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Flame Spray Strain Gages With Improved Durability and Lifetimes

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As the temperature capability of engine components is increased and new materials are developed to meet these new challenges, there is a growing need to assess material behavior in these environments so that structural models can be validated and engine materials can be further characterized. Towards this end, improved flame sprayed strain gages are being developed to meet these instrumentation needs and will be required to operate under more severe thermo-mechanical loadings and at higher temperatures than are possible with existing materials and designs. Specifically, new thermal spray instrumentation will help NASA and the propulsion engine companies meet the future demands of new materials and designs developed under NASA's Aerospace Propulsion and Power (APP) program. The overall program goal is to improve thermal sprayed instrumentation by focusing on ways to stabilize the high temperature mechanical and electrical properties of the two thermal sprayed layers in which the instrumentation is embedded: the metallic bond coat and the ceramic overcoat. Initially, the objective of the program was to extend the thermal fatigue life of commercially available thermal sprayed coatings. Since oxidation of the NiCoCrAlY and NiCrAlY based bond coats is a leading cause of failure in thermal spray instrumentation, a number of different approaches to improve the high temperature oxidation resistance of the thermally sprayed bond coats were investigated.

The first approach utilized different heat treatments in reduced oxygen partial pressures, to selectively oxidize the aluminum and chromium in the bond coat, i.e. as-sprayed bond coats were thermally oxidized in reduced oxygen partial pressures in the temperature range 1600 to 1900 °F. As a result of these heat treatments, the interfacial stresses that develop in the ceramic overcoats during oxidation of the metallic bond coats were dramatically reduced. This was due in part to the presence of both internal and external oxides formed within the surface layers of the bond coat, which resulted in a graded coating, of sorts, whereby the thermal expansion coefficient (TCE) of the bond coat was graded to more closely match that of the ceramic overcoat. Another important benefit of these heat treatments was an overall reduction in the oxidation of the metallic bond coat onto which the dielectric layer was applied. Several other approaches to improve oxidation resistance of the NiCoCrAlY bond coats were investigated, including the development of combinatorial libraries to determine the optimal composition of the starting bond coat powders, platinum diffusion barriers and/or heat treatments and the development of combinatorial libraries to optimize an intermediate coating that would effectively match the TCE of the metal bond coat to the ceramic overcoat at the "rough" or "wavy" interface formed between the bond coat and overcoat. In addition to the improvements made to the thermal sprayed bond coats, the thermal sprayed coatings and surface modifications were evaluated in our recently developed computer-controlled burner rig facility, which is ideally suited for studying the thermo-mechanical properties of the ceramic top coats and bond coats in which instrumentation will be embedded.

Preliminary data collected during the first nine months of the program indicate that the results of this research will substantially increase the lifetime and durability of thermal spray strain gage instrumentation, the industry standard for more than 25 years, at temperatures well above 1000°C. A detailed summary of the key accomplishments completed to date under the APP program is presented below and the major tasks (milestones/benchmarks) that have been accomplished within the proposed time frame are highlighted in italics.

Improved thermal sprayed bond coats via heat treatment in reduced oxygen ambients

The thermal fatigue properties of commercially available thermal sprayed bond coats including Praxair N171 (NiCoCrAlY) and Praxair N343 (NiCrAlY) were evaluated using a retractable Inconel 718 test fixture placed inside a conventional tube furnace as shown in Figure 1. Thermal fatigue lifetime was based on the of number of cycles to failure where each cycle consisted of a 1 hour hold at 1100 °C, rapid cooling to 150 °C (6 to 8 min) and reheating to 1100 °C. The fatigue lifetimes of the as-sprayed Praxair N171 (NiCoCrAlY) and Praxair N343 (NiCrAlY) bond coats were used as a baseline for comparison purposes. The lifetimes of the Praxair N171 (NiCoCrAlY) bond coats that were thermally oxidized in reduced oxygen partial pressures in the temperature range 1600 to 1900 °F, were substantially improved relative to the as-sprayed Praxair N171 bond coats. Of these different heat treatments investigated, those done at 1750 °F in reduced oxygen partial pressures yielded the largest increase in coating lifetime, relative to as-sprayed Praxair N171 coatings (110 cycles to failure vs. 42 cycles to failure). Heat treatment of the Praxair N343 (NiCrAlY) coatings in reduced oxygen partial pressures had little or no effect on coating lifetime at 1100 °C, with the Praxair N343 bond coats only surviving 2 to 3 cycles at 1100 °C, independent of heat treatment and surface treatment. In all cases, a top coat of magnesium aluminate spinel was thermally sprayed onto the Praxair N171 and Praxair N343 bond coats such that two ½ in wide stripes were formed on the surface of the coupon. In this way, more than one thermally sprayed coating could be evaluated per coupon to reduce testing cycle time. A typical heat treatment schedule is shown in Figure 2. It consisted of a series of 20 min ramps and 1 hour holds until the desired temperature was reached. After this temperature was reached, the furnace was ramped down to room temperature at a rate of 3/min until the sample was cool enough to remove from the furnace.

Inconel 718 coupons prepared with thermally sprayed Praxair N171 bond coats survived considerably more cycles to failure than Praxair N343 bond coats, when tested at 1100 °C. For comparison purposes, the as-sprayed Praxair N171 bond coats survived 42 cycles to failure and the heat treated Praxair N171 bond coats survived 79 to 144 cycles to failure at 1100 °C, whereas the Praxair N343 bond coats only survived 2 to 3 cycles to failure at 1100 °C, independent of heat treatment and surface treatment.

A summary of the thermal fatigue test results obtained from our furnace rig is presented in Table 1. Specifically, the lifetimes of the various thermal sprayed bond coats, heat treatments and coatings applied to the bond coats that were thermally cycled from 150 to 1100 °C are summarized in Table 1 below.

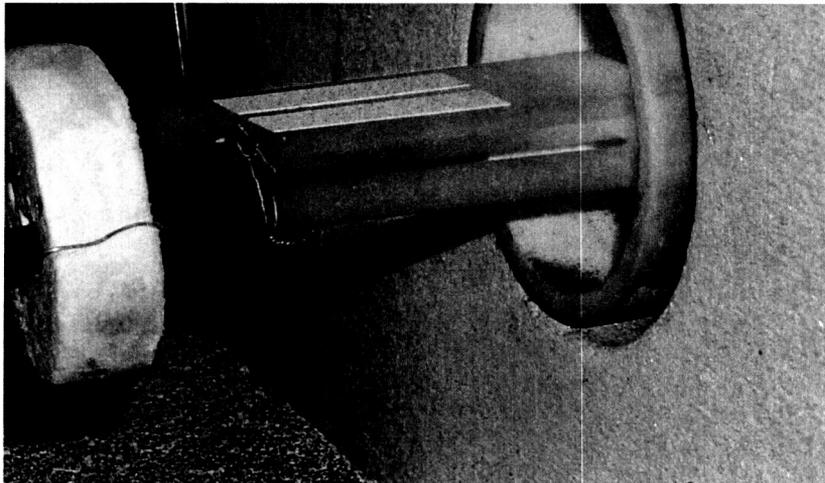


Figure 1. Photograph of an Inconel 718 test coupon being removed from the hot zone of a conventional tube furnace maintained at 1100 °C. Note the retractable Inconel 718 test fixture on which the test coupon was placed and the test coupon itself that was overcoated with 2 stripes of thermally sprayed magnesium aluminate spinel and attached to the test bed by platinum wire. A series of three heat shields was required to maintain the furnace temperature as the coupon was withdrawn from the hot zone and a thermocouple was placed underneath the coupon to monitor temperature.

