



Thermal Effects on a Low Cr Modification of PS304 Solid Lubricant Coating

Malcolm K. Stanford
Glenn Research Center, Cleveland, Ohio

Anne M. Yanke
Cleveland State University, Cleveland, Ohio

Christopher DellaCorte
Glenn Research Center, Cleveland, Ohio

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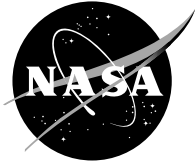
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NASA/TM—2004-213111



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Anne M. Yanke
Cleveland State University, Cleveland, Ohio

Christopher DellaCorte
Glenn Research Center, Cleveland, Ohio

National Aeronautics and
Space Administration

Glenn Research Center

June 2004

Acknowledgments

The authors would like to thank J.A. Buehler, D.J. Dixon, B.J. Edmonds, V. Lukaszewicz, T.R. McCue, M.S. McQuater, J.L. Smialek, and A.R. Zaldana for their technical assistance and helpful discussions during the course of this investigation.

This report is a formal draft or working paper, intended to solicit comments and ideas from a technical peer group.

This report contains preliminary findings, subject to revision as analysis proceeds.

This work was sponsored by the Low Emissions Alternative Power Project of the Vehicle Systems Program at the NASA Glenn Research Center.

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Malcolm K. Stanford
National Aeronautics and Space Administration
Glenn Research Center
Cleveland, Ohio 44135

Anne M. Yanke
Cleveland State University
Cleveland, Ohio 44115

Christopher DellaCorte
National Aeronautics and Space Administration
Glenn Research Center
Cleveland, Ohio 44135

Summary

PS304 is a high temperature composite solid lubricant coating composed of Ni-Cr, Cr₂O₃, BaF₂-CaF₂ and Ag. The effect of reducing chromium content on the formation of voids in the Ni-Cr particles after heat treatment in PS304 coating was investigated. Coatings were prepared with Ni-20Cr or Ni-10Cr powder and in various combinations with the other constituents of PS304 (i.e., chromia, silver and eutectic BaF₂-CaF₂ powders) and deposited on metal substrates by plasma spray. Specimens were exposed to 650 °C for 24 hr or 1090 °C for 15 hr and then examined for changes in thickness, coating microstructure and adhesion strength. Specimens with Ni-10Cr generally had less thickness increase than specimens with Ni-20Cr, but there was great variance in the data. Reduction of chromium concentration in Ni-Cr powder tended to reduce the appearance of voids in the Ni-Cr phase after heat exposure. The presence of BaF₂-CaF₂ resulted in a significant increase in coating adhesion strength after heat treatment, while coatings without BaF₂-CaF₂ had no significant change. Chemical composition analysis suggested that the void formation was due to oxidation of chromium in the Ni-Cr constituent.

Introduction

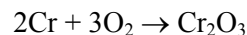
PS304 is a composite solid lubricant coating used in high speed, high temperature Oil-Free Turbomachinery developed at the NASA Glenn Research Center [1,2]. The coating feedstock is a powder blend containing 60 wt% Nichrome (Ni-20Cr), 20 wt% Cr₂O₃, 10 wt% eutectic BaF₂-CaF₂, and 10 wt% Ag. Nichrome acts as a binder, while chromia hardens the material. Silver and the eutectic fluoride are low temperature and high temperature solid lubricants, respectively. The composite powder is thoroughly mixed and fed into a plasma spray gun for deposition. During deposition, the material is heated to a molten or semi-molten state and propelled onto the surface to be coated. The coating is deposited to a thickness slightly greater than required and then ground to the desired dimension and surface finish.

After deposition, the coating undergoes a heat treatment at 650 °C for 100 hr to increase its cohesion strength from approximately 20 MPa (as-deposited) to approximately 30 MPa [3]. During the heat treatment, a change in the coating microstructure occurs. Figure 1 shows the appearance of the coating microstructure before and after exposure to 650 °C for 46 hr. The heat treated microstructure shows the formation of black Cr₂O₃ precipitates within the grey Nichrome phase shown in figure 1(b). The

precipitate formation was reported previously [3,4] as was an observed depletion of chromium from the nickel-chromium material in areas adjacent to these features. It is possible that voids may form due to the Kirkendall effect [5], where there is high diffusion of chromium out of the matrix. Such voids might also look like black precipitates. It has been very difficult to definitively characterize these features so far. The observed Cr₂O₃ particles could be smaller feedstock material that was encapsulated in the Ni-Cr during deposition or that is directly behind a Ni-Cr particle and observable due to the metallographic removal of material. Alternatively, voids could very well be cavities left from pull-out of Cr₂O₃ particles during the metallographic procedure. Clearly, more research is needed to understand the nature of these features.

It is thought that the fluorides may encourage the observed precipitate or void formation because they are highly reactive with some metals and ceramics at elevated temperatures. This behavior is well-known despite the general thermal stability of BaF₂-CaF₂ up to its melting point of approximately 1100 °C [6,7]. Therefore, it is suspected that the fluorides may catalyze a reaction with the Nichrome. The microstructural change also causes dimensional growth of the coating, which is unacceptable for some applications. Therefore, there is a need to understand the observed high temperature reaction.

Chemical analysis of heat treated coating microstructures indicated a depletion of chromium from the Ni-20Cr constituent, resulting in chromium content of about 10 wt% [4]. Based on this information, one possible reaction could be oxidation of Nichrome by the following process



reflecting a general depletion of chromium from the nickel-chromium solid solution and formation of a chromium oxide (CrO, CrO₃ or Cr₂O₃). Therefore, a nickel-chromium powder with only 10 wt% chromium (Ni-10Cr) was acquired for this investigation to test the proposed reaction. Conventional wisdom mandates a minimum concentration of 20 wt% chromium in high temperature alloys in order to maintain an oxidation resistant Cr₂O₃ layer. This study will determine if the diffusion of Cr into the Ni-Cr matrix can be limited by reducing the available chromium. The powder blend composed of Ni-10Cr, chromia, silver and fluorides was designated *LCR304* (a low-chromium version of PS304).

Metal oxidation is the combination of two spatially separated electrochemical processes, namely, reduction of gaseous oxygen at the interface of the gas and the ever-present metal oxide layer and oxidation of the metal at the metal-oxide interface [8,9]. The combination of these processes generates the metal oxide. Eventually, the transfer of metal ions out of the metal matrix and the influx of oxygen molecules can form voids [10]. The purpose of this investigation is to understand the role reducing chromium content in the nickel-chromium reactant may have in limiting or preventing the oxidation reaction that leads to void formation and coating growth. This will be assessed by measuring the effect of chromium content reduction on coating microstructure and chemistry, on coating thickness change with heat exposure and on coating cohesion strength.

Materials and Procedures

Figure 2 shows the fluoride, chromia and silver coating feedstock constituents used in this investigation. The fluoride particles are 45 to 106 μm in size, the chromia particles are 44 to 74 μm and the silver particles are 38 to 250 μm in diameter. The Ni-20Cr and Ni-10Cr shown in figure 3 are 44 to 74 and 53 to 150 μm in size, respectively.

Ni-20Cr is a commonly used material for industrial applications such as plasma spray bond coats, heating element wires and thermocouples. Ni-10Cr is not widely used and was procured specifically for this investigation. Both Ni-Cr powders were fabricated by atomization [11]. The powders used in this study are available commercially, except the fluorides, which were fabricated at NASA by a process described elsewhere [12].

