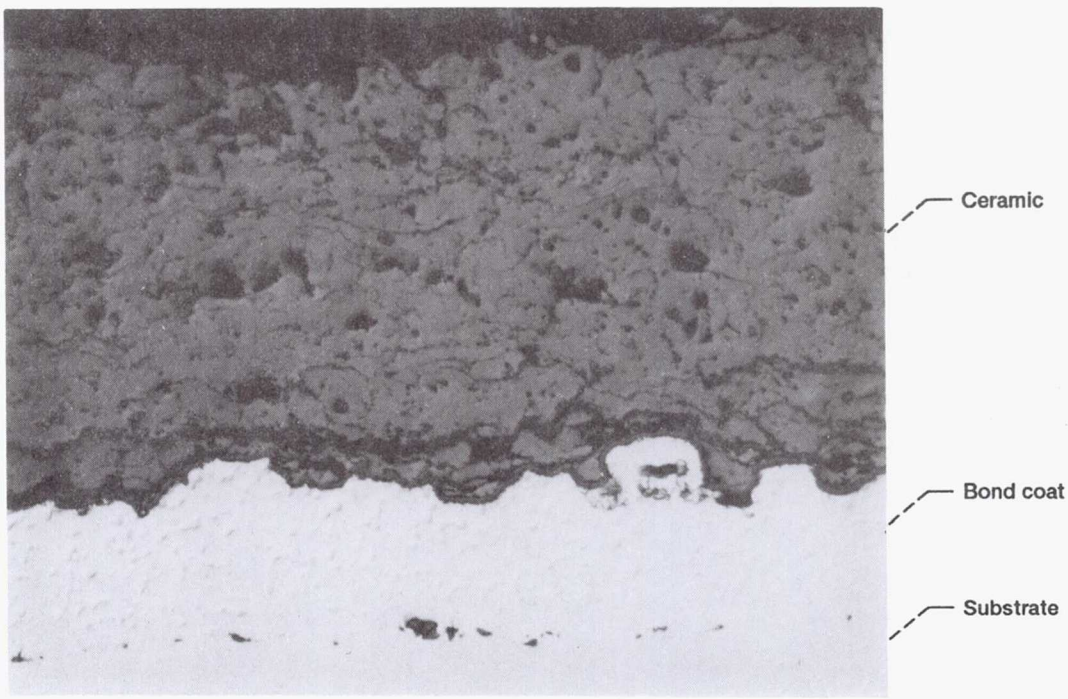
Hot zone after failure

(b) Lot B2, parameter set (35/40/2), order 2.

Figure 4.—Continued.