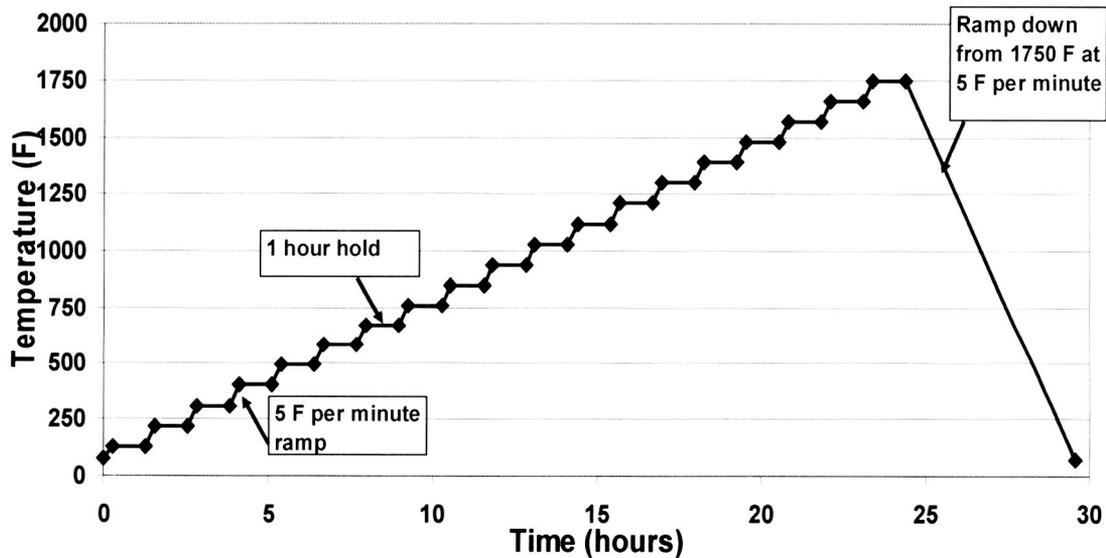


Figure 2. Typical heat treatment schedule for a Praxair N171 bond coat heated to 1750 °F in a reduced oxygen partial pressure ambient. The schedule consisted of a series of 20 min ramps and 1 hour holds until the desired temperature was reached. After reaching the terminal temperature the furnace was ramped down at a rate of 3/min until the Inconel 718 coupon was cool enough to be removed from the furnace.

Table 1. Summary of thermal fatigue test results obtained from our furnace rig to evaluate the different NiCoCrAlY and NiCrAlY bond coats, surface modifications and heat treatments.

Specimen ID	Bond Coat		Precoat	Heat treatment T (F)	Top coat type	Thermal cycling temp.			Comments
	type	thickness (inches)				T (C)	dwll (h)	cycles to failure	
2-N171-0216	NiCoCrAlY	0.002	none	1600	Rokide	1050	1	12	TC debonded from BC
2-N171-0216-p	NiCoCrAlY	0.002	Pt	1600	Rokide	1050	1	(42)	Cycling interrupted; No failure;
2-N343-0216	NiCrAlY	0.002	none	1600	Rokide	1150	1	2	The top coat failed during cooling at about 350 C; BC turned greenish; (possibly chromium oxide)
2-N343-0216-p	NiCrAlY	0.002	Pt	1600	Rokide	1100	1	2	TC debonded from BC
2-N171-0222	NiCoCrAlY	0.002	none	1750	Rokide	1100	1	79	TC debonded from BC
2-N171-0222-p	NiCoCrAlY	0.002	Pt	1750	Rokide	1100	1	81	TC debonded from BC
2-N343-0222	NiCrAlY	0.002	none	1750	Rokide	1100	1	2	TC debonded from BC
2-N343-0222-p	NiCrAlY	0.002	Pt	1750	Rokide	1100	1	1	TC debonded from BC
2-N171-0228-2p	NiCoCrAlY	0.002	2h Pt	1800	Rokide	1100	1	192	TC debonded from BC
2-N343-0228-2p	NiCrAlY	0.002	2h Pt	1800	Rokide	1100	1	6	TC debonded from BC
2-N171-0305-2p	NiCoCrAlY	0.002	2h Pt	1750	Rokide	1100	1	124	TC debonded from BC
2-N343-0305-2p	NiCrAlY	0.002	2h Pt	1750	Rokide	1100	1	7	TC debonded from BC
2-N171-0306-6Al	NiCoCrAlY	0.002	6h Al ₂ O ₃	1800	Rokide	1100	1	94	TC debonded from BC
2-N343-0306-6Al	NiCrAlY	0.002	6h Al ₂ O ₃	1800	Rokide	1100	1	3	TC debonded from BC
2-N171-0307	NiCoCrAlY	0.035	none	1750	Rokide	1100	1	99	TC debonded from BC
2-N343-0307	NiCrAlY	0.003	none	1750	Rokide	1100	1	25	TC debonded from BC
2-N171-0	NiCoCrAlY	0.002	none	none	Rokide	1050	1	52	Baseline sample
2-N343-0	NiCrAlY	0.002	none	none	Rokide	1100	1	2	Baseline sample
2-N171-0-B	NiCoCrAlY	0.005	none	none	Rokide	1100	1	143	Baseline sample
2-N171Ce-0501	NiCoCrAlY + 0.5% Ce	0.002 N171 0.001 0.5% Ce	none	1750	Rokide	1100	1	43*	*Sample prematurely failed after cooling to 60 C
2-N171-0501-B	NiCoCrAlY	0.003	none	1750	Rokide	1100	1	124	TC debonded from BC
2-N171-0501-p-B	NiCoCrAlY	0.003	Pt	1750	Rokide	1100	1	74*	*TC failure after cooling to RT when furnace shutdown after power outage
2-N171-0610-C	NiCoCrAlY	0.003 - .004	none	1750	Rokide	1100	1	144	TC debonded from BC

2-N171Si-0610	NiCoCrAlY + 1% Si	0.002 - .004	none	1750	Rokide	1100	1	49*	* Sample failed over the 49th cycle is last time when sample was known to be intact
2-N171Ce-0610-B	NiCoCrAlY + 0.5% Ce	0.0025 - 0.003	none	1750	Rokide	1100	1	71*	*Sample prematurely failed after cooling to 68 C
2-N171Ce-C	NiCoCrAlY + 0.5% Ce	0.002- 0.003	none	none	Rokide	1100	1	49	TC debonded from BC
2-N171-0-C	NiCoCrAlY	0.003	none	none	Rokide	1100	1	55	TC debonded from BC
2-N171Ce-0715	NiCoCrAlY + 0.5% Ce	0.0025	none	1750	Rokide	1100	1	47	TC debonded from BC
2-N171Ce1-0715	NiCoCrAlY + 1.0% Ce	0.003	none	1750	Rokide	1100	1	69	TC debonded from BC; TC started to buckle in the center of the strips
2-N171-0-D	NiCoCrAlY	0.003	none	none	Rokide	1100	1	71	014 HA; TC debonded from BC

Evaluation of platinum (and rhodium) diffusion barrier coatings for NiCoCrAlY bond coats

Platinum diffusion barriers substantially increased the lifetimes of thermal sprayed coatings when used in conjunction with heat treatments in reduced partial pressures of oxygen. Platinum coatings applied to the surfaces of N171 bond coats by rf sputtering and heat treated at 1800 °F in reduced oxygen partial pressures, yielded the highest number of cycles to failure (192 cycles to failure) at 1100 °C. A thermal sprayed N171 bond coat with a platinum diffusion barrier applied to the surface was the best performer of all the coatings and heat treatments investigated to date and represented an increase in the fatigue life of Praxair N171 coated samples of over 200%. By comparison, the as-sprayed N171 bond coats only survived 42 cycles to failure at 1100 °C and the N343 bond coated specimens only survived 2 to 3 cycles to failure at 1100 °C, regardless of heat treatment and surface modification. The sputtered platinum films were on the order of 2 µm in thickness and proved to be sufficiently thick to act as an effective oxygen diffusion barrier, slowing the growth of the internal oxides and promoting an alumina rich outer scale on the surface of the N171 bond coat.