Table 1 lists the coating materials and heat treatments used in this investigation. Three 25.4 mm diameter flat Inconel 718 or 13-8 Stainless Steel substrates were prepared and coated on one side by plasma spray with each desired coating (table 1). Plasma spray deposition was performed with commercially available equipment using the powder feedstock described earlier. The feedstock was heated to a molten or semi-molten state as it was propelled through an ionized gas en route to the substrate. The coating was deposited in lamellae, parallel to the substrate, until the desired coating thickness was obtained. Each specimen was sectioned diametrically with an electric discharge machine. One half was stored at room temperature while the other was subjected to the predetermined heat treatment (table 1). For examination of the coating microstructure, both halves of each specimen were mounted as shown schematically in figure 4, and then ground and polished using standard metallographic procedures. This method allows convenient comparison of the exposed and unexposed coating microstructure.

Microstructural analysis included metallography and scanning electron microscopy (SEM), and measurement of coating thickness before and after heat treatment with image analysis techniques. Coating thickness was measured as the distance between parallel lines fit through the coating-substrate interface and the top of the coating as displayed in figure 5. Image analysis also enabled measurement of void content within the Ni-Cr constituent after heat treatment by selecting individual Ni-Cr particles and measuring the percentage of black (void) versus white (metal) areas within the image field. Energy-dispersive x-ray spectrometry (EDS) provided chemical composition of microstructural features. The EDS system was controlled through commercially available software that enabled measurement of chemical composition at multiple points or across selected areas on the sample surface.

The general setup for coating adhesion strength testing is shown schematically in figure 6. Six specimens were plasma sprayed for each control case; three were heat treated and three were not. The pull test specimens were prepared with a standard technique [13]. An increasing tensile force was applied to the coating until failure occurred at the *burst pressure*, which was used to calculate the adhesion strength of the coating. After failure, the surface of the coating was examined and the percentage of failure within the coating (*cohesive failure*) was estimated and recorded. Specimens with failure only within the epoxy adhesive would indicate that the coating strength was greater than that of the epoxy (30 MPa). Typically, however, failure is observed within the coating [13].

Results and Discussion

Figure 7 is a plot of the spectra obtained from EDS analysis of the Ni-20Cr and Ni-10Cr powders used in this investigation, which shows the relative concentrations of chromium in the Ni-Cr solid solution. The relative magnitudes of the Cr peaks help verify the expected weight percentages of nickel and chromium in the powder. However, the trace elements in the two powders are not the same. The Ni-20Cr contains aluminum and silicon and Ni-10Cr contains a small amount of bromine. The effect these trace elements have on the oxidation of the resultant coatings is unknown and may require more research.

Selected coating microstructures after heat treatment are shown in figures 8 to 10. Each coating had the lamellar microstructure typical of plasma spray coatings. The coatings with only Ni-20Cr or Ni-10Cr showed no precipitate formation after either 650 or 1090 °C heat treatment. Ni-Cr had no precipitate formation with chromia or silver, as shown in figure 8. However, Ni-20Cr and Ni-10Cr (fig. 9) had 7 and 1 percent precipitate formation, respectively, with the addition of the fluorides, as measured by image analysis. In addition, PS304 and LCR304 (fig. 10) had 10 and 1 percent precipitate formation, respectively. The precipitate formation results are listed in table 1. The change in sample thickness (table 1) suggested that lower chromium content reduced coating growth, but the variation in these measurements warrants further investigation.

Several points on the cross-section of each specimen were examined by EDS. The analysis software also computed the weight percentages of each detected element, which allowed an estimation of the nickel to chromium ratio within Ni-Cr phases. In the Ni-20Cr/BaF₂-CaF₂ coating after heat treatment at 650 °C for 24 hr, the Ni-Cr material between two neighboring precipitates had 91 wt%Ni and 6 wt%Cr, which appears to show a depletion of chromium from Ni-20Cr. Within the precipitate, there was 18 wt%Ni to 67 wt%Cr, which may indicate the presence of a chromium oxide precipitate, chromium oxide lining a pore wall, or the coalescence of chromium near the edge of a pore. Similarly, the Ni-10Cr/BaF₂-CaF₂ coating had 52 wt%Ni to 39 wt%Cr at the edge of a precipitate. For PS304 after the 650 °C heat treatment, the Ni-10Cr phase had 86 wt%Ni to 9 wt%Cr, while the area between two precipitates was 87 wt%Ni to 8 wt%Cr and the edge of the precipitate had 32 wt%Ni to 61 wt%Cr. This result also seems to show a general depletion of chromium between precipitates and a concentration of chromium at the precipitate walls due to diffusion or oxidation of chromium. The LCR304 coating had approximately 90 wt%Ni to 10 wt%Cr within the Ni-Cr particles. Although these results follow the predicted trend, the fact that an electron beam used to perform EDS actually collects the spectra from a volume of material approximately 1 μm below the surface [14] means the spectra may contain information from a neighboring Cr₂O₃ particle in the chromium-containing coatings.

Figure 11 shows an elemental line scan taken across a Ni-Cr particle with a dark feature near its center and a dark grey phase to its right. The chemical composition shown in figure 12(b) indicates that the dark phase within the Ni-Cr particle is Cr₂O₃ as well as the dark grey phase to the right. As stated previously, however, it is unclear whether the Cr₂O₃ particle precipitated within the Ni-Cr particle, was entrapped during deposition or is simply adjacent to (behind) the Ni-Cr particle.

A compositional map of the PS304 coating after 24 hr at 650 °C is shown in figure 12. Comparing the SEM image with the chromium and oxygen maps, the dark areas within the Ni-Cr particles are clearly concentrated with chromium and oxygen (Cr₂O₃). The areas directly adjacent to the chromium oxide concentrations also appear to be Cr-depleted, as shown in the chromium map. This indicates diffusion of Cr to form Cr₂O₃.

The coating adhesion strengths, before and after heat treatment, are summarized in table 1. There was little change in the adhesion strength after heat treatment for the coatings that did not contain fluorides. Therefore, if these materials were considered for future applications, it should be noted that heat treatment may not be required prior to use. However, for Ni-Cr/BaF₂-CaF₂ coatings, the adhesion strength increased approximately 14 percent after heat treatment. The nature of the failure also changed from predominantly within the coating (*cohesive* failure) to the coating-substrate interface (*adhesive* failure), indicating an increase in the cohesive strength of the coating. Additionally, PS304 and LCR304 strength increased 60 and 78 percent, respectively, after heat treatment. These results suggest that the presence of the fluorides has a dramatic effect on coating strength as a function of heat treatment. The reasons for this effect call for continued study.

Summary

The purpose of this investigation was to understand the effect a reduction of chromium content in Ni-Cr powder has on the environmental durability of PS304 coating. Based on the results of the preliminary investigation, the following observations were made:

- The chemical composition of the coating microstructures after heat treatment exhibit a general depletion of chromium from Ni-Cr particles after heat treatment that suggests void formation in the coatings is due to diffusion and oxidation of chromium during heat treatment.
- Reducing the chromium content from 20 to 10 wt% in Ni-Cr powder tended to reduce the appearance of voids after exposure to 650 °C for 24 hr when BaF₂-CaF₂ was present in the coating.

- The chromium concentration had no noticeable effect on void formation after heat treatment when BaF₂-CaF₂ was not present in the coating.
- Heat treatment had a significant effect on coating adhesion strength when BaF₂-CaF₂ was included in the coating composition, but little effect when it was not.

