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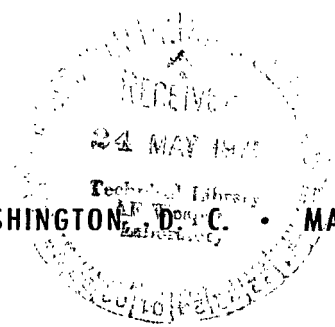
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EXPLORATORY STUDY OF  
OXIDATION-RESISTANT ALUMINIZED  
SLURRY COATINGS FOR IN 100  
AND WI-52 SUPERALLOYS

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16. Abstract <p>Two coatings, (1) nickel-aluminide and (2) iron-aluminide plus iron-chromium-aluminum, were produced on IN 100 and WI-52 by a two-step coating process: slurry spraying followed by pack aluminizing. The coated superalloys were cyclically furnace tested in air at 1093° C for 300 hr. Nickel-aluminide coatings offered good protection from oxidation throughout the test, whereas the iron-aluminide plus iron-chromium-aluminum coatings were protective to only 200 hr. Nickel-aluminide-coated IN 100 also survived 300 hr of testing in a high-velocity burner rig at 1093° C.</p>			
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# EXPLORATORY STUDY OF OXIDATION-RESISTANT ALUMINIZED SLURRY COATINGS FOR IN 100 AND WI-52 SUPERALLOYS

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## SUMMARY

An investigation was made to study the feasibility of producing oxidation-resistant coatings for superalloys by slurry techniques. Lacquer slurries of nickel powder and an iron-30-percent-chromium powder mixture were sprayed on IN 100 and WI-52. These slurries were subsequently pressed, sintered, and pack aluminized to form (1) nickel-aluminide (NiAl) and (2) iron-aluminide (FeAl) plus iron-chromium-aluminum (Fe-Cr-Al) coatings.

These four systems were oxidized in static air at 1093<sup>o</sup> C for 300 hr. The test results included weight change data, metallography, and X-ray diffraction analyses.

The oxidation resistance of the NiAl aluminized slurry coatings compared well with that of commercial aluminide coatings on both IN 100 and WI-52 superalloys. The slurry coatings remained intact after 300 hr of cyclic furnace testing at 1093<sup>o</sup> C. The NiAl aluminized slurry coating on IN 100 also survived burner rig testing for 300 hr at 1093<sup>o</sup> C.

The performance of the FeAl plus Fe-Cr-Al aluminized slurry coating on IN 100 compared well to an iron-chromium-aluminum-yttrium (Fe-Cr-Al-Y) cladding of similar thickness on IN 100, but the slurry coating was converted by interdiffusion to a Ni-base material during the 300 hr of testing at 1093<sup>o</sup> C. The FeAl plus Fe-Cr-Al aluminized slurry coating on WI-52 was inferior to an Fe-Cr-Al-Y cladding of similar thickness on WI-52. In 300 hr this slurry coating was obviously degraded by oxidation at 1093<sup>o</sup> C.

## INTRODUCTION

Advanced gas turbine engines for some military and commercial aircraft will require metal temperatures in excess of 980<sup>o</sup> C in order to increase operating efficiency. The nickel- and cobalt-base superalloys used as blades and vanes in these engines must

be coated to resist oxidation and to extend their useful lifetime when used at such high temperatures. Aluminide diffusion coatings are most commonly used to protect superalloys in current engines. By interdiffusion with aluminum, the superalloy surfaces are converted to complex nickel or cobalt aluminides. These coatings are presently used for thousands of hours at temperatures below  $980^{\circ}\text{C}$ , but must be improved for extensive use at higher temperatures.