In a similar series of experiments, rhodium diffusion barriers were investigated to try and extend the lifetimes of the thermal sprayed N171 bond coats. However, these experiments were done with electroplated rhodium coatings and not sputtered coatings as was the case for the platinum diffusion barriers. The reason being, that this plating technique could be readily transferred to bench level technicians without the need for specialized training and deposition equipment. In essence, this pen plating process was used in conjunction with a special rhodium plating solution that was developed for the kind of control needed for this precision plating process. The results were not as

dramatic as those obtained from the platinum diffusion barriers prepared by sputtering but they resulted in a substantial increase in bond coat lifetime relative to the as-sprayed N171 bond coats. Ultimately, a pen plating process is envisioned for the platinum diffusion barriers but is still under development at this time.

Evaluation of alternative coatings and additives to NiCoCrAlY bond coats

Several other approaches to improve the oxidation resistance of the NiCoCrAlY bond coats were investigated, including the use of PVD alumina coatings to slow the growth of the internal oxides formed within the surface layers of the N171 bond coats. When Al₂O₃ coatings on the order of 1.5 μm in thickness were deposited onto Praxair N171 bond coats and heat treated in reduced oxygen partial pressures at 1800 °F, a substantial increase in fatigue life was observed relative to the as-sprayed Praxair N171 substrates (94 cycles to failure vs. 42 cycles to failure), as shown in Table 1. However, when PVD Al₂O₃ coatings on the order of 1.5 μm in thickness were deposited onto the N343 bond coated coupons, they only survived 3 cycles to failure at 1100 °C, as shown in Table 1 of this report.

In the literature, partially stabilized zirconia (PSZ) thermal barrier coatings were applied to superalloy substrates by a combination of PVD and thermal spraying to achieve improved adherence and oxidation resistance. The thermo-mechanical behavior of these coated superalloys at high temperature was dramatically improved using this approach. This study provided the motivation to use a combination of PVD and thermally sprayed alumina to improve the fatigue life of the Inconel 718 coupons bond coated with Praxair N171 by improving the adhesion and oxidation resistance. Recent experiments employing 5 to 20 μm thick Al₂O₃ coatings have indicated that thermal fatigue life of the thermal sprayed coatings can be further improved by using thicker pre-coats of alumina. The thermal fatigue results of thermal sprayed coatings employing these thicker alumina diffusion barrier coatings are presented in a latter section of this report where a computer controlled burner rig was used to evaluate the lifetime of the coatings.

In addition to the approaches described above to improve oxidation resistance of the NiCoCrAlY bond coats, metallic powders were added to the bond coat powders prior to spraying to improve the fatigue life of the Praxair N171 bond coats. These additives showed only marginal improvement in fatigue life. Specifically, silicon additions (1 wt%) to the Praxair N171 powders in conjunction with heat treatments to 1750 °F in reduced O₂ partial pressures showed little or no improvement in the fatigue life of the Inconel 718 coupons tested at 1100 °C. Similarly, ceria additions (0.5 to 1wt%) in conjunction with heat treatments to 1750 °F in reduced O₂ partial pressures yielded a marginal increase in the fatigue life of Praxair N171 coated samples (69 cycles to failure vs. 63 cycles to failure) as shown in Table 1 of this report. In all cases, a ceramic top coat of magnesium aluminate spinel was thermally sprayed onto the Praxair N171 bond coats.

A computer controlled burner rig for evaluating new thermal sprayed coatings

Two different test beds were used to evaluate the lifetimes of the thermal sprayed Inconel 718 coupons. Initially, testing of the thermal sprayed coupons was carried out in retractable Inconel 718 test fixture placed inside a conventional tube furnace. Thermal fatigue tests were based on the number of cycles to failure and consisted of a 1 hour hold at 1100 °C, followed by rapid cooling to 150 °C and then reheating to 1100 °C. More recently, a computer-controlled, burner rig (oxygen fueled torch) was designed and constructed to evaluate the various bond coats (and heat treatments) under conditions more similar to jet engine behavior. These tests consisted of rapid heating of the thermal sprayed ceramic top coat (front of Inconel 718 coupon) to 1150 to 1200 °C in an open flame, holding in the flame for 60 min followed by rapid cooling to 150 °C (3 min) and then reheating to 1150 to 1200 °C. A 150 °C temperature difference was maintained between the front of the Inconel 718 coupon and the back of the Inconel 718 coupon. The surface temperature of the ceramic was continuously monitored using an optical pyrometer. The burner rig provided a more realistic testing environment to evaluate the combination of the thermal sprayed bond coats and top coats while the furnace tests were employed to study sintering and oxidation effects of the various thermal spray coatings but did not have the severity of the burner rig tests. A photograph of an Inconel 718 test coupon overcoated with 2 stripes of thermally sprayed magnesium aluminate spinel and placed in front of an oxygen fueled torch is shown in Figure 3. The coupon shown in Figure 3 was periodically moved in and out of the flame via our computer-controlled burner rig. A block diagram of the computer controlled burner rig with the components comprising the apparatus is shown in Figure 4 and a photograph of the actual computer-controlled burner rig used to evaluate the thermal fatigue properties of the various coatings is shown in Figure 5. Here, the various components comprising the rig are visible, including the oxygen fueled torch and a small fan designed to cool the coupon from the test temperature within a few minutes. Located underneath the table are a variety of motors, controllers and interface boxes that control the movement of the Inconel 718 test coupon on the rail.

A summary of the thermal cycling test results obtained on our computer controlled burner rig showing the lifetimes of the various thermal sprayed coatings and treatments at 1150 C is presented in Table 2. Only Praxair N171 (NiCoCrAlY) bond coats were evaluated using this much more severe testing protocol. Those heat treatments and coatings applied to the Praxair N171 (NiCoCrAlY) bond coats that showed the most promise in terms of improving the fatigue life were repeated on the computer controlled burner rig; i.e. not all of the Inconel 718 test coupons bond coated with NiCoCrAlY were evaluated using this burner rig.

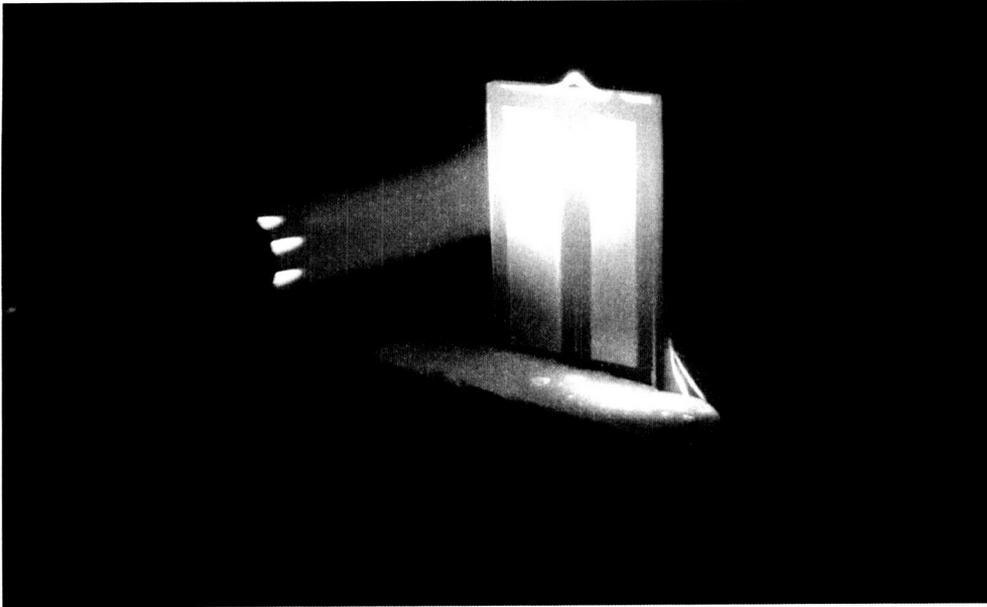


Figure 3. Photograph of an Inconel 718 test coupon overcoated with 2 stripes of thermally sprayed magnesium aluminate spinel and placed in front of an oxygen fueled torch. The coupon is moved into and out of the flame via the computer-controlled burner rig.