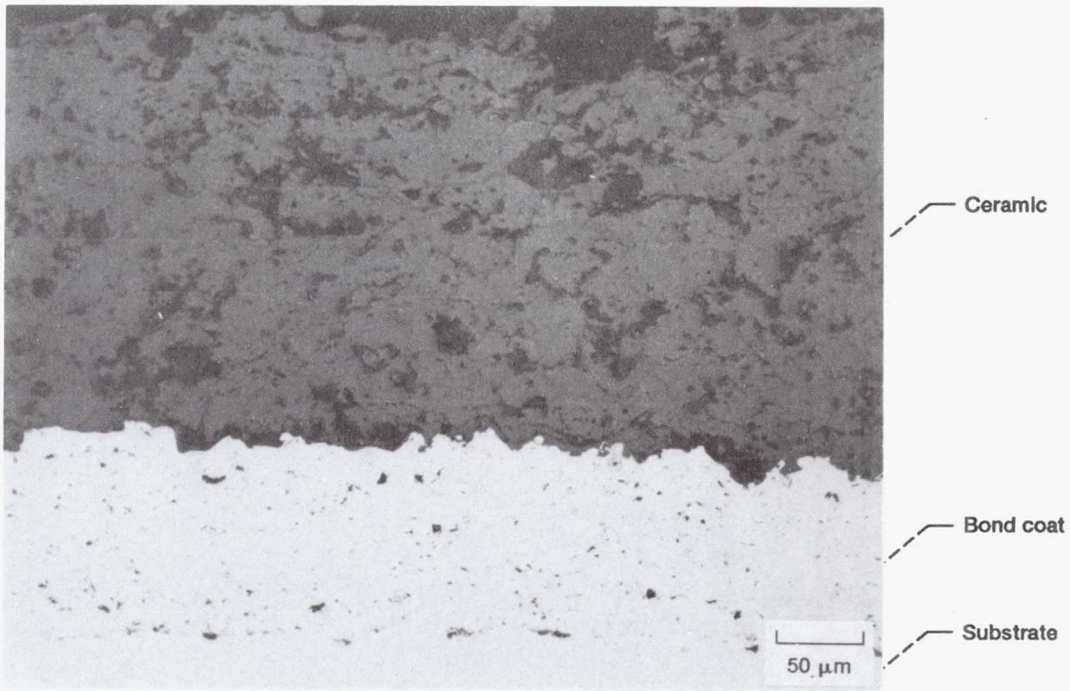
As-sprayed microstructure



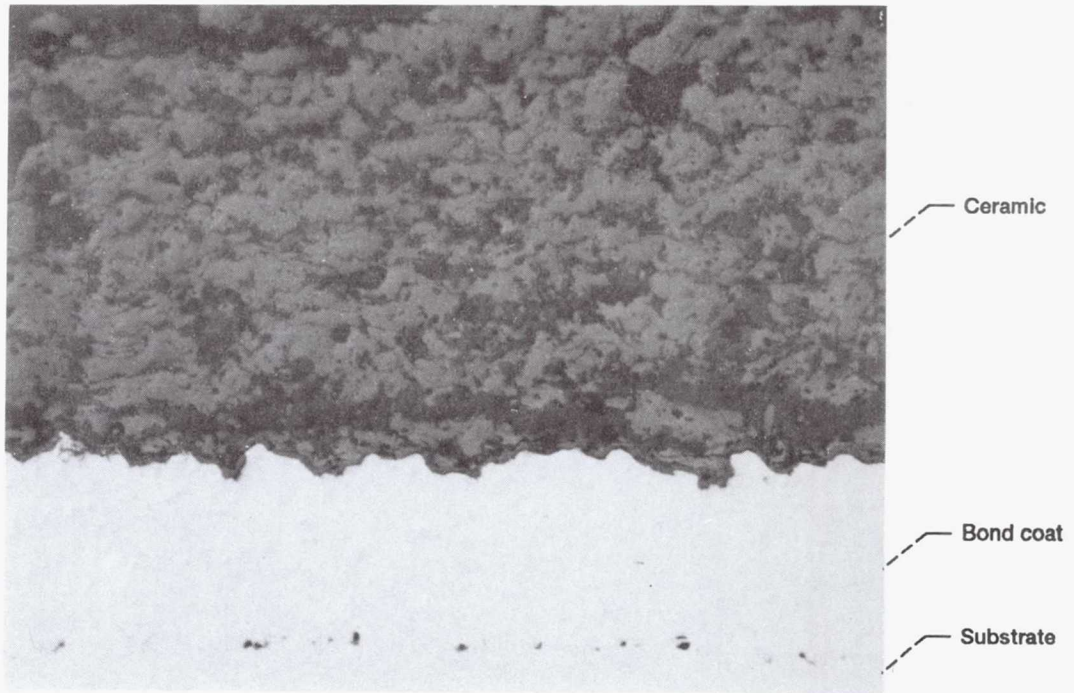
Hot zone after failure

(c) Lot B3, parameter set (35/40/2), order 1.

Figure 4.—Continued.