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Table 1. Effect of heat treatment (HT) on coating properties

Coating material	HT, in air	Void formation after HT, percent	Thickness increase, μm	Adhesion strength, MPa/cohesive failure, percent	
				Before HT	After HT
Ni-20Cr	650 °C/24 hr	0	--*	--	--
Ni-20Cr	1090 °C/15 hr	0	43	--	31 / 0
Ni-10Cr	650 °C/24 hr	0	37	34 / 32	36 / 0
Ni-10Cr	1090 °C/15 hr	0	12	--	--
Ni-20Cr/Ag	650 °C/24 hr	0	0	33 / 0	32 / 0
Ni-20Cr/Cr ₂ O ₃	650 °C/24 hr	0	-1	31 / 0	32 / 0
Ni-20Cr/BaF ₂ -CaF ₂	650 °C/24 hr	7	30	28 / 72	32 / 2
Ni-10Cr/BaF ₂ -CaF ₂	650 °C/24 hr	1	16	28 / 97	32 / 0
Ni-20Cr/Cr ₂ O ₃ /Ag/BaF ₂ -CaF ₂ (PS304) [13]	650 °C/24 hr	10	28	20 / --	32 / --
Ni-10Cr/Cr ₂ O ₃ /Ag/BaF ₂ -CaF ₂ (LCR304)	650 °C/24 hr	1	4	18 / 99	32 / 20

*Dashes indicate that no measurement was taken at this condition.

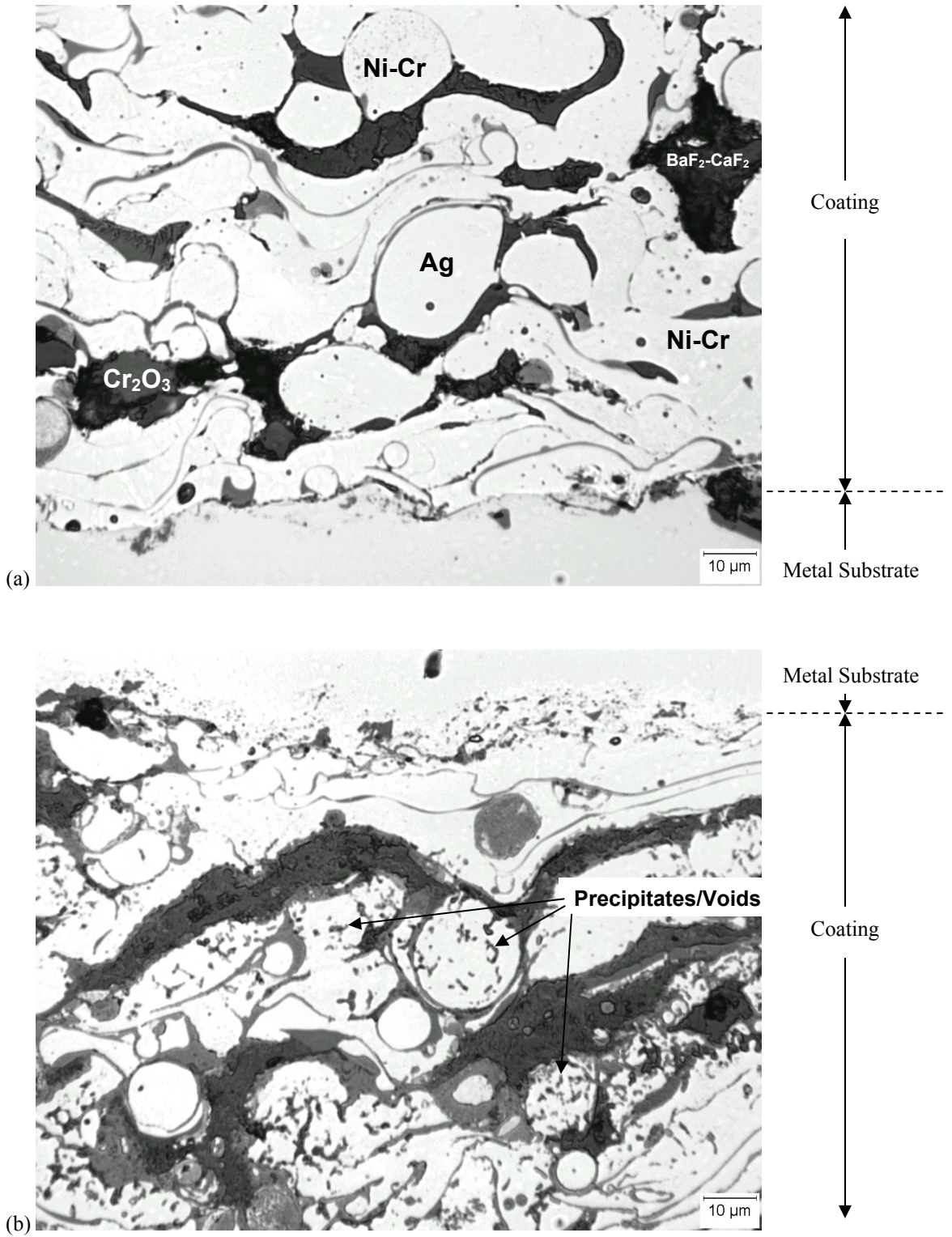
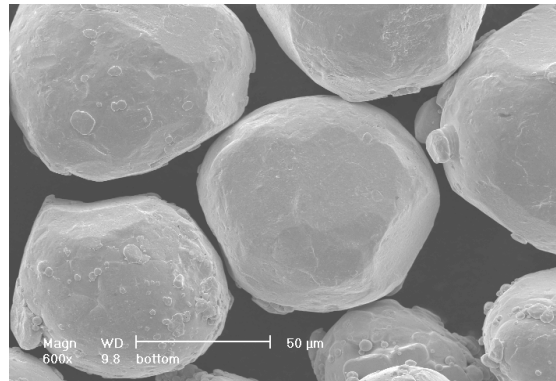


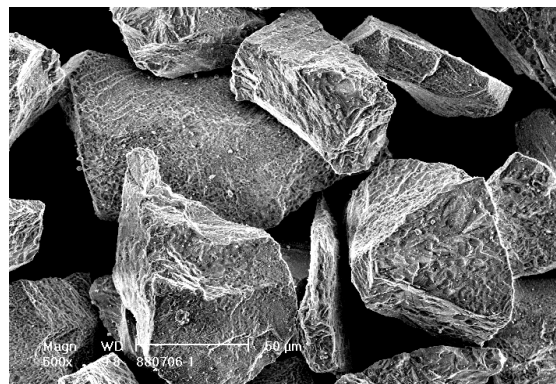
Figure 1. Typical PS304 microstructure (a) before heat treatment (b) after heat treatment for 46 hr at 650 °C (original magnification 500×).



(a) Chromia (Cr₂O₃)



(b) Silver (Ag)



(c) Fluorides (BaF₂-CaF₂)

Figure 2. Chromia, silver and fluoride feedstock powders (original magnification 600×).