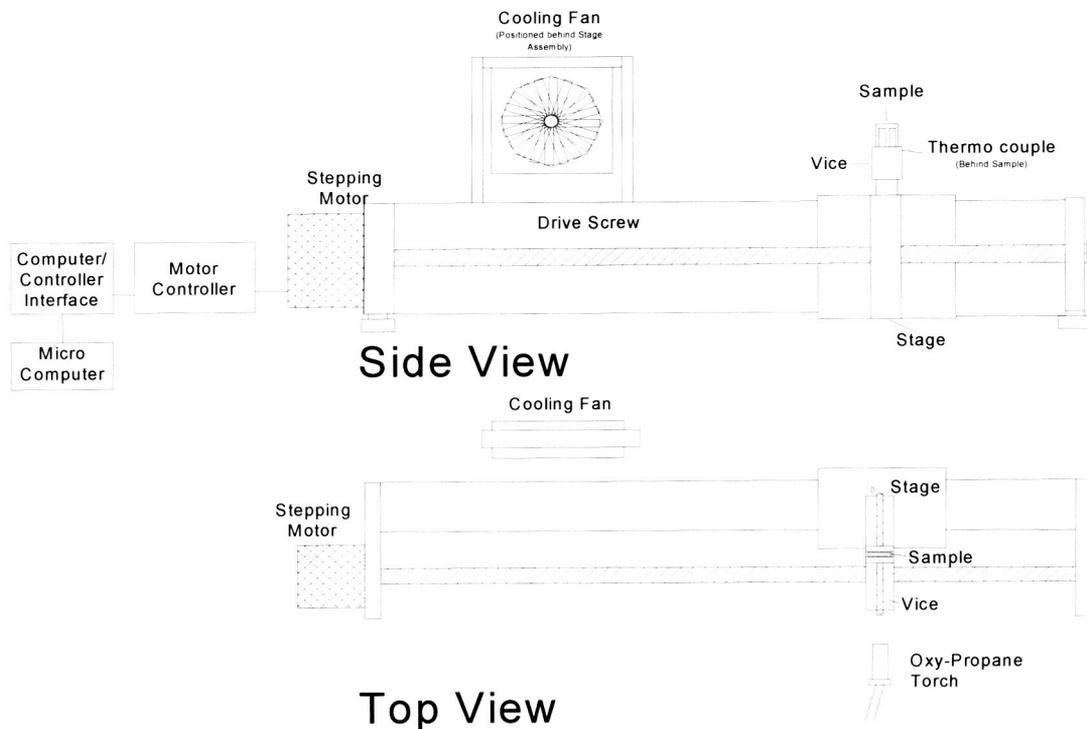


Figure 4. Schematic of our recently developed computer-controlled burner rig showing the various components comprising the apparatus. The rail mechanism, including the motors, controllers and interface boxes that control the movement of the Inconel 718 test coupon are also shown.

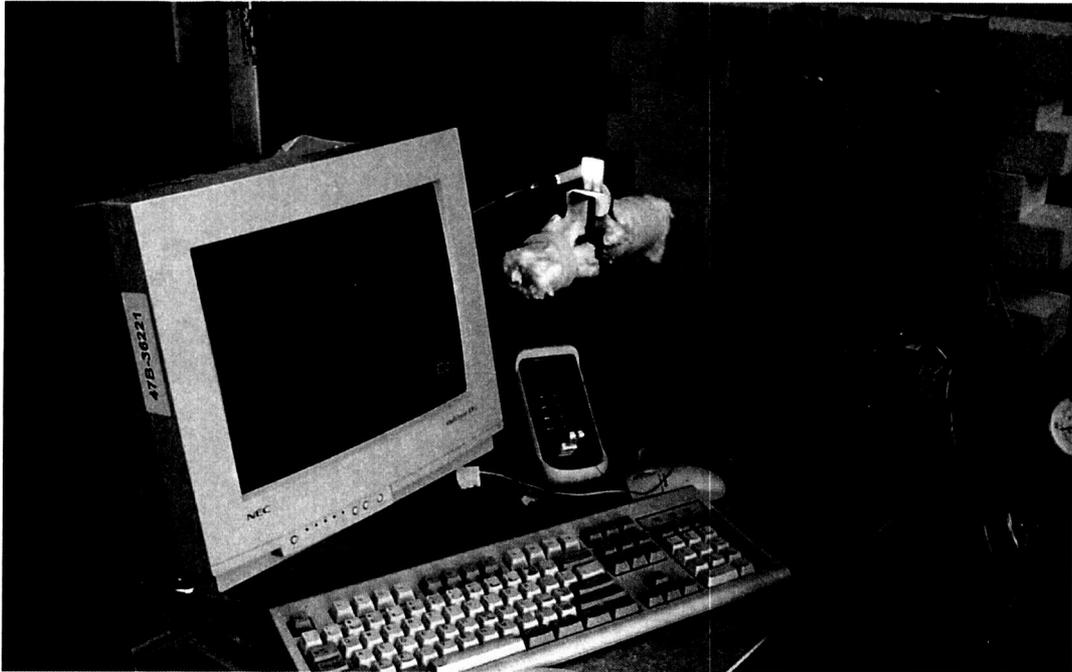


Figure 5. Photograph of our recently developed computer-controlled burner rig showing the various components comprising the apparatus, including the oxygen fueled torch. Located underneath the table are a variety of motors, controllers and interface boxes that control the movement of the Inconel 718 test coupon on a rail. Also pictured is a small fan that is designed to cool the coupon from the test temperature within a few minutes.

Presentation of key accomplishments at PWIG meetings (May 2002 and October (2002)

An initial review of key accomplishments within the first five months of NASA's APP program to improve thermal sprayed instrumentation was presented to members of the Ohio Aerospace Institute's Propulsion Instrumentation Working Group (PWIG) in San Diego, CA, at the May 2002 meeting. A second review of key accomplishments within the first 10 months of the program will be made at the October 2002 meeting of the Ohio Aerospace Institute's Propulsion Instrumentation Working Group (PWIG) in Morgantown, WV. The improvements in thermal spray instrumentation made over the first five months of the program were well received at the first PWIG meeting and all indications to date are that the temperature range and reliability of the new thermal spray instrumentation will be extended well beyond the current state of the art with the improvements made thus far in this program.

Table 2. Summary of thermal fatigue test results obtained from our computer controlled burner rig. Only Praxair N171 (NiCoCrAlY) bond coats, associated surface modifications and heat treatments were evaluated using this testing protocol.

Specimen ID	Bond Coat		Surface Treat	Heat treat T (F)	Thermal cycling					Comments
	type	thickness (inches)			type	thickness (inches)	T (C)	dwll (h)	cycles to failure	
2R-N171-0	NiCoCrAlY	0.0035	none	none	Rokide	0.016 - 0.017	1200	1	2	Sample previously fatigued using 1 min heating and 3.5 min cooling cycles
2R-N171-0-B	NiCoCrAlY	0.003 - 0.004	none	none	Rokide	0.018	1200	1	1	Failure during cooling from 1st cycle
2R-N171-0805	NiCoCrAlY	0.0035 - 0.004	none	1750	Rokide		1200	1	1	Failure during heating to 2nd cycle
2R-N171-0805-B	NiCoCrAlY	0.005 - 0.006	none	1750	Rokide		1150	1	1	Failure during heating to 2nd cycle
2R-N171-0811-2p	NiCoCrAlY	0.003 - 0.0035	2h Pt	1800	Rokide		1150	1	1	Failure during cooling from 1st cycle
2R-N171-0811-2p-B	NiCoCrAlY	0.003	2h Pt	1800	Rokide		1150	1	2	Failed during heating to 3rd cycle
2R-N171-0-C	NiCoCrAlY	0.007	none	none	Rokide	0.014	1150	1	4	Spalling of TC during cooling from 4th cycle
2R-N171-0-D	NiCoCrAlY	0.006	none	none	Rokide	0.014	1150	1	4	Decomposition of TC during cycle
2R-N171-0909-Rh	NiCoCrAlY	0.007	1 layer Rh pen plating	1800	Rokide	0.013-0.014	1150	1	5	Gradual spalling of TC during cycling; Extensive blistering during 4th & 5th cycle
2R-N171-0909-2Rh	NiCoCrAlY	0.007-0.009	2 layers Rh pen plating	1800	Rokide	0.013-0.014	1150	1	5	Extensive blistering apparent during 4th cycle, limited spalling on heating and cooling;
2R-N171-0909-20Al-B	NiCoCrAlY	0.003	20h Al ₂ O ₃	1800	Rokide	0.014-0.017	1150	1	10	Extensive spalling of the TC
2R-N171-0909-20Al	NiCoCrAlY	0.003	20h Al ₂ O ₃	1800	Rokide	0.014-0.017	1150	1	4	Extensive spalling of the TC