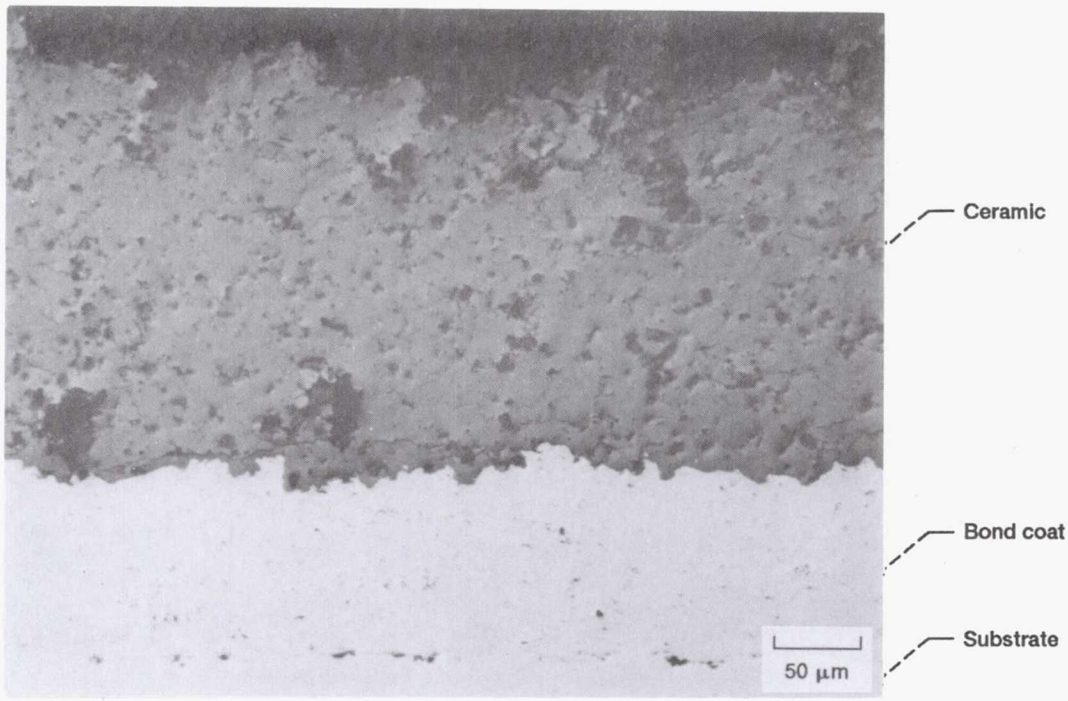
As-sprayed microstructure



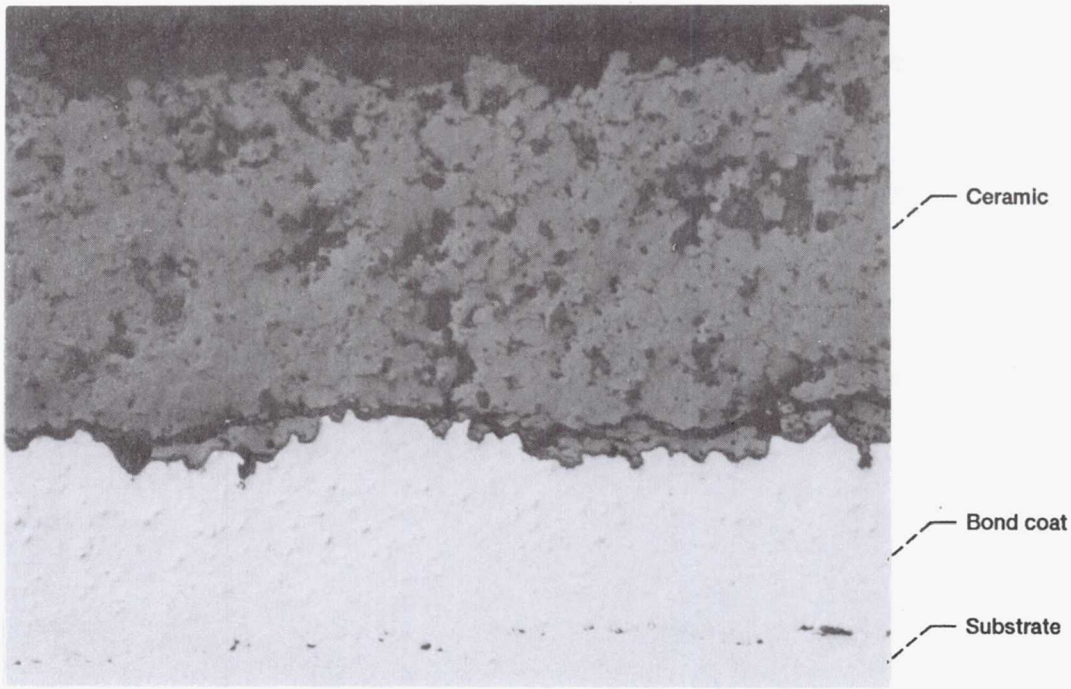
Hot zone after failure

(d) Lot B4, parameter set (35/40/2), order 2.

Figure 4.—Continued.