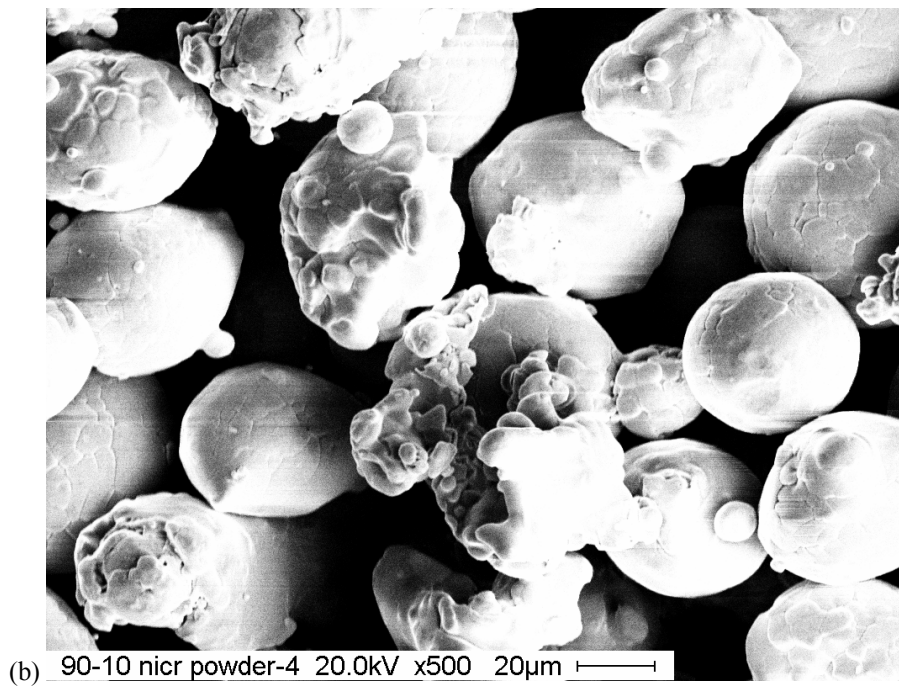
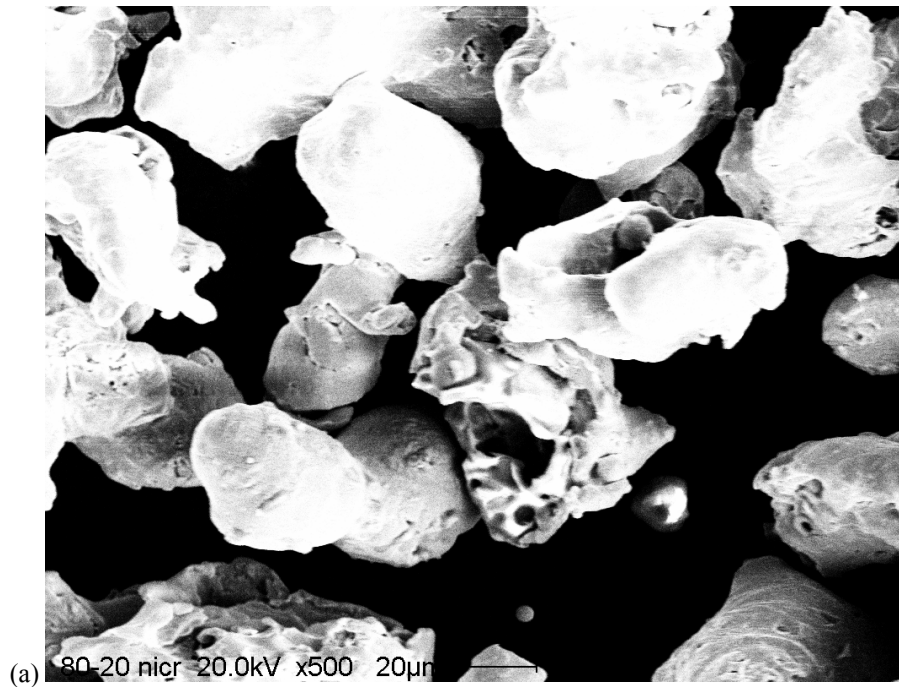


Figure 3. Nickel-chromium powders used in this investigation
(a) Ni-20Cr (b) Ni-10Cr (original magnification 500×).

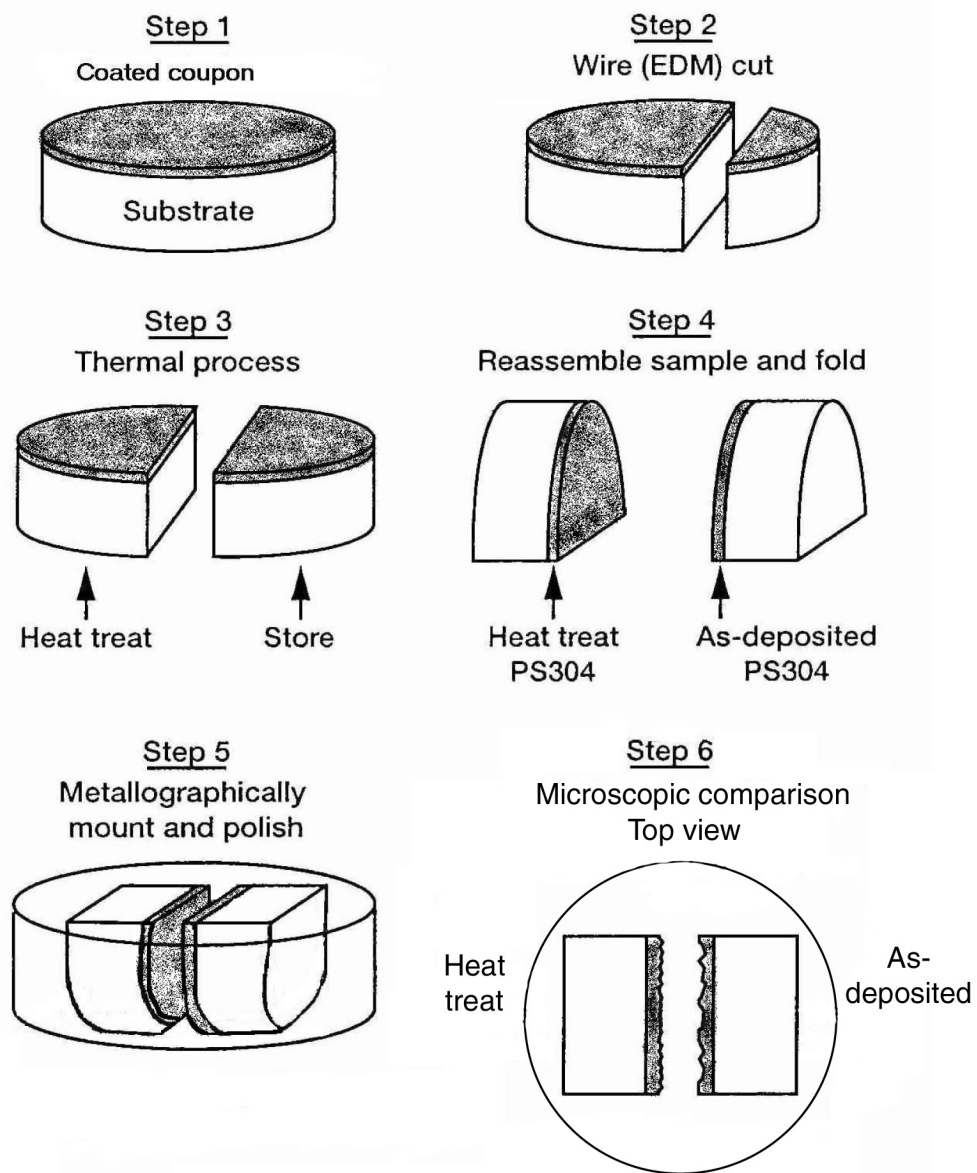


Figure 4. Metallographic mounting procedure.