Controlled composition modifications of aluminide coatings is one approach to increasing their oxidation resistance. For example, diffusion-inhibiting oxides (ref. 1) and aluminum-rich phases added to the coatings may increase their time of protection. However, it is extremely difficult to control the types, compositions, and amounts of coating phases by using conventional pack cementation coating techniques. Also, the coating phases produced by such processes are often limited to those whose compositions are inherently related to substrate chemistry. Development of more versatile coating processes which can deposit a controlled number and distribution of coating phases is needed. One successful laboratory approach to providing improved oxidation resistance has been the pressure bonding of 0.127-mm oxidation-resistant metal clads to superalloys (ref. 2). Both iron-chromium-aluminum-yttrium (Fe-Cr-Al-Y) and nickel-chromium-aluminum (Ni-Cr-Al) clads offered protection for the cobalt-base alloy WI-52, and a Ni-Cr-Al clad offered protection for the nickel-base alloy IN 100 at  $1093^{\circ}\text{C}$  in static air. Other oxidation-resistant materials not easily fabricated into thin sheet cannot be used as claddings. However, coating application of oxidation-resistant compositions by slurry techniques offers promise because the desired coating additions can be made easily by powder additions to the slurry before application.

The purpose of this investigation was twofold: (1) to determine the feasibility of depositing simple 0.05- to 0.10-mm oxidation-resistant coatings on nickel- and cobalt-base superalloys using slurry techniques, and (2) to evaluate the oxidation resistance of the slurry-coated materials in comparison to that of similar coatings produced by cladding or pack cementation techniques. Two coatings, (1) nickel-aluminide (NiAl) and (2) iron-aluminide (FeAl) plus Fe-Cr-Al, were applied to IN 100 and to WI-52 by a duplex slurry-sinter and pack aluminizing process. The four systems were oxidized in  $1093^{\circ}\text{C}$  cyclic furnace tests for 300 hr. Nickel-aluminide-coated IN 100 was also tested in a Mach 1 burner rig at  $1093^{\circ}\text{C}$  for 300 hr. The processing and oxidation resistance of the aluminized slurry coatings were evaluated by metallographic, X-ray diffraction, and gravimetric analyses.

## EXPERIMENTAL PROCEDURE

### Materials and Specimen Preparation

A nickel slurry was made by mixing carbonyl nickel powder (3  $\mu\text{m}$  average diameter) with nitrocellulose lacquer. A 70Fe-30Cr powder mixture slurry was made by mixing carbonyl iron powder (10  $\mu\text{m}$  average diameter) and electrolytic chromium powder (-325 mesh) with nitrocellulose lacquer. (All compositions are in weight percent.) The substrate materials coated were nickel-base IN 100 and cobalt-base WI-52 superalloys. Nominal IN 100 composition was nickel - 15 cobalt - 10 chromium - 3 molybdenum - 5.5 aluminum - 4.7 titanium - 0.18 carbon (Ni-15Co-10Cr-3Mo-5.5Al-4.7Ti-0.18C); nominal WI-52 composition was cobalt - 21 chromium - 11 tungsten - 2 columbium - 2 iron - 0.45 carbon (Co-21Cr-11W-2Cb-2Fe-0.45C). The specimens were 0.25- by 2.54- by 5.08-cm castings.

The specimens were prepared for coating by ball milling with alumina followed by ultrasonic cleaning in trichloroethylene. The Ni and Fe-Cr slurries were then sprayed onto these specimens by a conventional paint-spray gun. The slurry-coated specimens were air dried and vacuum sealed in a plastic bag. The bagged specimens were cold isostatically pressed at  $482 \text{ MN/m}^2$  (70 000 psi) to improve the density of the green coating. The sprayed specimens were then sintered in a 0.010-torr (10- $\mu\text{m}$ -Hg) vacuum at  $1093^\circ \text{C}$  for 4 hr.

Pack aluminizing of the sintered coating was performed in flowing argon (500  $\text{cm}^3/\text{min}$ ) at  $1093^\circ \text{C}$  in a pack of 98 percent aluminum oxide (-100 mesh powder), 1 percent Al (-100 mesh powder), and 1 percent sodium chloride activator. The Ni-coated specimens were aluminized for 18 hours so as to add  $13 \text{ mg/cm}^2$  aluminum and to convert only the nickel slurry to the NiAl intermetallic compound. The Fe-Cr-coated specimens were pack aluminized for 9 hr to add  $10 \text{ mg/cm}^2$  aluminum in order to produce an average coating composition of Fe-25Cr-20Al. However, since the aluminizing process is diffusion controlled, there is an aluminum concentration gradient from the as-coated surface to the substrate interface.