2R-N171-0904-2p-C	NiCoCrAlY	0.004-0.005	2h Pt	1800	Rokide	0.016	1150	1	6	Extensive spalling and blistering in contact area
2R-N171-0904-2p-D	NiCoCrAlY	0.004	2h Pt	1800	Rokide	0.017	1150	1	8	Strong degradation of TC
W	NiCoCrAlY	0.004	none	none	100% Alumina	0.014 HT	1150	1	6	TC debonded from BC during cooling from 6th cycle
X	NiCoCrAlY	0.003	none	none	100% Alumina	0.014 HT	1150	1	24*	*No failure or degradation of the TC after three interruptions
Y	NiCoCrAlY	0.003	none	none	Rokide + EZ TBC	0.015	1150	1	1	catastrophic TC failure upon heating to 2nd cycle; TC peeled off; EZ TBC sprayed from 5" distance
Z	NiCoCrAlY	0.003	none	none	Rokide + EZ TBC	0.015	1150	1	6	Failure on heating to 6th cycle; inside corners of TC started peeling off

Optimization of bond coat chemistry and intermediate TCE coating chemistry using combinatorial libraries.

Two types of combinatorial chemistry experiments were conducted to improve the fatigue lifetimes and electrical properties of the thermal sprayed bond coats. The first combinatorial approach to optimize bond coat chemistry was to reformulate the composition of the starting NiCoCrAlY powders, such that the electrical properties of the thermally sprayed bond coat could be optimized for dielectric strength; i.e. a combinatorial method for synthesizing thin film libraries that could be rapidly screened by electrical measurements (probing the surface of each library) to identify optimal compositions that can lead to improved dielectric strength. To accomplish this polished Inconel 718 substrates were placed in between an aluminum target and a NiCoCrAlY target such that different compositions could be achieved by co-sputtering into well-defined areas via a shadow mask to form the combinatorial library.

A schematic of the combinatorial library, complete with the location of the substrates and spatial arrangement of the shadow masks is shown in Figure 6. In this way, a wide range of alloys were produced on the Inconel 718 coupons depending on the distance (spatial arrangement of the shadow mask) from each sputtering target as shown in Figure 7. The specimens were oxidized under reduced oxygen partial pressures at 1750 °F after which each combinatorial library was tested for dielectric strength using a Kiethley picometer/voltage source. The breakdown voltage was determined by measuring the

current as the voltage was ramped from zero to the breakdown voltage in 10 volt increments. At the breakdown voltage, the current rapidly increased by orders of magnitude. The areas showing the greatest dielectric strength were analyzed for chemical composition by energy dispersive analysis of x-rays (EDS). Those compositions will be used as starting point in preparing new thermal spray powders. The results of combinatorial approach to optimize dielectric strength of the combinatorial library produced by co-sputtering aluminum/NiCoCrAlY films (formed on Inconel 718 substrates) are shown in Figure 6. Based on this analysis, intermediate combinations of aluminum/NiCoCrAlY provided the largest dielectric strengths of all combinations tested in this manner. The chemical analysis using energy dispersive analysis of x-rays (EDS) to determine the optimal chemical composition for dielectric strength has recently been completed. It was determined that alloys containing ~ 20% Al as compared to 13% Al in the NiCoCrAlY provided the greatest dielectric strength.

A second combinatorial approach to optimize the chemistry of an intermediate TCE coating was also completed. Here the basic idea was to match the TCE of the metallic and ceramic layers at the wavy interface formed by the thermal sprayed coatings. This requires mixing the two materials in a ratio that will minimize the thermal stresses in the wavy interface. It is generally agreed upon in the literature that these thermal sprayed coatings fail as a result of thermal expansion mismatch between the ceramic and metallic layers in these coating systems. To form the combinatorial library, Inconel 718 coupons were thermally sprayed with Praxair N171 bond coats and were used as substrates. These substrates were placed between an aluminum oxide target and a NiCoCrAlY target and the material was co-sputtered into well-defined areas via a shadow mask to form a combinatorial library, as shown in Figure 8. Thus, a wide range of alloy compositions was produced, depending on the distance (spatial arrangement of the shadow masks) from each sputtering target as shown in Figure 9. These segmented regions or combinatorial libraries were thermally sprayed with a magnesium aluminate top coat and subjected to repeated thermal cycling (from 150 to 1100 °C) in our furnace rig to determine which compositions minimize cracking of the ceramic top coat, i.e. optimize the composition of the intermediate coating that leads to minimal thermal expansion differences between the peaks and valleys of the wavy interface formed by the thermal sprayed bond coat. The combinatorial library showing the least cracking was analyzed for chemical composition by energy dispersive analysis of x-rays (EDS). The optimized composition will be made into a new powder mix that will be thermally sprayed as a thin layer on the N171 bond coat to affect the TCE mismatch at the very "irregular" bond coat/ top coat interface. The chemical analysis using energy dispersive analysis of x-rays (EDS) is still underway to determine the optimal chemical composition of the intermediate TCE coating.

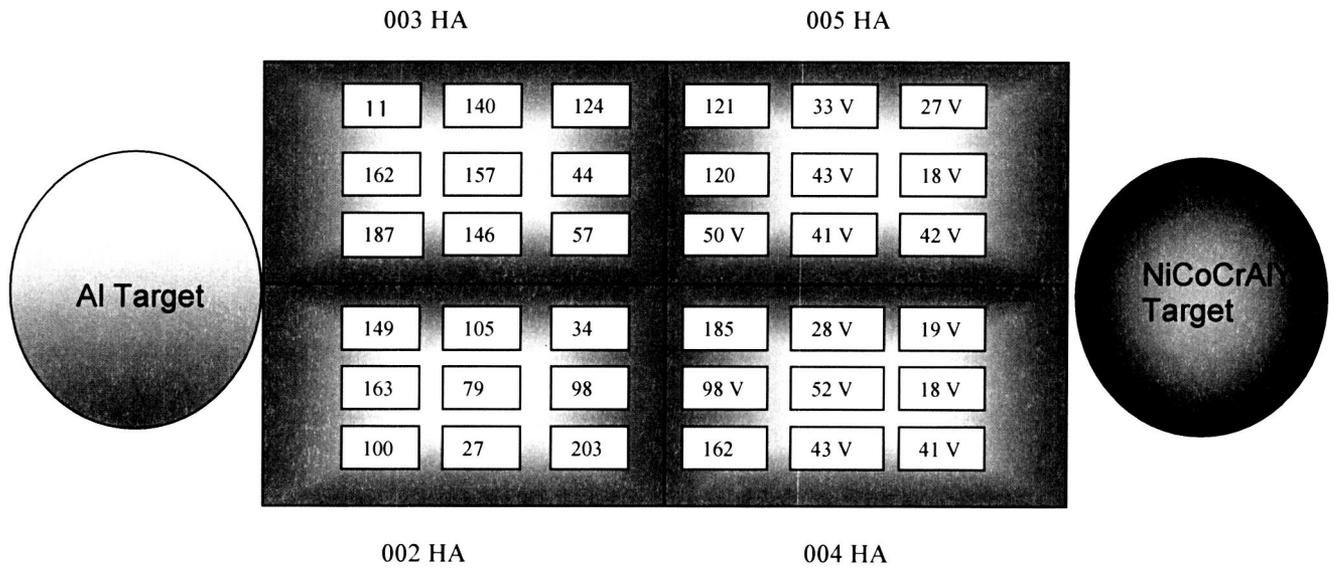


Figure 6. Results of combinatorial chemistry experiments to evaluate dielectric strengths (breakdown voltages) of the combinatorial libraries formed by co-sputtering aluminum and NiCoCrAlY on Inconel 718 substrates. The breakdown voltages associated with each combinatorial library are included in the schematic. The boxed numbers correspond to the breakdown voltages within each combinatorial library.