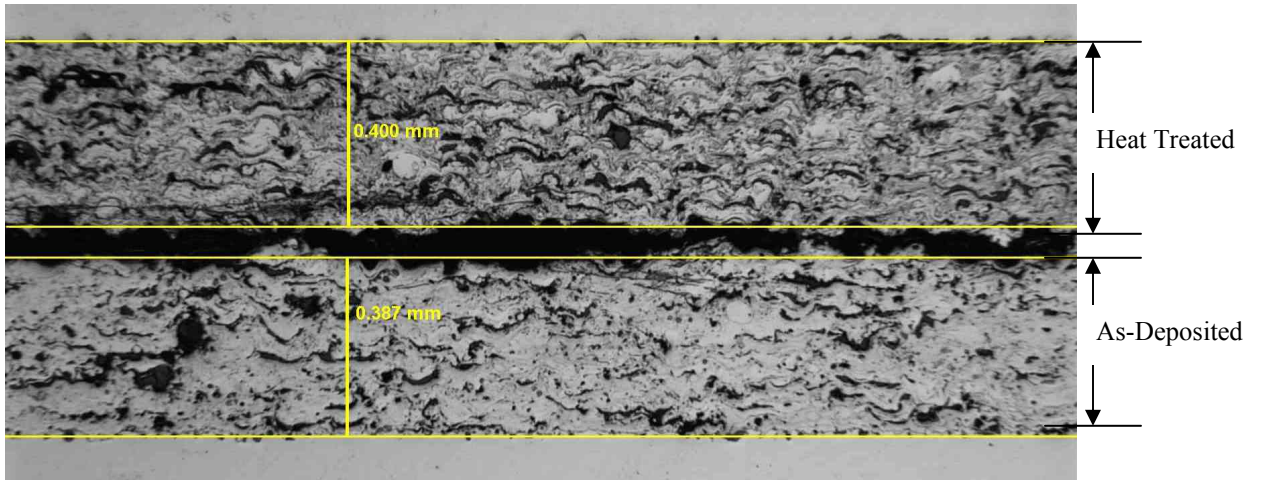


Figure 5. Illustration of a typical coating thickness measurement.

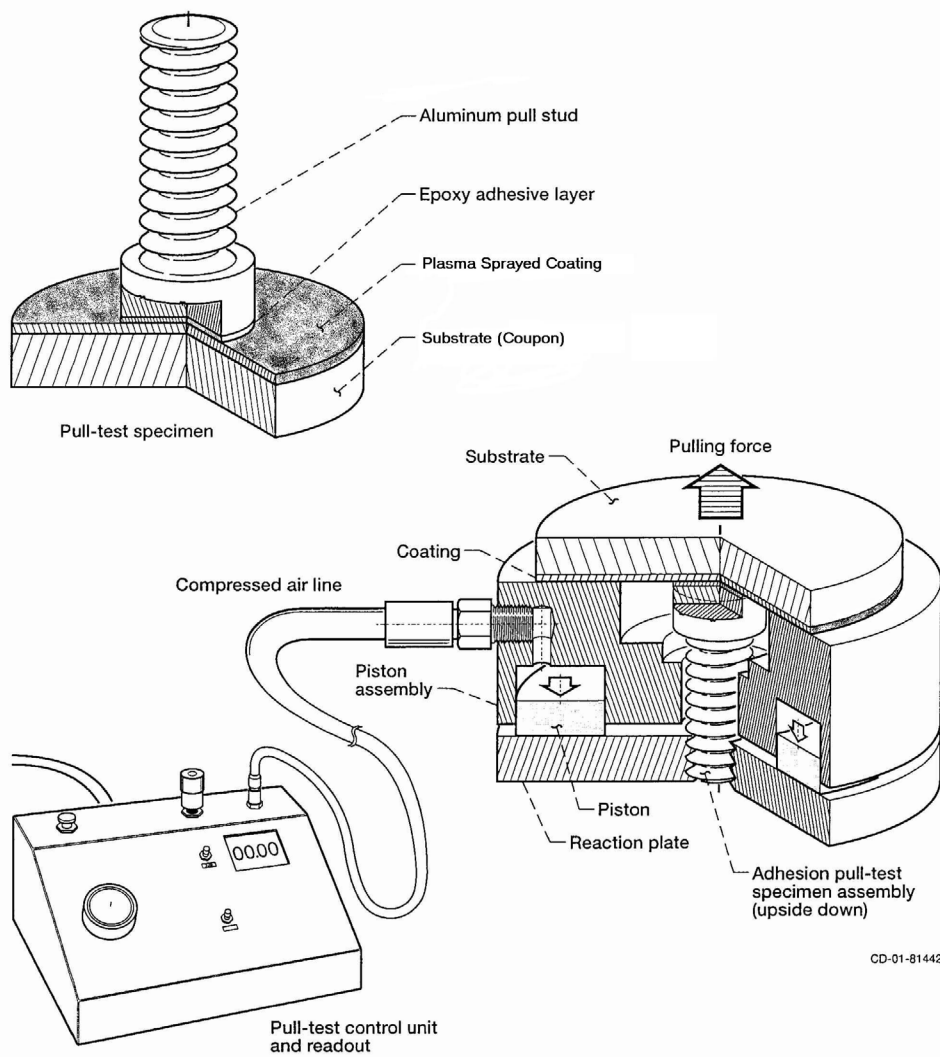
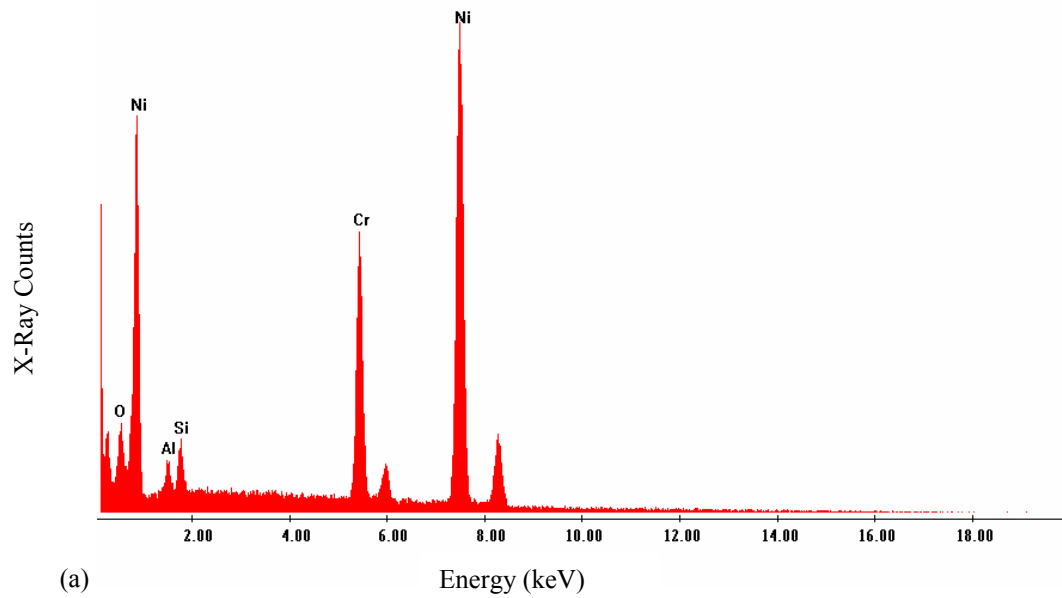
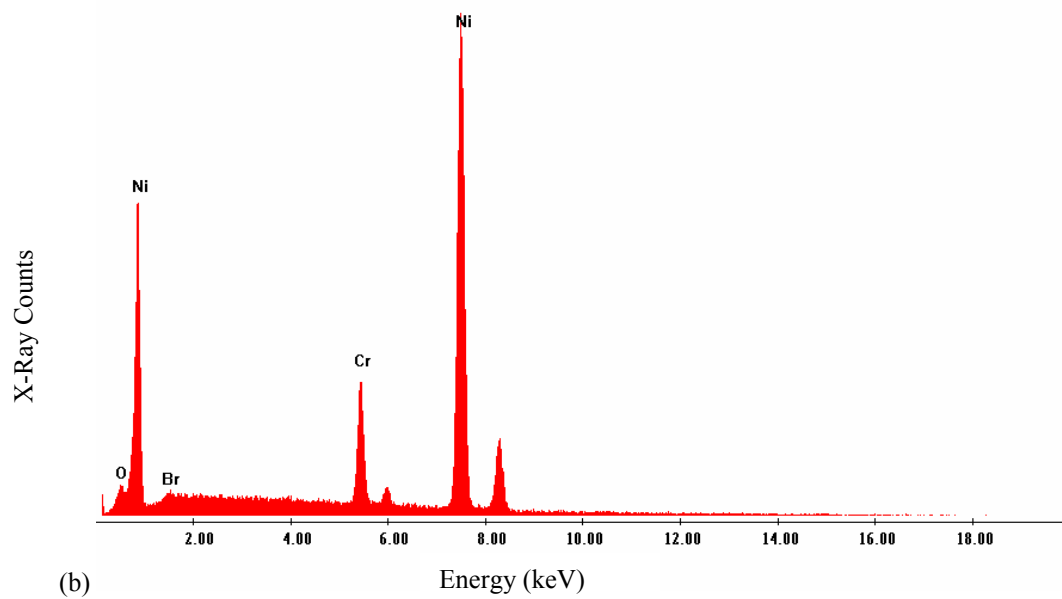


Figure 6. Schematic representation of coating adhesion strength test apparatus.



