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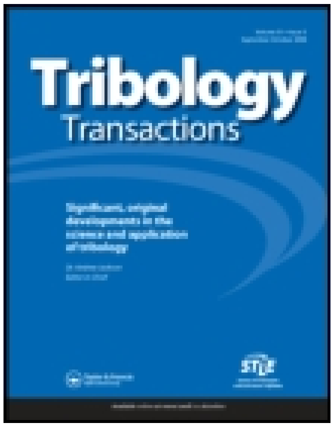

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Thermal Processing Effects on the Adhesive Strength of PS304 High Temperature Solid Lubricant Coatings

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Thermal Processing Effects on the Adhesive Strength of PS304 High Temperature Solid Lubricant Coatings©

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The effects of post deposition heat treatments on the strength properties of PS304, a plasma sprayed nickel-chrome based, high temperature solid lubricant coating are studied. Coating samples were exposed in air at temperatures from 432 to 650°C for up to 500 hr to promote residual stress relief, enhance particle to particle bonding and increase coating to substrate bond strength. Coating pull off strength was measured using a commercial adhesion tester.

The as-deposited (untreated) samples either delaminated at the coating-substrate interface or failed internally (cohesive failure) at about 17 MPa. Samples heat treated at temperatures above 540°C for 100 hr or at 600°C or above for more than 24 hr exhibited strengths above 31 MPa. Coating failure occurred inside the body of the coating (cohesive failure) for nearly all of the heat-treated samples and only occasionally at the coating substrate interface (adhesive failure). A microstructural change is believed to be partially responsible for the coating strength increase. Increasing the heat treatment temperature, exposure time or both accelerate the heat treatment process.

KEY WORDS

Solid Lubrication; Self-Lubricating Composites; High Temperature

INTRODUCTION

PS304 is a plasma sprayed, high temperature solid lubricant coating developed to reduce friction and wear of sliding contacts operating from below room temperature to over 750°C (DellaCorte, et al. (1995), (1996), (1997)). PS304 is comprised of a nickel-chromium (NiCr) matrix (60 wt.%) combined with chrome oxide (20 wt.%) as a hardening phase and the solid lubricant additives silver (10 wt.%) and barium fluoride/calcium fluoride eutectic (10 wt.%). For the PS304 composite coating, each constituent contributes functionality. For example, silver, a soft metal, provides low temperature lubrication while the fluorides provide high temperature lubrication. The chrome oxide acts as a thermochemically stable hardening phase. The NiCr matrix bonds the components to each other and the substrate. Since NiCr bonds well to candidate stainless steel and superalloy substrates the PS304 coating is routinely applied directly to the surface of parts needing lubrication without the use of intermediate bond coat layers. Figure 1 shows a typical cross-section photomicrograph of PS304.

Extensive development work has been conducted and reported on this composite coating. This work includes compositional tailoring for thermal expansion coefficient control (DellaCorte, et al. (1997)), friction and wear testing (DellaCorte, et al. (1995), (1996)) and coating performance as a start/stop shaft lubricant for foil air bearings (DellaCorte, et al. (2000)). During the course of these studies it was observed that the coating undergoes a one-time dimensional change (coating thickness increase of a few percent) accompanied by an apparent strength (hardness) increase when exposed to moderate temperatures (~500°C) for long periods (~100 hr). Routine metallographic cross sections have shown evidence of a complex interfacial reaction layer between the coating and superalloy substrates as well as an apparent microstructural coarsening of the coating's matrix (NiCr) phase. Figure 2 shows such changes for a PS304 coated nickel based superalloy specimen exposed to 650°C in air for over 700 hr. These microstructural and interfacial changes do not appear to negatively affect coating performance but warrant further study.

Since coating adhesion can play a critical role in coating performance it has been selected for further investigation in the cur-

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TABLE 1—PS304 COMPOSITION

CONSTITUENT	wt.%	FUNCTION
NiCr[a]	60	binder
Cr_2O_3	20	hardener
Ag	10	low temperature lubricant
BaF_2/CaF_2[b]	10	high temperature lubricant

[a] Ni/Cr ratio is 80/20 by wt.%.