Originally aluminum was incorporated as an elemental powder in the slurry, but this hindered the sintering and densification of the coating. By pack aluminizing in a second step, good sintering of the slurry coating was allowed. An additional advantage to this two-step procedure was that pack aluminizing removed voids which remained in the coating after sintering.

## Cyclic Oxidation Testing

Furnace tests. - Furnace oxidation tests were performed at  $1093^{\circ}\pm 5^{\circ}$  C for 15 cycles of 20 hr duration each (300 hr total). Each specimen rested on a high-purity alumina boat, and each boat was placed in its own horizontal alumina tube of a multitube furnace. Air flowed through these tubes at a rate of about  $500\text{ cm}^3/\text{min}$ . After each cycle the specimens were removed from the furnace and weighed to the nearest 0.1 mg. Any loose oxide was then brushed off, and the specimens were weighed again to obtain an indication of the amount of weakly adherent oxide scale.

Burner rig tests. - A NASA burner rig test bar of IN 100 was slurry coated with nickel and pack aluminized in the same way as were the furnace test specimens. The burner bar configuration was 0.64 by 2.54 by 10.16 cm and had a tapered leading edge (ref. 3). The specimen was mounted on the edge of a turntable rotating at 900 rpm, and it was exposed to a Mach 1 stream of natural gas combustion products (fuel-air ratio, 1/30) at  $1093^{\circ}\pm 8^{\circ}$  C (ref. 3). High-temperature exposures were 1 hr each, followed by air jet cooling to  $93^{\circ}$  C. Every 20 cycles the specimen was removed and weighed to the nearest 0.1 mg. After 300 hr of exposure the test was stopped and the specimen was analyzed. The maximum hot zone area was estimated to be  $30\text{ cm}^2$  (ref. 3). Thus, by dividing the weight changes by  $30\text{ cm}^2$ , the burner rig test data could be roughly compared to gravimetric furnace oxidation data.

## Evaluation Techniques

X-ray diffractometer scans of the as-sintered, as-aluminized, and as-oxidized coating surfaces were obtained on the furnace test specimens. Debye-Scherrer X-ray diffraction powder patterns were obtained from oxide scrapings of the burner bar. Copper- $K\alpha$  radiation was used with a nickel filter for these X-ray diffraction analyses. Metallographic analyses of cross sections of the as-sintered, as-aluminized, and as-oxidized specimens were also obtained.

All polished IN 100 metallographic specimens were etched electrolytically at 3V in an electrolyte of  $50\text{ cm}^3$  of glycerine,  $20\text{ cm}^3$  of water,  $30\text{ cm}^3$  of hydrochloric acid,  $10\text{ cm}^3$  of nitric acid, and  $1\text{ cm}^3$  of hydrofluoric acid. All the WI-52 specimens were first etched electrolytically at 3V in an electrolyte of  $70\text{ cm}^3$  of 5 percent sulfuric acid and  $30\text{ cm}^3$  of water and boric acid (saturated). They were then immersion etched in a solution of  $33\text{ cm}^3$  of acetic acid,  $33\text{ cm}^3$  of nitric acid,  $33\text{ cm}^3$  of water, and  $1\text{ cm}^3$  of hydrofluoric acid.

## RESULTS AND DISCUSSION

Coating thicknesses and phases for as-sintered, as-aluminized, and as-oxidized specimens are summarized in table I. These data will be discussed in the following paragraphs.

### Aluminized Nickel Coatings

Sintered coatings. - Pressing and sintering of the nickel slurries resulted in relatively dense, adherent nickel coatings on IN 100 and WI-52. Microstructures of the cross sections are shown in figure 1. Approximately 0.04 to 0.05 mm of 90 percent dense nickel coating was deposited on both substrates. (Coating densities were approximated visually from the photomicrographs. Coating thicknesses were measured from the photomicrographs.) A diffusion layer greater than 0.030 mm was formed in both cases where the strengthening phases in the substrates were dissolved. The only surface phase shown by X-ray diffraction was face-centered-cubic (fcc)  $\gamma$ -Ni solid solution.