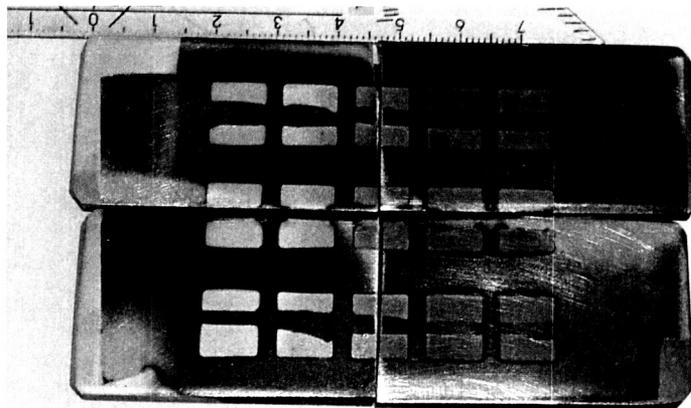


Figure 7. Photograph of combinatorial libraries formed on Inconel 718 substrates by co-sputtering aluminum and NiCoCrAlY onto Inconel 718 substrates.

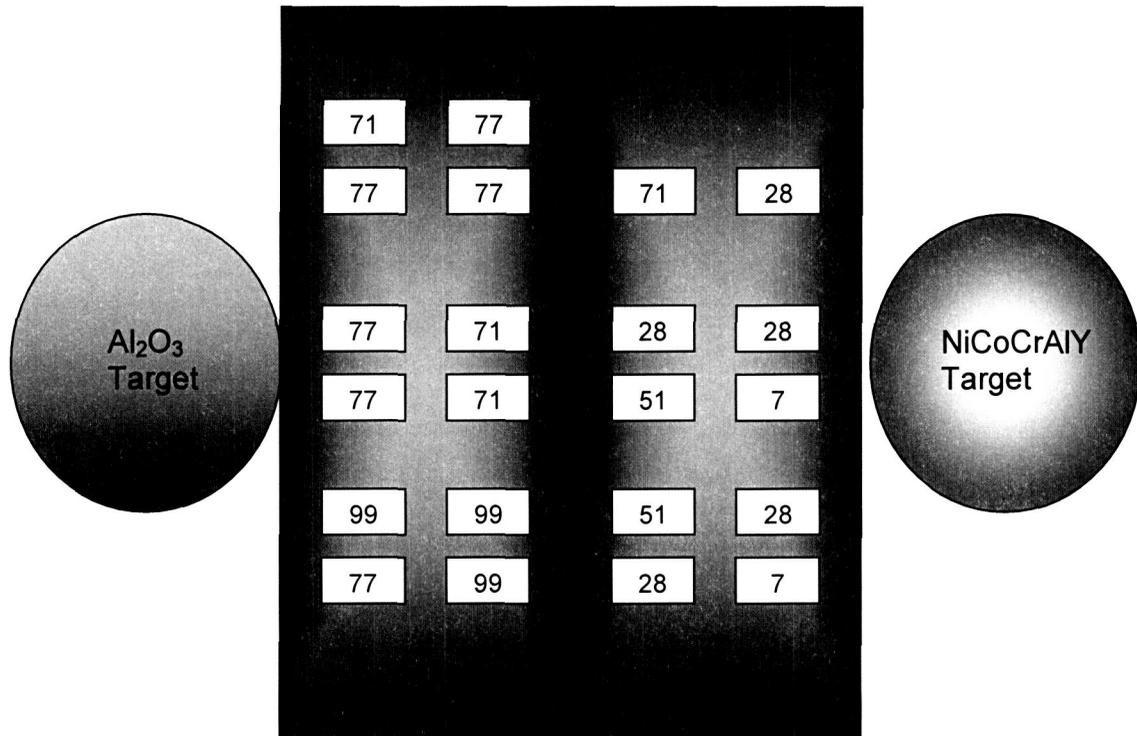


Figure 8. Results of combinatorial chemistry experiments to optimize intermediate coating chemistry that would match the TCE of the metallic and ceramic layers at the wavy interface formed by the thermal sprayed coatings. The combinatorial libraries were formed by co-sputtering aluminum oxide and NiCoCrAlY onto Inconel 718 substrates that were thermally sprayed with Praxair N171 bond coats. The lifetimes of each combinatorial library (in hours) are included in the schematic. These times (boxed numbers) correspond to the onset of microcracking and delamination within each combinatorial library.

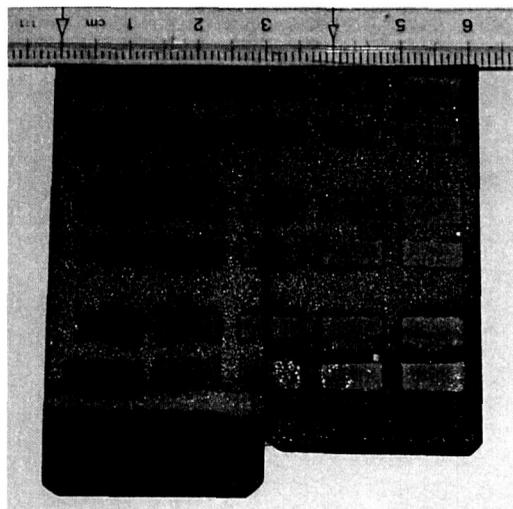


Figure 9. Photograph of combinatorial libraries formed on the surface of Praxair N171 bond coats by co-sputtering aluminum oxide and NiCoCrAlY.

Characterization of thermal spray ceramic coatings

All failed test coupons were cross sectioned using standard metallographic procedures and examined by scanning electron microscopy (SEM) using our state of the art JEOL 5900 Environmental Scanning Electron Microscope, equipped with light element EDS and 3 backscattered electron detectors. Cross sections of the failed coatings were examined in backscattered mode, since this provides the best contrast from an imaging viewpoint; i.e. this mode provides the greatest sensitivity to atomic number differences of the various constituents comprising the thermal sprayed coatings. The interface regions of failed Inconel 718 test coupons thermally sprayed with Praxair N343 indicated adhesive failure within the bond coat whereas those sprayed with Praxair N171 failed by decohesion/ delamination at top coat/bond coat interface. Representative examples of the two different types of failure in the thermally sprayed bond coats are shown in the SEM micrographs of Figures 10(a) and (b), respectively. The white phase present in each cross section is the platinum diffusion barrier applied to each bond coat. In general, those test coupons thermally sprayed with thicker bond coats and top coats resulted in a greater number of cycles to failure at 1100 °C.