[b] BaF_2/CaF_2 ratio is 62/38 by wt.%.

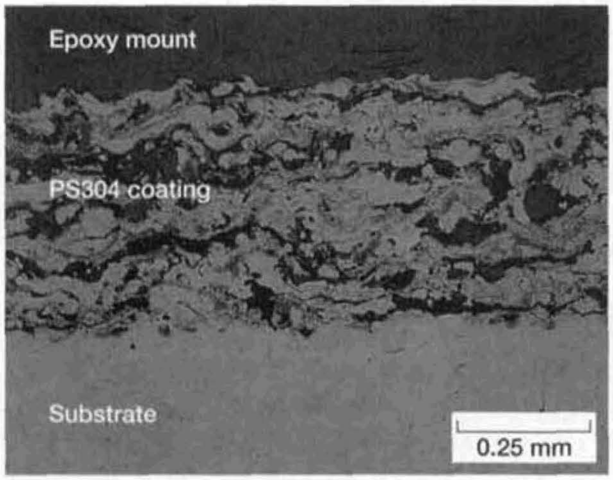

Fig. 1—Cross-section photomicrograph of an as-deposited PS304 coating on a 13-8 Mo stainless steel substrate.

rent work. Others have observed similar strengthening effects in NiCr based coatings following heat treatments but offer no explanation (Walther, (1979)). It is believed that study of coating adhesion properties may provide insight into the phenomena described above and help establish processing parameters for more widespread coating implementation. The following sections describe the results of adhesion tests on PS304 coatings deposited on stainless steel prior to and after heat treatments in air at varying temperatures and durations. Stainless steel is the selected substrate because it is a likely coating substrate and is the shaft material being used in an oil-free turbocharger using PS304 (Howard, (1999), Heshmat, et al., (2000)).

The ultimate goals of this work are to develop a better understanding of the role of the heat treatment and to better identify and understand the mechanism(s) in action during thermal exposure. It is anticipated that the results will also be used to develop a preliminary heat treatment process for the PS304 coating to foster commercialization of this coating technology.

TEST PROCEDURES AND APPARATUS

PS304 Coating and Deposition

The PS304 coating is plasma spray deposited using conventional plasma spray procedures described fully in (DellaCorte, et al. (1987)). Briefly, a powder blend of the constituents in PS304 (shown in Table 1) is fed by a hopper into an argon gas stream and injected into an argon plasma in a plasma spray gun. The high temperature in the plasma gun melts the powder and accelerates it out of the gun nozzle. The molten powder then impinges onto the substrate where it "splats" and solidifies forming a thin, discontinuous coating. Multiple passes are applied to build up a thick (~350 μm) coating layer. Following deposition the coating is ground to achieve a smooth surface finish and the desired thickness. The deposition and solidification process occurs rapidly and often creates residual stresses in the coating. Figure 1 shows a representative cross-section photomicrograph of the PS304 coating deposited onto a stainless steel substrate. Table 2 shows selected properties and the composition of the stainless steel used in this work. PS304 coatings have a layered structure where each layer is the result of each coating pass. For PS304, ~20 μm of coating is deposited per pass and 10 to 20 passes are applied to build up a 250 to 350 μm thick coating shown. As seen in the photomicrograph, the PS304 coating is a heterogeneous composite in which each constituent appears as a distinct phase.