Aluminized coatings. - Pack aluminizing of the nickel coatings resulted in a NiAl coating on both IN 100 and WI-52. These aluminized nickel coatings are shown in figure 2. The outer aluminide phase on IN 100 was about 0.052 mm thick, followed by a 0.040 mm two-phase region. The outer aluminide phase on WI-52 was 0.088 mm thick, followed by 0.024 mm of two-phase region plus depleted zone. Almost all porosity was removed by the aluminizing process. The dark and light etching layers in the NiAl coating correspond to high and low aluminum concentrations, respectively (ref. 4). X-ray diffractometer scans of the coating surfaces identified  $\beta'$ -NiAl as the only phase on both coated IN 100 and coated WI-52.

Oxidized coatings. - After 300 hr of cyclic furnace oxidation at 1093<sup>o</sup> C the NiAl-slurry-coated IN 100 and WI-52 performed as well as the commercial pack aluminide coatings on the same superalloys. Cyclic oxidation weight change data for these NiAl aluminized slurry coatings are shown in figure 3. Data for widely used commercial aluminide conversion coatings on IN 100 (unpublished data obtained by M. A. Gedwill, NASA Lewis Research Center) and WI-52 (ref. 5) are also presented.

As evidenced by the small, steadily increasing weight gains in figure 3(a), the NiAl slurry coating on IN 100 provided oxidation protection for the full 300 hr of testing. The plateau region of the weight change curve indicated that a slow-growing, nonspalling oxide film had formed on the coating. No spalling was observed visually, and at each weighing there was no appreciable oxide spall brushed off. The total weight gain of only 0.5 mg/cm suggested that only a small amount of coating had been consumed by oxidation.

A polished cross section of this oxidized specimen is shown in figure 4(a). No continuous oxide scale was observed in the photomicrographs of this and the other oxidized coatings of this study. This is common for highly oxidation-resistant coatings, probably since only a very thin layer of scale is present. The outer layer of NiAl increased in thickness from 0.052 to 0.068 mm, and the diffusion zone increased from 0.040 to 0.060 mm (see table I). The striated structure in the coating was typical of the NiAl martensite phase which has been recently identified in oxidized NiAl coatings at this laboratory (ref. 6). An X-ray diffractometer scan of this coating surface identified the  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> and NiAl martensite phases (ref. 7). It is interesting to note that none of the carbide phases usually seen beneath an aluminide conversion coating on a nickel superalloy were observed metallographically. This would be expected in contrast to normal conversion coatings, since no nickel was required to diffuse outward from the substrate to form the surface aluminide and leave a heavy concentration of carbide-forming alloying elements.

The NiAl aluminized slurry coating on WI-52, however, was not as protective as on IN 100. Weight gain data in figure 3(b) show that a weight loss of 1.5 mg/cm<sup>2</sup> resulted after 300 hr at 1093<sup>o</sup> C. The specimen weight before and after brushing off the loose surface oxide is shown by the top and bottom, respectively, of the data bars. An average of 0.5 mg/cm<sup>2</sup> of oxide was brushed off after each cycle. The weight gain curve for slurry-coated WI-52 was very similar to that for a widely used commercial aluminide conversion coating on WI-52 tested at 1093<sup>o</sup> C.

The microstructure of the oxidized slurry coating on WI-52 is shown in figure 4(b). Again striations were present in the coating which indicated that martensite had formed in the NiAl coating. The thickness of this NiAl phase, though, had decreased from 0.088 to 0.028 mm. Also it appeared that oxides were formed up to 0.064 mm beneath the coating. Metallography of the commercially aluminized WI-52, however, showed that coating to have been completely consumed after only 200 hr at 1093<sup>o</sup> C (ref. 5). Thus, the NiAl coating was more protective than the CoAl coating which forms naturally when WI-52 is aluminized. An X-ray diffractometer scan of the oxidized slurry coating surface identified the  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> and spinel (lattice parameter  $a_o = 8.16 \times 10^{-10}$  m; 8.16 Å; probably nickel aluminate (NiAl<sub>2</sub>O<sub>4</sub>)) oxides. The metallic coating surface phases identified after oxidation were fcc  $\gamma$ -Ni solid solution and/or  $\gamma'$ -Ni<sub>3</sub>Al. An X-ray diffractometer scan was obtained after 0.025 mm of the surface was ground off and the diffraction pattern for NiAl martensite was identified.