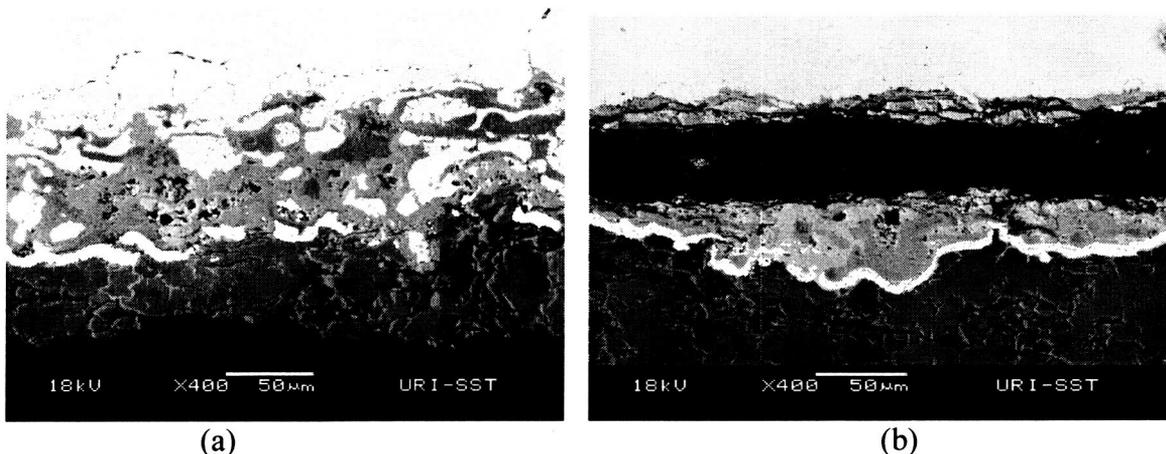


Figure 10. (a) SEM micrograph of a failed Praxair N171 thermal sprayed bond coat showing failure by decohesion / delamination at top coat/bond coat interface and (b) SEM micrograph of a failed Praxair N343 thermally sprayed bond coat showing adhesive failure within the bond coat.

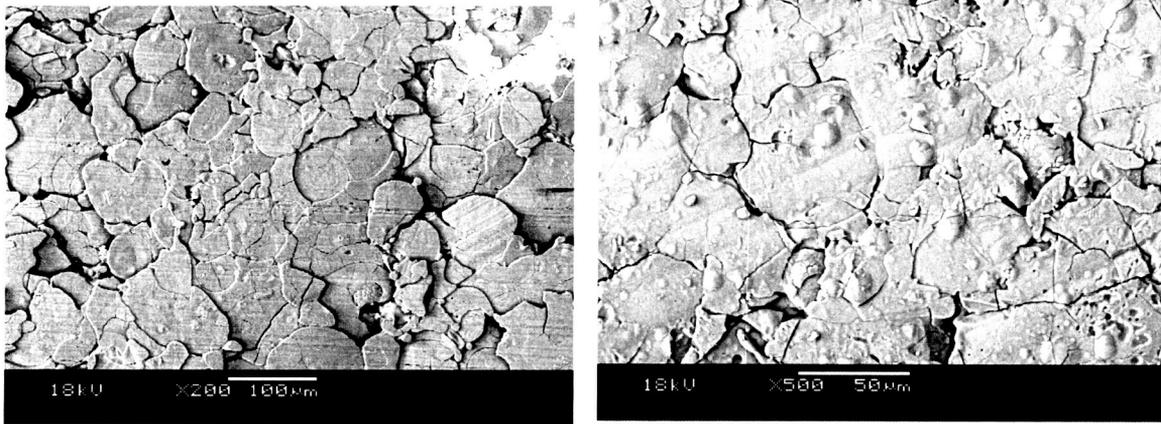
The failure mechanisms observed for the thermal sprayed coatings fatigue tested in our computer-controlled burner rig were very different than those observed for the thermal sprayed coatings tested in our furnace rig. The Inconel 718 coupons with Praxair N171 bond coats that were overcoated with high purity alumina, failed by adhesive failure and delamination at the top coat/bond coat interface similar to the magnesium aluminate spinel overcoated Inconel 718 coupons that were tested in our furnace rig. However, Inconel 718 coupons with Praxair N171 bond coats that were overcoated with magnesium aluminate spinel and fatigue tested in our computer-controlled burner rig failed by cohesive failure due to disintegration (localized spalling and/or flaking of the ceramic top coat). It was also observed that the surfaces of the magnesium aluminate spinel top coats

became very rough after thermal cycling and the surface morphology changed considerably, similar to the changes observed in thermal sprayed zirconia TBC's. In these systems, sintering effects and phase changes in the thermal sprayed zirconia TBC coatings have been linked to failure.

SEM micrographs of as-sprayed magnesium aluminate spinel and as-sprayed high purity alumina are shown in Figures 11(a) and (b). The surface morphology of the two top coats shown in Figure 11 appears to be somewhat similar. However, after thermal cycling to 1400 °C, fine structure that is barely visible in the microstructure of the magnesium aluminate spinel shown in Figure 11(a), becomes much more pronounced as shown in the SEM micrograph in Figure 12. Here there is evidence that crystallization and/or a phase change has occurred in the bulk ceramic upon heating to these temperatures. This surface morphology is very similar to that of amorphous silicon dioxide that has been thermally cycled to 1200 °C. When Si_3N_4 ceramics are oxidized at 1200 °C, an amorphous SiO_2 is formed on the surface. However, after repeated thermal cycling, devitrification of the silica occurs and small crystallites of cristobalite (crystalline SiO_2) grow within the amorphous SiO_2 matrix, similar to the surface morphology shown in Figure 12.

These SEM micrographs may hold several clues as to why Inconel 718 coupons prepared with Praxair N171 bond coats and overcoated with high purity alumina survived a significantly greater number of cycles to failure when tested in our computer-controlled burner rig than did the N171 bond coats overcoated with magnesium aluminate spinel (24 + cycles to failure vs. 4 cycles to failure). A differential scanning calorimetry (DSC) spectra of the two thermally sprayed top coats (magnesium aluminate spinel and high purity alumina) cycled from room temperature to 1400 °C is shown in Figures 13 and 14. These DSC spectra confirm the SEM findings in that a large peak is present in the DSC spectra of the magnesium aluminate spinel at 850 °C (Figure 13) but no such peaks are present in the DSC spectra of the high purity alumina (Figure 14).

The fatigue lifetimes of the Praxair N171 (NiCoCrAlY) bond coats that were thermally oxidized in reduced oxygen partial pressure, were not improved relative to the as-sprayed Praxair N171 bond coats, when tested in our computer-controlled burner rig. Platinum diffusion barriers (2 μm thick) applied to Praxair N171 (NiCoCrAlY) bond coats resulted in considerable improvement in fatigue life relative to the as-sprayed Praxair N171 bond coats, essentially doubling the lifetime (8 cycles to failure vs. 4 cycles to failure) as shown in Table 2. When relatively thick Al_2O_3 pre-coats (5 μm thick) have been applied to Praxair N171 (NiCoCrAlY) bond coats as diffusion barrier coatings, the fatigue life of the thermal sprayed coatings was substantially improved relative to the as-sprayed Praxair N171 (NiCoCrAlY) bond coats (10 cycles to failure vs. 4 cycles to failure) when tested in our computer-controlled burner rig, as shown in Table 2.



(a)

(b)

Figure 11. (a) SEM micrograph of as-sprayed magnesium aluminate spinel coating and (b) SEM micrograph of as-sprayed high purity alumina coating.

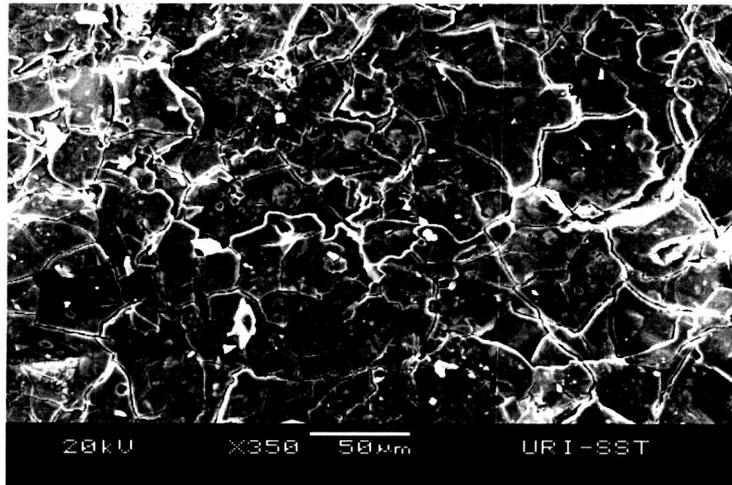


Figure 12. SEM micrograph of an as-sprayed magnesium aluminate spinel coating. There is evidence that crystallization and/or a phase change has occurred in the bulk ceramic upon thermal cycling to 1400 °C.