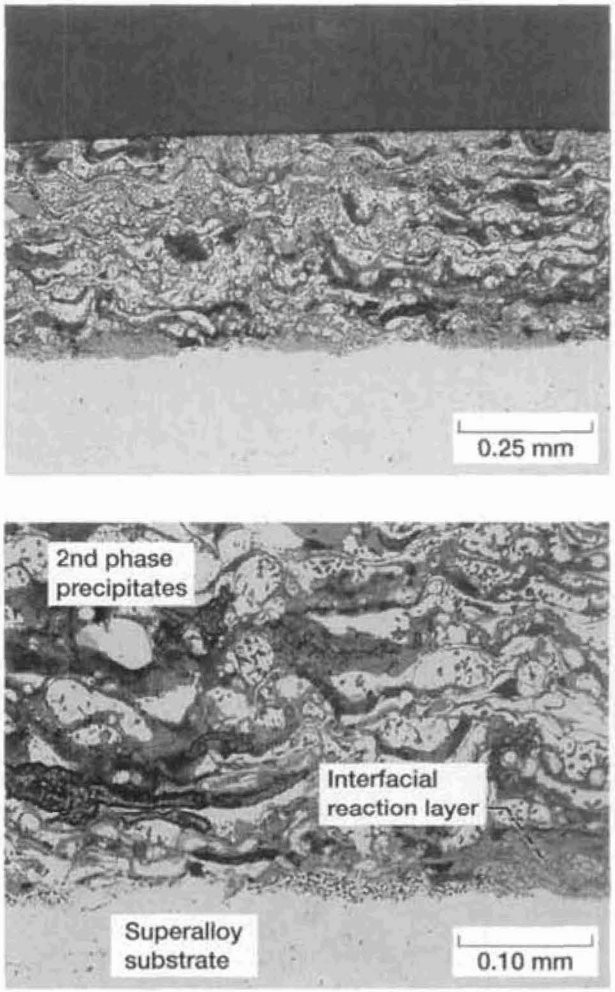

Fig. 2—Cross-section photomicrographs of PS304 coating deposited on a nickel based superalloy substrate after exposure in air at 650°C for 700 hr. Note interfacial reaction layer and second phase precipitates in NiCr matrix.

PULL/ADHESION TEST

To evaluate coating strength, a commercial adhesion tester is used. This test hardware, shown schematically in Fig. 3, consists

TABLE 2—13-8 MO STAINLESS STEEL COMPOSITION

PROPERTY	VALUE
Composition, wt.%	Fe-76, Cr-12.5, Ni-8.0, Mo-2.5, Al-1.0
Density, g/cc	7.8
Ultimate tensile strength, MPa	1480
Yield strength, MPa	1415
Elastic modulus, GPa	221
Thermal expansion coefficient/°C	11.0 x 10^{-6}
Thermal conductivity, W/m-k	14
Heat capacity, J/g-°C	0.46

TABLE 3—ADHESION DATA SUMMARY FOR PS304 DEPOSITED ON 13.8 MO STAINLESS STEEL

HEAT TREATMENT		ADHESION STRENGTH	
TEMPERATURE °C	TIME, hr	MPA	ksi
None as-deposited coating		(17.2±1.2)	2.51±0.43
482	75	(17.7±0.3)	2.64±0.16
482	150	(22.8±1.1)	3.39±0.16
482	250	(20.2±1.3)	3.00±0.20
482	500	(22.8±2.0)	3.40±0.30
538	100	(20.2±3.4)	3.00±0.50
538	150	(32.5±3.3)	4.83±0.49
538	200	(30.2±3.4)	4.50±0.50
538	500	(36.6±1.4)	5.45±0.20
593	25	(28.8±8.1)	4.29±0.12
593	50	(35.9±0.7)	5.35±0.10
593	100	(37.4±1.0)	5.56±0.15
593	150	(34.4±1.4)	5.12±0.20
593	200	(37.5±1.2)	5.58±0.15
650	25	(33.3±0.7)	4.95±0.10
650	50	(31.2±0.7)	4.65±0.10
650	100	(33.9±1.5)	5.05±0.20
650	150	(32.4±1.8)	4.82±0.22
650	200	(36.5±1.2)	5.43±0.17

of an aluminum pull stud which is glued to the coating surface, a pneumatic piston system for applying the pull of force and an electronic pressure controller/readout for measuring and recording the pull off pressure. The head of the pull stud is attached to the coating surface using a two part epoxy glue which is mixed, applied, air cured for about 8 hr then oven cured at 75°C for an additional 24 hr prior to pull testing. The epoxy has a rated strength of about 60 MPa under ideal conditions and typically exceeds 12 MPa when attached to the rough plasma spray coating. For reference, plasma sprayed coatings like PS304 that exhibit adhesion strength greater than 4 MPa are usually considered acceptable for most tribological applications.