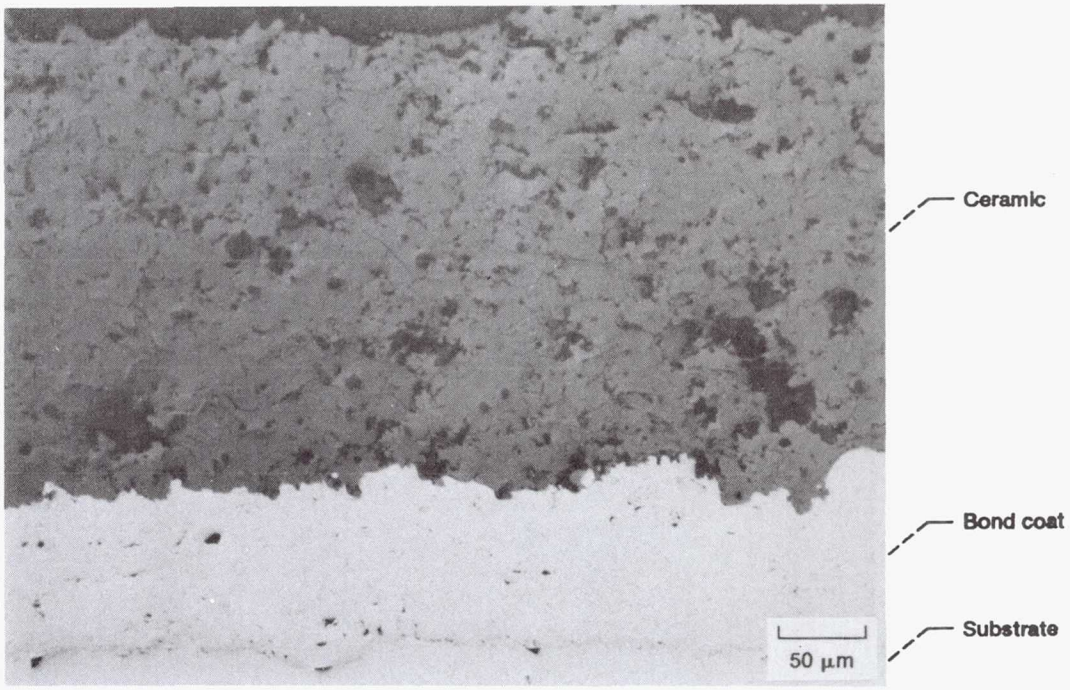
As-sprayed microstructure



Hot zone after failure

(e) Lot B5, parameter set (35/40/2), order 2.

Figure 4.—Continued.