(a)



(b)

Figure 7. Spectra showing chemical composition of (a) Ni-20Cr (b) Ni-10Cr powders used in this investigation.

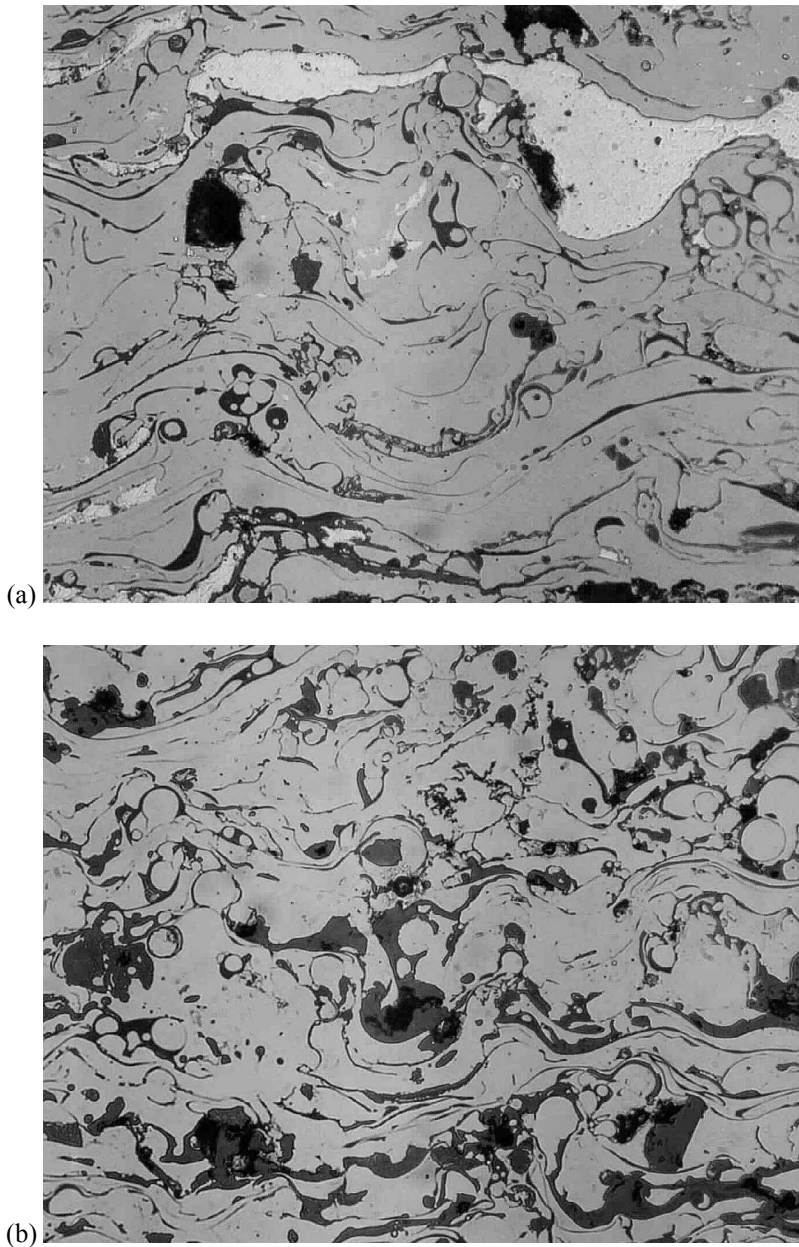


Figure 8. Plasma spray deposited Ni-20Cr with (a) silver (b) chromia after 650 °C/24 hr heat treatment (original magnification 500×). Note the absence of voids or precipitates in the Ni-Cr phase.

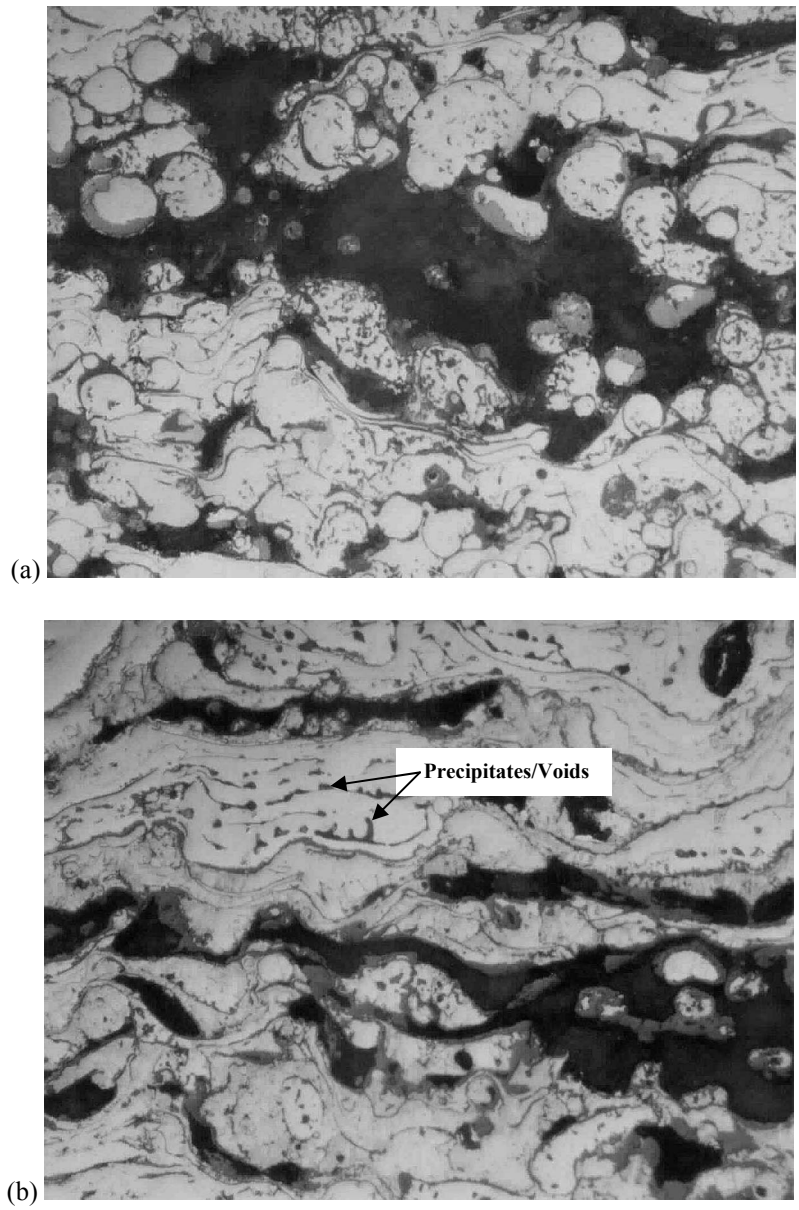


Figure 9. Plasma spray deposited Ni-Cr with BaF₂-CaF₂ (a) Ni-20Cr (b) Ni-10Cr (original magnification 500×). Note the presence of voids and/or precipitates in the Ni-Cr phase.