To conduct a test, a coated and glued specimen assembly is placed into the fixture shown previously in Fig. 3. The controller is started which gradually pressurizes the loading piston thereby increasing the applied pull force until it exceeds the strength of the coating or the epoxy bond. When the pull off force exceeds the strength the stub detaches from the sample and the test ends. A digital pressure readout on the controller displays the peak pressure applied during the test. It is recorded and converted to coating strength in MPa using a manufacturer's generated data sheet. Since nonuniform epoxy application, misaligned studs and surface irregularities present in the as-deposited PS304 samples can result in data scatter at least three repeat tests are done for each test condition.

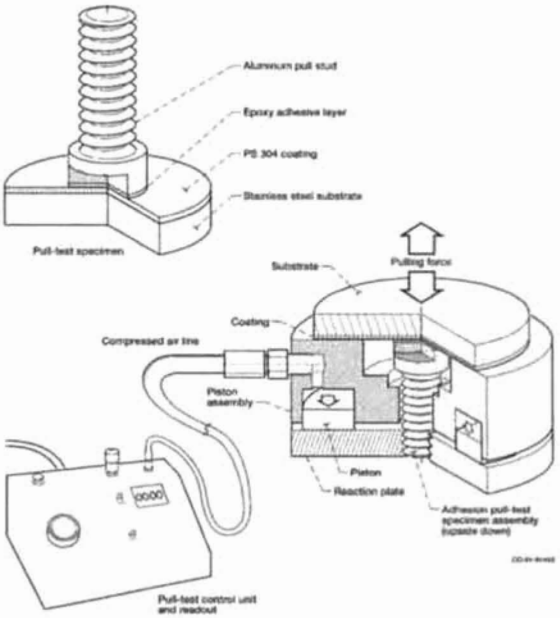

Fig. 3—Adhesion pull-test specimen assembly and system.

The samples can fail in one of three ways. If the coating to substrate bond is weakest the stud will pull off the sample removing the full coating and exposing the stainless steel substrate. If the bond is strong but the coating cohesive strength is low the stud will pull off the sample and remove some of the coating with failure occurring within the coating. Finally, if both the coating and its bond to the substrate are strong the stub will pull off the sample with failure occurring within the epoxy layer. By observing where failure occurs, coating properties (e.g., bond strength, apparent tensile strength etc.) and the effect of differing heat treatments can be assessed.

COATING MICROSTRUCTURAL ANALYSES

Selected coating samples were mounted in metallographic potting compound (epoxy) and prepared by conventional metallographic methods for analyses. Viewing the coating cross sections before and after thermal processing can lend insight into microstructural changes and their effects on strength. Both optical and Scanning Electron Microscopic (SEM) techniques were employed in this work. For SEM analyses, the samples were coated with a thin layer of gold to prevent charge build-up that can obscure the images.

RESULTS AND DISCUSSIONS

The PS304 exhibits a pull off strength of 17 MPa in the as-deposited condition with no heat treatment. Failure occurs within the coating in a cohesive manner suggesting that the bond between the coating and the substrate is stronger than the coating itself. Coatings exposed to high temperatures for brief periods or moderate temperatures for long periods exhibit strength around 35 MPa. Table 3 shows the averaged data for all of the tests conduct-

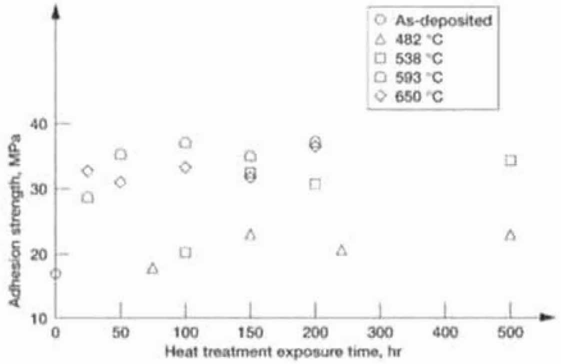

Fig. 4—Adhesion strength *vs.* exposure time at various temperatures for PS304 deposited on 13-8 Mo stainless.