## Aluminized Iron-Chromium Coatings

Sintered coatings. - Sintering of pressed Fe-30Cr powder mixture slurries resulted in adherent, slightly porous iron-chromium coatings on IN 100 and WI-52. Microstruc-



tures of the as-sintered coatings are shown in figure 5. As determined from the photomicrographs, the coatings were from 0.064 to 0.089 mm thick and were approximately 75 percent dense. X-ray diffractometer scans of the coating surfaces identified the body-centered cubic (bcc)  $\alpha$ -Fe(Cr) phase(s) on both substrates.

Aluminized coatings. - Pack aluminizing of the sintered coatings resulted in dense FeAl plus Fe-Cr-Al coatings on both IN 100 and WI-52. The microstructures of these aluminized coatings are shown in figure 6. The aluminized coatings were about 0.10 mm thick (see table I). X-ray diffractometer scans of the coating surfaces identified the ordered  $\beta_2$ -FeAl intermetallic compound. An X-ray diffractometer scan was also obtained after 0.05 mm was ground off the coatings. Here a bcc phase, presumably Fe-Cr-Al solid solution, was identified for both substrates. On WI-52,  $\text{Cr}_{23}\text{C}_6$  was also identified. The dispersed phase in the coating on IN 100 was not identified.

Oxidized coatings. - Cyclic furnace oxidation tests at 1093<sup>o</sup> C showed that the oxidation resistance of the 0.10-mm FeAl plus Fe-Cr-Al aluminized slurry coatings on IN 100 and WI-52 were comparable to 0.127-mm Fe-25Cr-4Al-1Y clads on the same superalloys. Weight gain data for the aluminized slurry coatings and the clads (ref. 2) are shown in figure 7.

From the weight gain curves in figure 7(a) the aluminized slurry coating on IN 100 gained only 1.3 mg/cm<sup>2</sup> in the initial 200 hr of testing, whereas the clad specimen gained 3.4 mg/cm<sup>2</sup>. No appreciable loose oxide brushed off the slurry coating until after 200 hr, as shown by the data bars. However, after 200 hr the slurry-coated specimen began to lose weight and continued to do so until the test was stopped at 300 hr. (Testing of the cladding was stopped at 200 hr.)

The microstructure of the oxidized slurry-coated IN 100 is shown in figure 8(a). No oxidation of the substrate had occurred, but the diffusion zone had increased from 0.02 to 0.15 mm. An X-ray diffractometer scan of the oxide surface identified  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> and a complex spinel ( $a_0 = 8.22 \times 10^{-10}$  m; 8.22 Å). Face-centered cubic  $\gamma$ -Ni solid solution and/or  $\gamma'$ -Ni<sub>3</sub>Al was identified as the major coating phase. This indicated that the bcc  $\beta_2$ -FeAl and the bcc  $\alpha$ -Fe-Cr-Al phase had been converted to the fcc Ni phase(s) by extensive interdiffusion with the substrate. Another X-ray diffractometer scan of the coating surface was obtained after this fcc light-etching phase had been ground off. The dark etching layer was identified as an ordered cesium chloride (CsCl) type bcc phase, presumably NiAl and FeAl. A weaker pattern for a metal-carbide phase, probably TiC, was also observed.

From the weight gain curves of figure 7(b) the 0.100-mm FeAl plus Fe-Cr-Al slurry coating on WI-52 was not as protective as the 0.127-mm Fe-25Cr-4Al-1Y cladding on WI-52 (ref. 2). After 240 hr the slurry-coated specimen began to lose weight very rapidly, and the amount of oxide brushed off (data bars) became considerable.