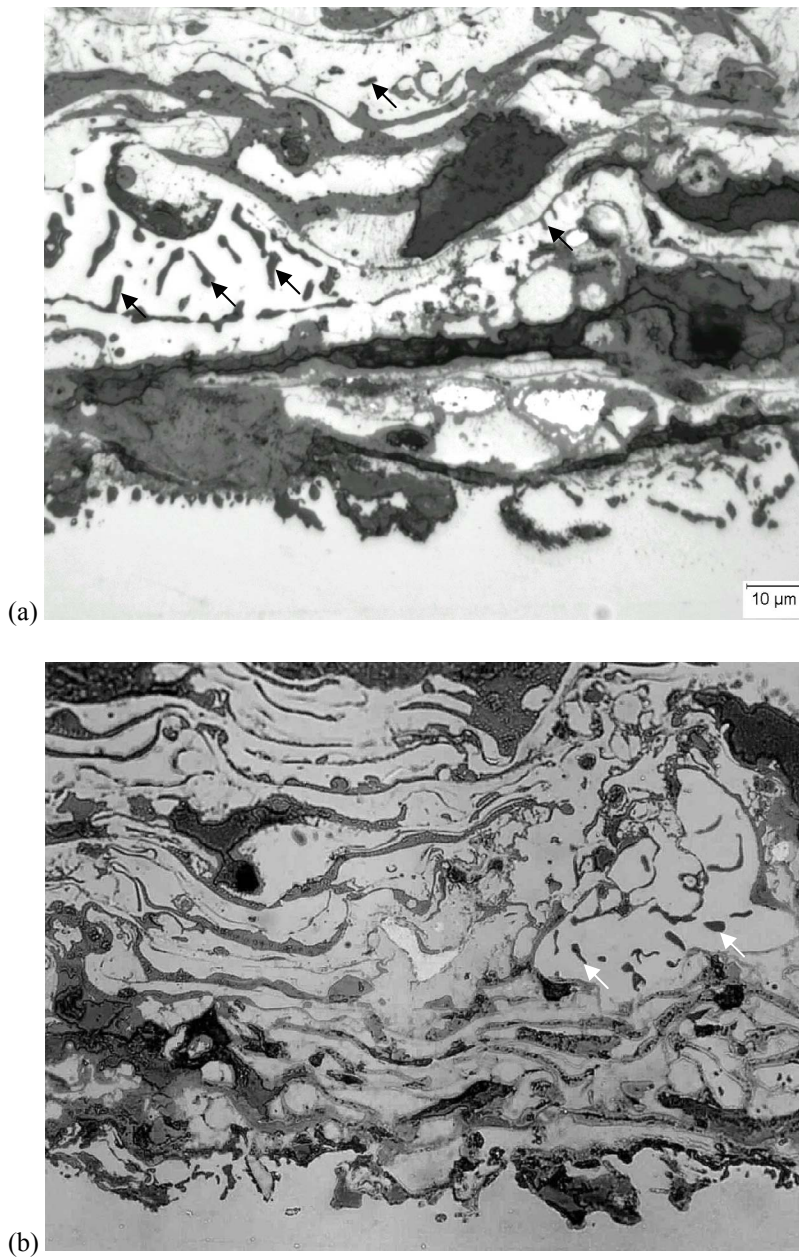


Figure 10. Plasma spray deposited (a) PS304 (b) LCR304 after 650 °C/24 hr heat treatment (original magnification 500×). Note that there are more precipitates/voids (marked with arrows) in the PS304 microstructure than in LCR304.

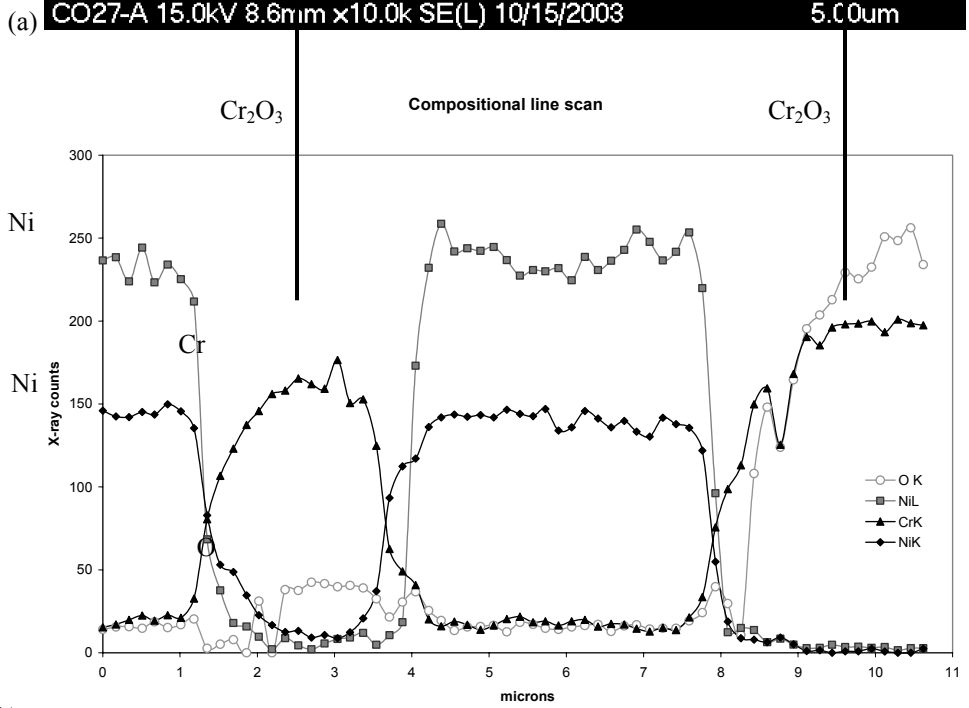
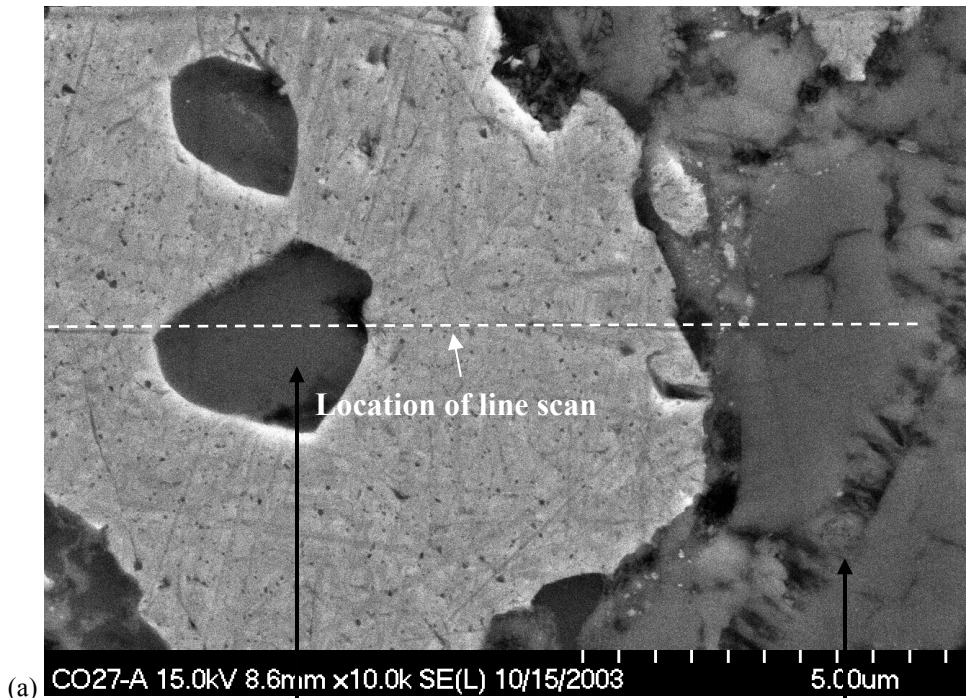


Figure 11. Photomicrograph (a) and compositional line scan plot (b) indicating presence of Cr_2O_3 within a Ni-Cr particle in a PS304 coating cross-section after heat treatment at 650°C for 24 hr.