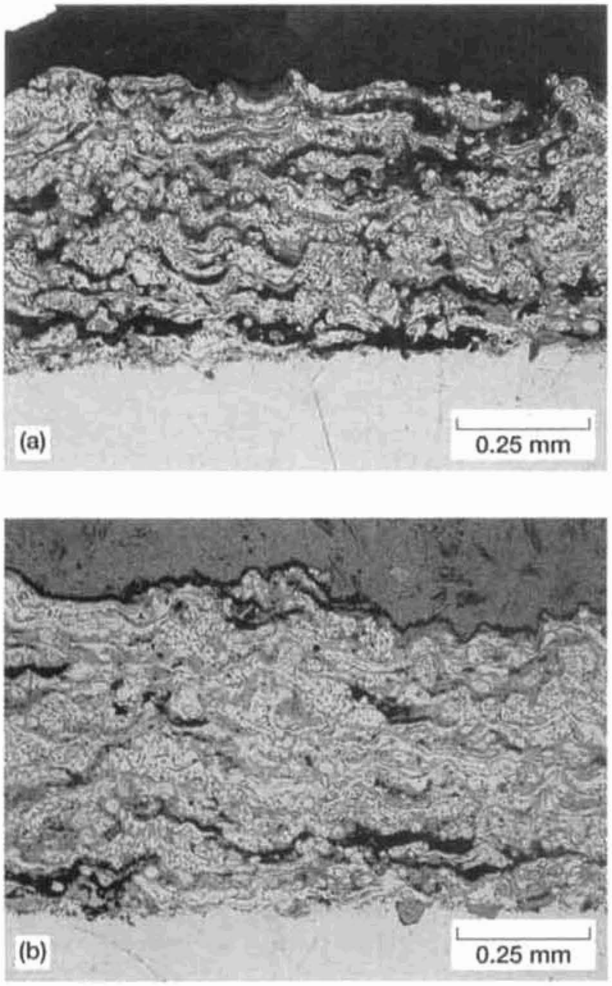

Fig. 5—Cross-section photomicrographs of PS304 exposed in air for 150 hours.
(a) 593°C
(b) 650°C

ed ranging in duration from 24 to 500 hrs at temperatures from 432 to 650°C. This data is plotted in Fig. 4.

The data show that at lower temperatures longer exposure times are needed to achieve the strengthening effect. This type of behavior is common for diffusion based metallurgical changes (Shewmon, (1963), Boyer, et al. (1985)). Abrupt phase transformations, such as the well known alpha to beta transformation that occurs for zirconium oxide, rapidly occur once a specific temperature threshold is exceeded (Kingery, et al. (1975)). For the PS304 coating, the adhesion data suggests that more gradual changes occur which are enhanced by high temperatures and time. For instance, after exposure for 500 hr at 482°C the adhesion strength has only reached ~2/3 of full strength (~35 MPa). Yet at 650°C, a mere 24 hr appears more than adequate to fully heat treat the PS304 coating. Several coating samples have been exposed for over 700 hr at 650°C and exhibit strengths around 35 MPa suggesting that the strengthening effects have a terminus (reach a final value).