The microstructure of the oxidized slurry coating in figure 8(b) shows that void formation and internal oxidation were prevalent. An X-ray diffraction scan of the oxidized surface identified  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>, a complex spinel ( $a_o = 8.34 \times 10^{-10}$  m; 8.34 Å), and chromium oxide (Cr<sub>2</sub>O<sub>3</sub>). The fcc  $\gamma$ -Co solid solution phase was the only metallic phase observed by X-ray diffraction. Similar to the FeAl plus Fe-Cr-Al slurry-coated IN 100, this indicated that the bcc FeAl plus Fe-Cr-Al phases had been converted to the fcc Co phase by extensive interdiffusion with the substrate. Thus, the FeAl plus Fe-Cr-Al aluminized slurry coating on WI-52 was not very protective; much oxide spalling and coating degradation had occurred after 300 hr at 1093° C.

### High-Velocity Burner Rig Testing of Nickel-Aluminide-Slurry-Coated IN 100

From the results of cyclic furnace oxidation tests of the four systems, NiAl-slurry-coated IN 100 was the system with the best oxidation resistance. This system was therefore given a preliminary test under the more severe conditions in the Mach 1 burner rig at 1093° C. From weight gains and metallography this NiAl coating was shown to be protective in high-velocity testing also.

The burner rig weight gain data for the NiAl slurry coating and for a very protective commercial aluminide conversion coating (unpublished data obtained from M. A. Gedwill, NASA Lewis Research Center) are compared in figure 9. The aluminized slurry coating curve exhibited a plateau at 20 mg and gradually increased to only 30 mg. Because the specimen area at 1093° C was approximately 30 cm<sup>2</sup>, the specific weight gain was less than 1 mg/cm<sup>2</sup> after 300 hr of testing. This was as good or better than the performance of the commercial aluminide coating, which showed a distinct weight loss after 260 hr.

The appearance of the specimen surface after 300 hr of testing is shown in figure 10. The overall surface was gray, and scrapings from these areas were identified by X-ray diffraction powder patterns to be  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>. The areas marked A<sub>1</sub> and A<sub>2</sub> were light blue in color, and the oxide scrapings here were identified by X-ray diffraction powder patterns to be  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> plus a spinel ( $a_o = 8.05 \times 10^{-10}$  m; 8.05 Å; probably NiAl<sub>2</sub>O<sub>4</sub>). At B the coating on the trailing edge blistered. Oxides here were identified as nickel oxide (NiO) plus a complex spinel ( $a_o = 8.20 \times 10^{-10}$  m; 8.20 Å). Except for this blister and some small spot failures believed to be caused by nonuniform processing, the specimen and coating were intact. The commercial aluminide coating was only slightly degraded by oxidation, but a thermal fatigue crack first observed after only 120 hours (or cycles) had propagated through about one-quarter of the width of the specimen after 300 hr. The absence of thermal fatigue cracks in the slurry-coated IN 100 bar suggests that it is a more desirable system.

The microstructure of the leading edge of the slurry-coated burner bar is shown in figure 11. From a starting thickness of 0.113 mm (4.5 mils), approximately 0.120 mm of the protective NiAl phase remained, as evidenced by the striated NiAl martensite grains. The lightly etched phase on the outer surface of the NiAl grains was identified by an X-ray diffractometer scan to be fcc  $\gamma$ -Ni and/or  $\gamma'$ -Ni<sub>3</sub>Al. In general, the NiAl slurry coating was shown to be intact after 300 hr of high-velocity testing at 1093° C and appeared to have considerable useful life remaining.