DSC Results For Flame-sprayed Magnesium Aluminate

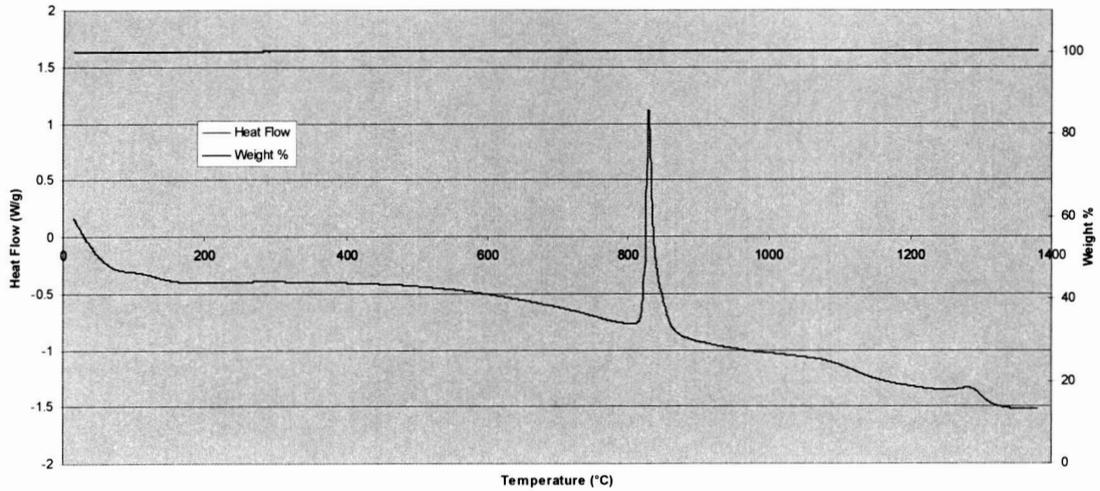


Figure 13. DSC scan of an as-sprayed magnesium aluminate spinel coating. Note the peak at 850 °C, suggesting that crystallization and/or a phase change has occurred in the bulk ceramic upon thermal cycling to 1400 °C.

DSC Data of SDT Norton 1702-12 Powder

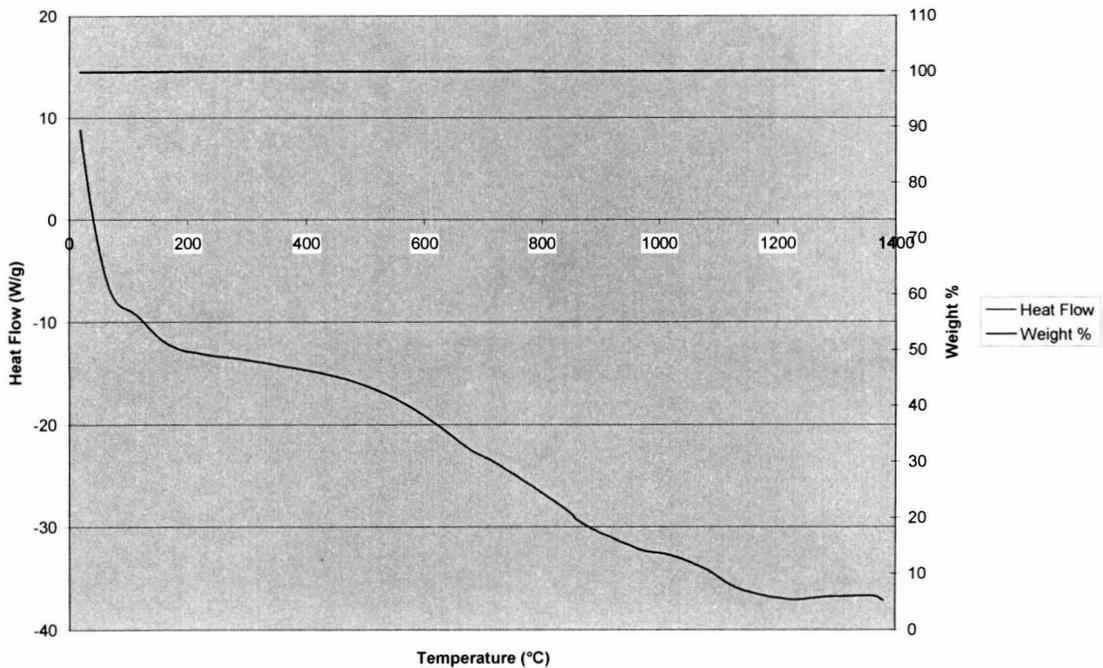


Figure 14. DSC scan of an as-sprayed high purity alumina coating. Note that there are no sharp peaks present in the spectra, suggesting that no crystallization/phase changes should occur in the bulk ceramic when thermally cycled to 1400 °C.

Conclusions

The focus of this APP research program was to improve the bond coats used in the fabrication of flame sprayed instrumentation. Typically, a bond coat is applied to a superalloy surface prior to the application of a thin dielectric coating onto which instrumentation is placed. After affixing the instrumentation, a much thicker ceramic topcoat is typically applied to protect the instrumentation from harsh environments. The fatigue life of NiCoCrAlY coated superalloys was extended beyond current state-of-the-art by relatively simple and cost effective means. Heat treatment in reduced oxygen partial pressures at 1750 to 1800 °F effectively doubled the fatigue life of NiCoCrAlY coated substrates relative to as-sprayed substrates and when used in conjunction with platinum diffusion barriers yielded a four fold increase in the fatigue life of NiCoCrAlY coated substrates. Further improvements in the fatigue life of thermally sprayed coatings were made by employing intermediate coatings, which minimized thermal expansion differences between the bond coat and top coat. Combinatorial chemistry experiments yielded an optimum composition for an intermediate TCE matching coating that showed considerable promise in extending the fatigue life of thermal spray instrumentation. The intermediate coating had two functions: to reduce the surface roughness of the peaks and valleys associated with the as-sprayed NiCoCrAlY bond coat, and to produce a thin layer of a mixture of Al₂O₃ and NiCoCrAlY that exhibited an intermediate TCE. The optimal composition of the intermediate coating consisted of 60 wt% Al₂O₃ and 40 wt% NiCoCrAlY, as determined by energy dispersive analysis of x-rays (EDS). Intermediate coatings having this composition were prepared by physical vapor deposition and the resulting coating systems are being evaluated in our test facility.

The Ohio Aerospace Institute's PWIG team members from Pratt and Whitney and Honeywell engines have supplied all of the necessary superalloy substrates to date. These included both Inconel 718 coupons and Inconel 718 parts for the test rig as well as single crystal superalloys (Pratt and Whitney). NASA and The Ohio Aerospace Institute's Propulsion Instrumentation Working Group will verify the modifications to the existing flame spray technology by applying them to propulsion hardware such that structural models can be validated and the lifetime of components predicted.

Presentations—NASA APP program: “Flame Spray Strain Gages with Improved Durability and Lifetimes

Ohio Aerospace Institute -Propulsion Instrumentation Working Group; “Flame Spray Strain Gages with Improved Lifetimes and Durability”, O.J. Gregory, Markus Downey, Steve Wnuk and Vince Wnuk, San Diego, CA, May 2002

Ohio Aerospace Institute -Propulsion Instrumentation Working Group; “Flame Spray Strain Gages with Improved Lifetimes and Durability”, O.J. Gregory, Markus Downey, Tim Starr, Steve Wnuk and Vince Wnuk, San Diego, CA, Morgantown, WV, Oct. 2002

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