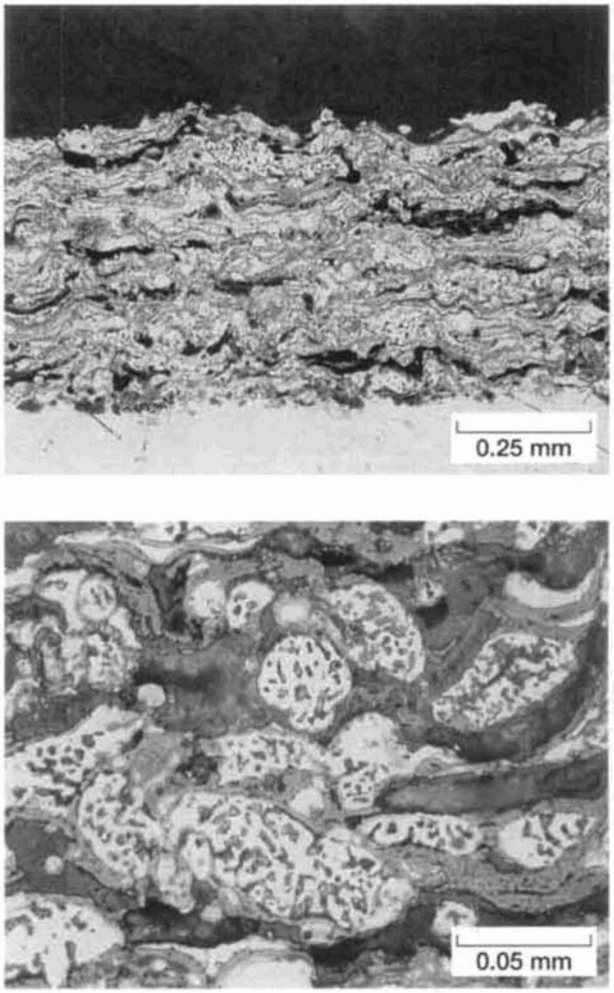

Fig. 6—Cross-section photomicrographs of PS304 after exposure in air at 650°C for 50 hr.

Many of the candidate substrates for PS304 are heat treated superalloys and stainless steels. Since a wide range of thermal exposure temperatures enhance coating strength it is likely that the coating thermal processing can be achieved coincidentally with a substrate heat treatment. In this case, the expense of an additional separate coating heat treatment could be avoided.

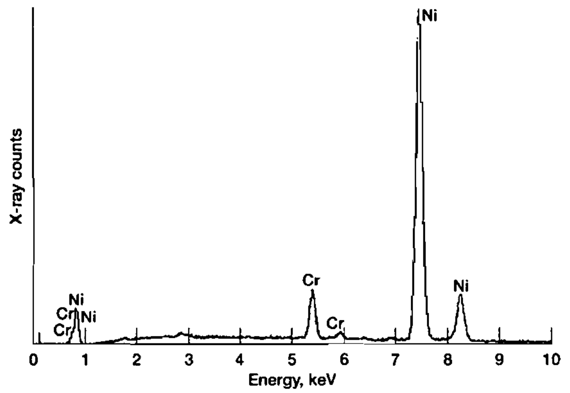

Fig. 7—EDS spectra of NiCr matrix phase of PS304 as deposited coating. Ni/Cr peak height ratio is ≈ 6.2.

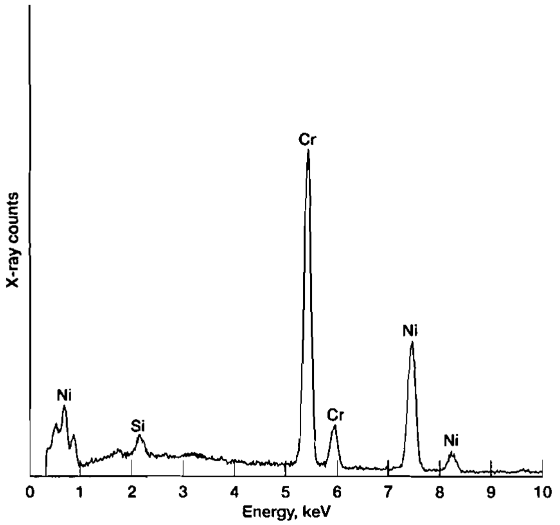

Fig. 8—EDS spectra of dark chromium rich precipitate inside NiCr matrix phase of PS304 exposed at 650°C for 150 hours. Ni/Cr peak height ratio is ≈ 0.5.