## SUMMARY OF RESULTS

A two-step slurry plus pack aluminizing process was developed as a versatile way of depositing oxidation-resistant coatings on nickel- and cobalt-base superalloys. Slurries of nickel powder and of a mixture of iron and chromium powders were successfully sintered and subsequently pack aluminized on IN 100 and WI-52. The resulting coatings of both (1) nickel-aluminide and (2) iron-aluminide plus iron-chromium-aluminum on both IN 100 and WI-52 were 100 percent dense and were 0.05 to 0.10 mm thick. All coated specimens were furnace oxidation tested at 1093° C. Nickel-aluminide coated IN 100 was also tested in the NASA Mach 1 burner rig at 1093° C. Oxidation testing of the slurry coatings showed the following:

1. Nickel-aluminide slurry-coated IN 100 was at least as oxidation resistant as commercially aluminized IN 100 in both furnace and burner rig testing at 1093° C. After 300 hr in either test the original aluminized slurry coating remained intact and was capable of providing further protection. Thermal fatigue cracks were observed in the burner rig testing of the commercial aluminide coating but not in the aluminized slurry coating.

2. Nickel-aluminide slurry-coated WI-52 compared well with commercially aluminized WI-52 in furnace tests at 1093° C. Approximately 0.028 mm of the original 0.088-mm nickel-aluminide phase remained in the slurry coating after 300 hr of testing, while no aluminide layer remained in the commercial aluminide after 200 hr of testing.

3. Iron-aluminide plus iron-chromium-aluminum slurry-coated IN 100 compared well with iron - 25 chromium - 4 aluminum - 1 yttrium clad IN 100 for 200 hr of furnace testing at 1093° C. After 300 hr, however, the original slurry coating had been converted by interdiffusion to a nickel-base material.

4. Iron-aluminide plus iron-chromium-aluminum slurry-coated WI-52 was the least protective of the four systems studied. Very little coating remained after 300 hr at 1093° C, and much oxide spalling was observed.

This study showed that oxidation-resistant coatings could be produced using slurry techniques and that the compositions of these coatings depended only slightly on the

substrate composition. In addition to this advantage, slurry techniques also offer the versatility of adding potentially beneficial alloying elements, oxides, or intermetallic compounds to the coating structure.

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National Aeronautics and Space Administration,  
Cleveland, Ohio, February 23, 1971,  
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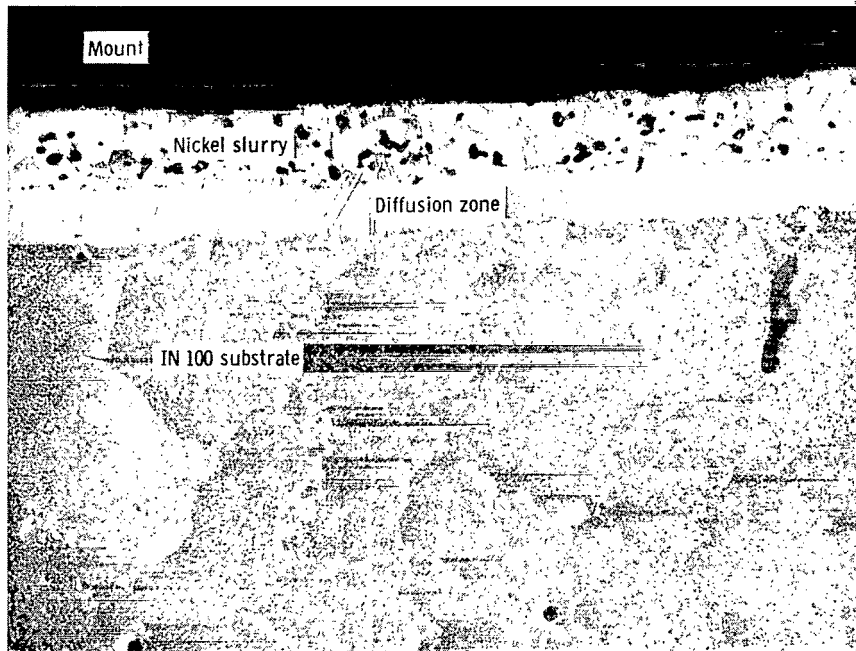
TABLE I. - SUMMARY OF X-RAY DIFFRACTION AND METALLOGRAPHY DATA