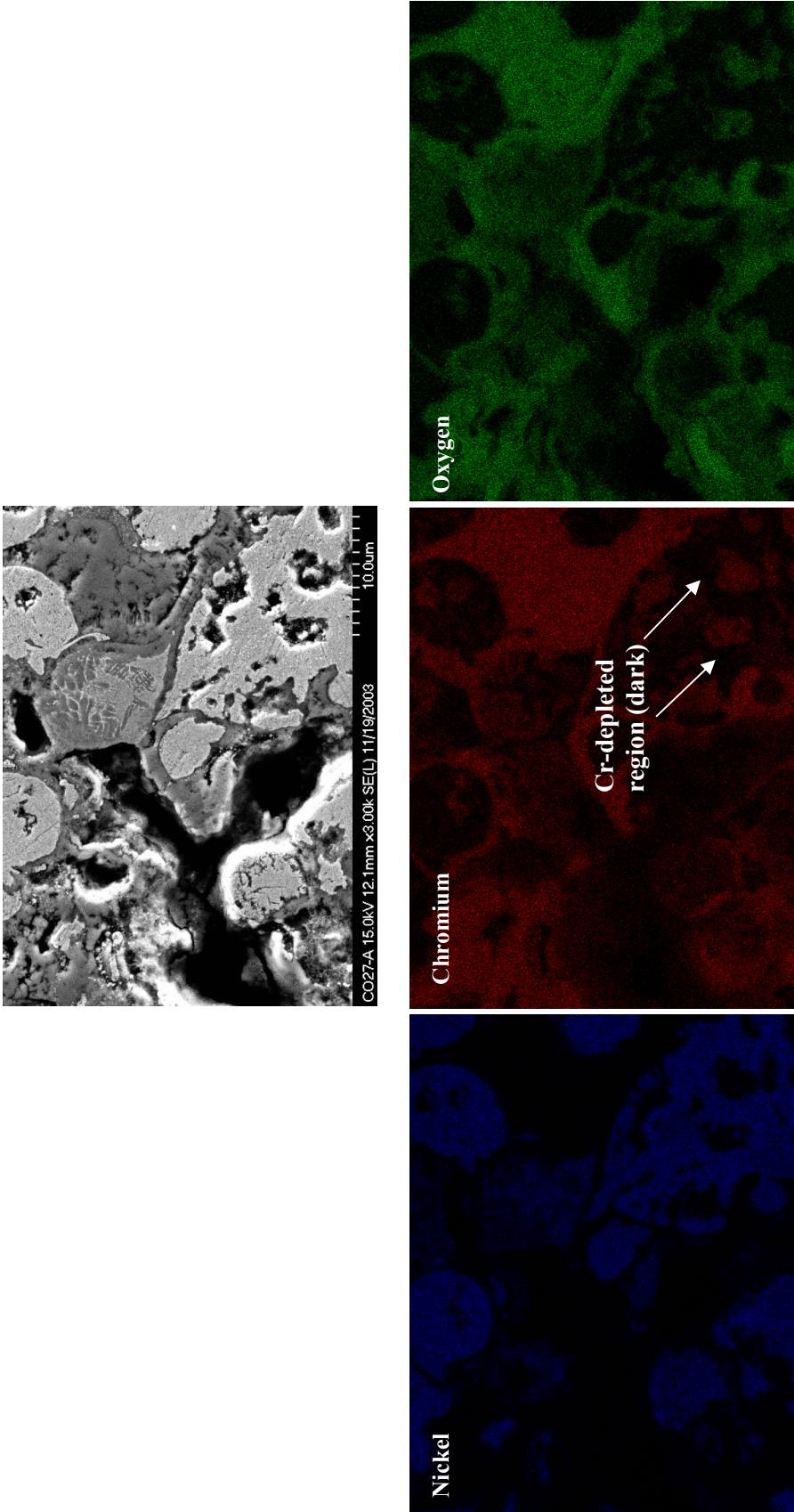


Figure 12. Photomicrograph and elemental maps of nickel, chromium and oxygen in PS304 coating after heat treatment at 650 °C for 24 hr. Note colored areas in the Cr and O maps corresponding to the dark voids or precipitates in the photomicrograph indicating the presence of chromium oxide and the depletion of Cr at the edge of the chromium oxide features as indicated in the Cr map.

REPORT DOCUMENTATION PAGE

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1. AGENCY USE ONLY (<i>Leave blank</i>)		2. REPORT DATE June 2004	3. REPORT TYPE AND DATES COVERED Technical Memorandum	
4. TITLE AND SUBTITLE Thermal Effects on a Low Cr Modification of PS304 Solid Lubricant Coating			5. FUNDING NUMBERS WBS-22-708-18-04	
6. AUTHOR(S) Malcolm K. Stanford, Anne M. Yanke, and Christopher DellaCorte				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) National Aeronautics and Space Administration John H. Glenn Research Center at Lewis Field Cleveland, Ohio 44135-3191			8. PERFORMING ORGANIZATION REPORT NUMBER E-14585	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) National Aeronautics and Space Administration Washington, DC 20546-0001			10. SPONSORING/MONITORING AGENCY REPORT NUMBER NASA TM-2004-213111	
11. SUPPLEMENTARY NOTES Malcolm K. Stanford and Christopher DellaCorte, NASA Glenn Research Center; and Anne M. Yanke, Cleveland State University, Cleveland, Ohio 44115. Responsible person, Malcolm Stanford, organization code 5960, 216-433-3387.				
12a. DISTRIBUTION/AVAILABILITY STATEMENT Unclassified - Unlimited Subject Category: 23 Available electronically at http://gltrs.grc.nasa.gov This publication is available from the NASA Center for AeroSpace Information, 301-621-0390.			12b. DISTRIBUTION CODE	
13. ABSTRACT (<i>Maximum 200 words</i>) PS304 is a high temperature composite solid lubricant coating composed of Ni-Cr, Cr ₂ O ₃ , BaF ₂ -CaF ₂ and Ag. The effect of reducing chromium content on the formation of voids in the Ni-Cr particles after heat treatment in PS304 coating was investigated. Coatings were prepared with Ni-20Cr or Ni-10Cr powder and in various combinations with the other constituents of PS304 (i.e., chromia, silver and eutectic BaF ₂ -CaF ₂ powders) and deposited on metal substrates by plasma spray. Specimens were exposed to 650 °C for 24 hr or 1090 °C for 15 hr and then examined for changes in thickness, coating microstructure and adhesion strength. Specimens with Ni-10Cr generally had less thickness increase than specimens with Ni-20Cr, but there was great variance in the data. Reduction of chromium concentration in Ni-Cr powder tended to reduce the appearance of voids in the Ni-Cr phase after heat exposure. The presence of BaF ₂ -CaF ₂ resulted in a significant increase in coating adhesion strength after heat treatment, while coatings without BaF ₂ -CaF ₂ had no significant change. Chemical composition analysis suggested that the void formation was due to oxidation of chromium in the Ni-Cr constituent.				
14. SUBJECT TERMS Coatings; Lubrication; Environmental durability; Oxidation			15. NUMBER OF PAGES 22	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified	20. LIMITATION OF ABSTRACT	