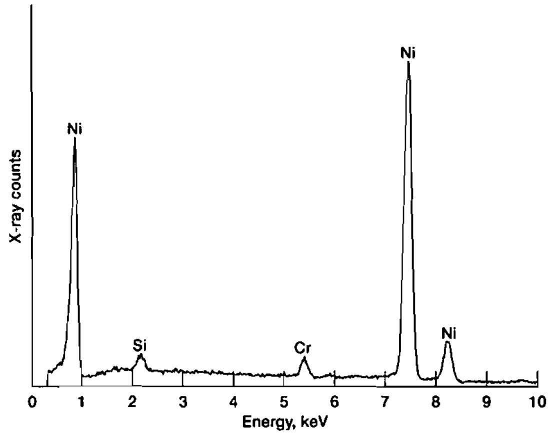

Fig. 9—EDS spectra of NiCr phase surrounding dark chromium rich precipitate from PS304 exposed at 650°C for 150 hours. Note apparent depletion of chromium content. Ni/Cr peak height ratio is ≈ 10.6.

The adhesion data alone suggests diffusion controlled changes are occurring to the PS304 during the thermal processing. The observation that the strengthening occurs much more rapidly above 538°C further indicates a solid state diffusion controlled effect. The main component in PS304 is NiCr which has a melting point of 1390°C (1663°K). A heat treatment temperature of 538°C (811°K) is nearly 0.5 that of NiCr's melting point. In solid state diffusion, atomic mobility rates increase dramatically when the ambient temperature exceeds about 40 percent of the absolute melting point (Shewmon, (1963)). Therefore, it is not surprising to observe what appears to be diffusion based property (adhesion strength) changes in the coating when exposed to temperatures above about 500°C.

Post heat treatment metallographic cross sections of the PS304 specimens help elucidate the reasons for the observed strength increase. Figures 5 and 6 show metallographic images, at various magnifications, of the PS304 coated stainless steel substrates at selected test temperatures and exposure times. The as deposited samples exhibit microstructural features typical for thermal spray coatings. The PS304 starts out as a powder blend of the individual constituents and each particle melts separately in the hot plasma and deforms upon impact with the substrate creating a layered coating structure made up of "splats" on top of one another. When examined under an optical microscope the different phases can be readily detected and identified. The NiCr binder is predominant and bright, the silver is metallic and develops a slight tarnish, the chrome oxide is gray and the fluorides, when viewed with oblique lighting, appear transparent or slightly opaque. Black, nonreflective regions are porosity, which is typically about 15 percent of the structure.

Since NiCr is PS304's major constituent its properties are likely to dominate the coating's properties, especially strength. Therefore, it is anticipated that changes to the NiCr matrix can have significant effects on the coating strength properties. The silver and fluorides are lubricant phases, therefore, any changes to these constituents, such as chemical reactions, degradation, or decomposition, are anticipated to have significant effects to the lubrication properties of the coating.

Examination of the coating cross sections reveals that the only readily observed change due to the thermal processing is an apparent phase segregation or, rather, second phase precipitation inside the NiCr binder regions. When PS304 deposited onto nickel based superalloys is heat treated a reaction layer forms at the interface. For PS304 deposited on stainless steel, no interfacial reaction layer is observed. The appearance of the silver, chrome oxide and fluoride phases remains unchanged even after exposure at 650°C for over 100 hr. The NiCr phases, however, develop a striking microstructural feature which has the appearance of dark islands often locked inside NiCr particles. These regions, at first, were assumed to be some oxide phase. However, NiCr would be expected to develop an oxide passivating layer on its surface when exposed to air at high temperature not internal oxide phases. On the contrary, there does not appear to be any film or layer of oxide on the surface of the NiCr phases. In addition, the appearance of the "two phase" NiCr matrix is uniform throughout the coating. That is, its appearance is the same at the coating's outer surface which is directly exposed to air during the heat treatment and at the substrate interface which is well protected from the atmosphere. These observations suggest that during the heat treatment some type of diffusion driven second phase precipitation has occurred.