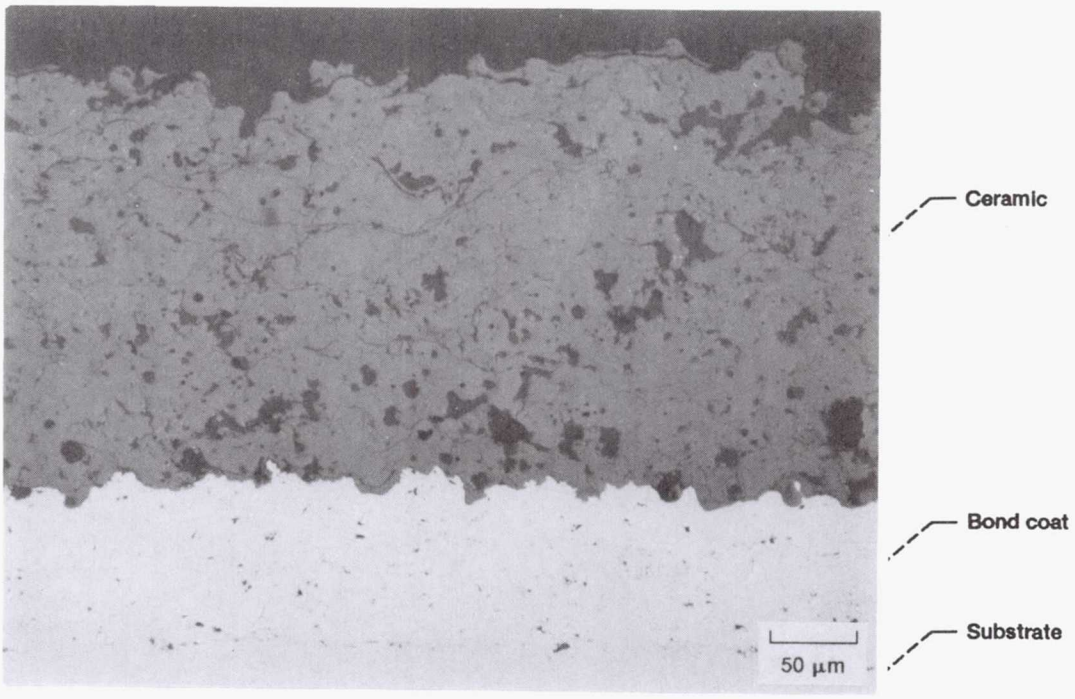


As-sprayed microstructure

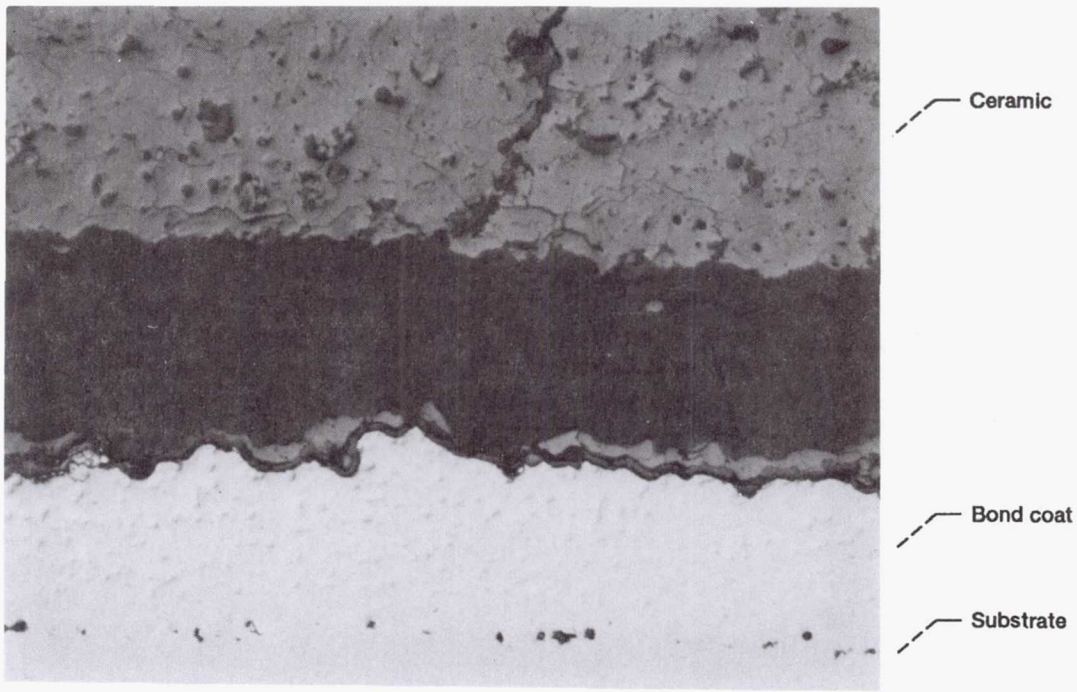
Hot zone after failure

(f) Lot A2, parameter set (35/40/2), order 2.

Figure 4.—Continued.



As-sprayed microstructure



Hot zone after failure

(g) Lot A2, parameter set (40/40/4.5), order 1.

Figure 4.—Concluded.

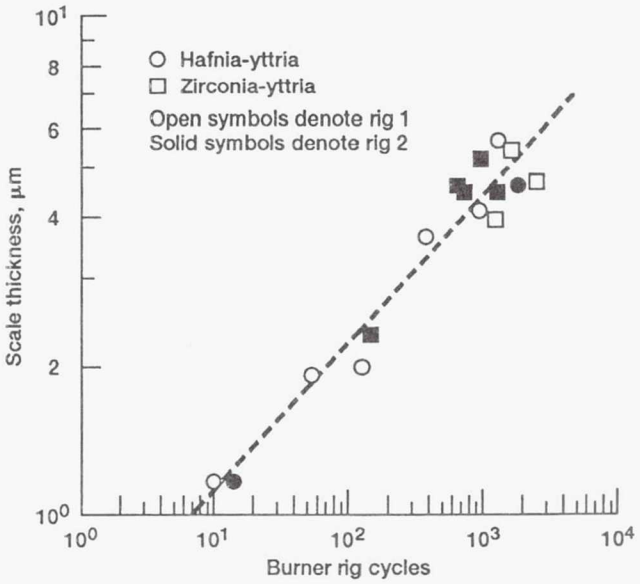


Figure 5.—Log-log plot of scale thickness versus cycles in burner rig.

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13. ABSTRACT ***(Maximum 200 words)***

This is the second of two reports which discuss initial experiments on thermal barrier coatings prepared and tested in newly upgraded plasma spray and burner rig test facilities at Lewis Research Center. The first report, Part I, describes experiments designed to establish the spray parameters for the baseline zirconia-yttria coating. Coating quality was judged primarily by the response to burner rig exposure, together with a variety of other characterization approaches including thermal diffusivity measurements. That portion of the study showed that the performance of the baseline NASA coating was not strongly sensitive to processing parameters. In this second part of the study, new hafnia-yttria coatings were evaluated with respect to both baseline and alternate zirconia-yttria coatings. The hafnia-yttria and the alternate zirconia-yttria coatings were very sensitive to plasma-spray parameters in that high-quality coatings were obtained only when specific parameters were used. The reasons for this important observation are not understood.

14. SUBJECT TERMS

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