Scanning Electron Microscopic (SEM) analyses were conducted, coupled with Energy Dispersive X-Ray analyses (EDS), to elucidate the composition and nature of the changes to the NiCr matrix. The results are suggestive but not conclusive. For comparative purposes, an EDS spot spectrum of a NiCr area in an as deposited coating is shown in Fig. 7. The unheat treated NiCr, which has an 80/20 wt.% composition, exhibits a nickel to chromium peak height ratio of 6.2. Figure 8 shows EDS spot spectra of the NiCr matrix and its second phase precipitates. The dark, chromium rich precipitate phases are high in chromium with smaller peaks of nickel and silicon. Oxygen and other species (fluorine, carbon, barium, calcium etc.) are not detected. The Ni/Cr ratio for the precipitate phase is about 0.5. EDS spectra of the lighter surrounding NiCr matrix show large nickel content with a moderate chromium peak. When compared to the EDS spectra of as deposited NiCr, the heat treated lighter phase appears somewhat chromium depleted. The Ni/Cr ratio in this case is about 10.2.

This comparison indicates that the chromium concentration in the precipitated phase is a diffusion migration from the surrounding NiCr phase. The presence of silicon in the spectra is interesting. Silicon is not an intentional coating constituent. It is present as a ubiquitous additive in the NiCr powder produced commercially. Silicon, in the form of a silicate, is added to the NiCr powder as a powder flow enhancer. It is present at the 1 percent or less level and is often found in commercial thermal spray powders. It is also possible that the silicon could be present as dispersed silicon dioxide or at NiCr grain boundaries. More detailed analyses would be required to conclusively determine this. The presence of a strong silicon peak in the precipitated phase suggests the formation of some type of chromium-silicon compound, perhaps a chrome silicide. Although not yet confirmed, coating thickness increases after heat treatment of up to about 3 percent have been observed and may be linked to the second phase precipitation.

The structure of both the dark precipitated phase and the surrounding NiCr matrix cannot be exactly determined using SEM/EDS techniques. Further analytical work using X-Ray diffraction and less complex (perhaps pure NiCr coatings) samples may be required to determine the true changes taking place during the heat treatments. It can be speculated, however, that during the heat treatment a chromium rich second phase precipitates inside the NiCr matrix leading to a strengthening effect. It is also possible that the heat treatment promotes residual stress relief and internal diffusional bonding (e.g. crack healing) leading to enhanced strength. In addition, since the microstructure appears to be stable following the formation of the second phase, it is expected that the adhesion and strength properties of the PS304 coating will remain stable over longer periods. In fact, samples exposed to high temperature air for over seven hundred hours display adhesion properties and microstructures virtually indistinguishable from samples exposed for 24 hr at 650°C.

CONCLUDING REMARKS

The PS304 composite plasma sprayed coating exhibits an increase in adhesion strength of ~100 percent following exposure to temperatures above 500°C for various lengths of time. The data shows that as the exposure temperature increased the time required to achieve strengthening dramatically decreased suggesting some type of diffusion controlled metallurgical change is occurring in the coating. Specimens exposed for long time periods at high temperatures do not experience any further increase in strength properties suggesting some type of metallurgical change occurs which reaches an equilibrium condition. Post test metallographic cross-sections coupled with SEM and EDS analyses indicate that a second phase, rich in chromium, precipitates in the NiCr matrix. The second phase, which appear as dark islands surrounded by a NiCr matrix, are often observed to contain silicon most likely coming from a silicate flow enhancer found in the NiCr powder feedstock. EDS elemental analysis shows that the Ni to Cr ratio in heat treated samples is lower than as deposited samples suggesting that the chromium in the second phase is coming from the NiCr matrix and not the chrome oxide constituent of PS304.

Preliminary measurements also indicate that a volume expansion of a few percent occurs following heat treatment. Similar to the adhesion strength, after the heat treatment is complete no further volume change occurs even after long exposure periods. These results indicate the need for a heat treatment of the PS304 coating after plasma spraying and prior to finish machining to insure dimensional stability and maximum adhesion properties. Further research is warranted to elucidate the exact nature of the coating changes that occur and to understand the possible effects, if any, of the substrate and environments on the performance and environmental durability of PS304.

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