Coating system	As-sintered		As-aluminized				As-oxidized		
	Surface phases	Thickness, <sup>a</sup> mm		Surface phases	Thickness, <sup>a</sup> mm		Surface phases	Thickness, <sup>a</sup> mm	
		Coating	Diffusion zone		Coating	Diffusion zone		Aluminide coating	Diffusion zone
NiAl on IN 100	Ni <sup>b</sup>	0.040	0.040	NiAl	0.052	0.040	Al <sub>2</sub> O <sub>3</sub> NiAl (martensite)	0.068	0.060
NiAl on WI-52	Ni <sup>b</sup>	0.044	0.028	NiAl	0.088	0.024	Al <sub>2</sub> O <sub>3</sub> Spinel ( $a_o = 8.16 \times 10^{-10}$ m; 8.16 Å) Ni <sup>b</sup> or Ni <sub>3</sub> Al NiAl (martensite)	0.028	0.028
Fe-Cr-Al on IN 100	Fe-Cr <sup>b,c</sup>	0.076	-----	FeAl Fe-Cr-Al <sup>b,c</sup>	0.100	0.016	Al <sub>2</sub> O <sub>3</sub> Spinel ( $a_o = 8.22 \times 10^{-10}$ m; 8.22 Å) Ni <sup>b</sup> or Ni <sub>3</sub> Al NiAl or FeAl	0.040	0.152
Fe-Cr-Al on WI-52	Fe-Cr <sup>b,c</sup>	0.064	0.036	FeAl Fe-Cr-Al <sup>b,c</sup> Cr <sub>23</sub> C <sub>6</sub>	0.100	-----	Al <sub>2</sub> O <sub>3</sub> Spinel ( $a_o = 8.34 \times 10^{-10}$ m; 8.34 Å) Cr <sub>2</sub> O <sub>3</sub> Co	Consumed by oxidation	
NiAl on IN 100 burner bar	-----	-----	-----	-----	0.113	0.030	Al <sub>2</sub> O <sub>3</sub> Spinel ( $a_o = 8.05 \times 10^{-10}$ m; 8.05 Å) Ni <sup>b</sup> or Ni <sub>3</sub> Al	0.120	0.040

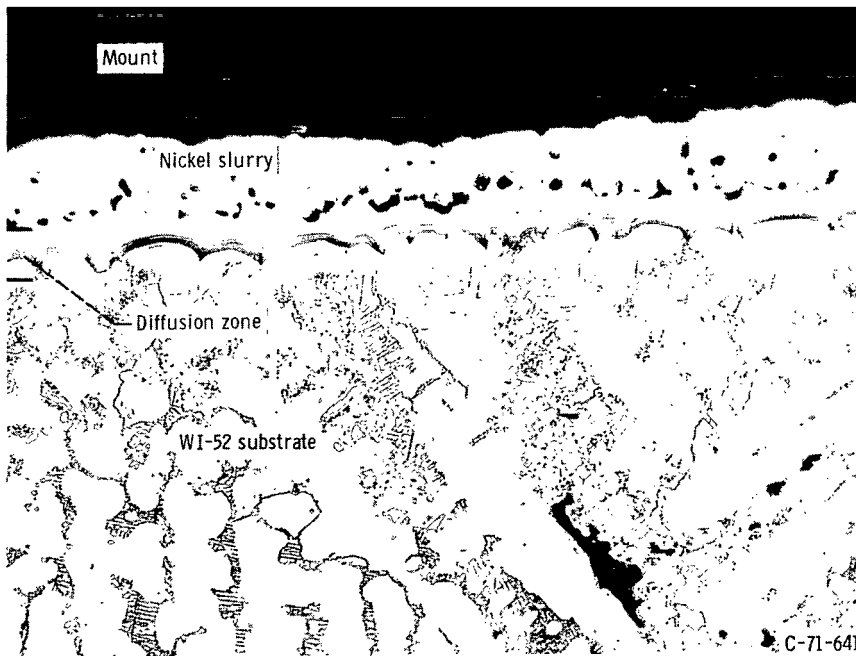
<sup>a</sup>Determined from photomicrographs.

<sup>b</sup>Solid solution.

<sup>c</sup>Observed bcc X-ray diffraction pattern applies for  $\alpha$ -phase Fe, Cr, Fe-Cr, and Fe-Cr-Al solid solutions.

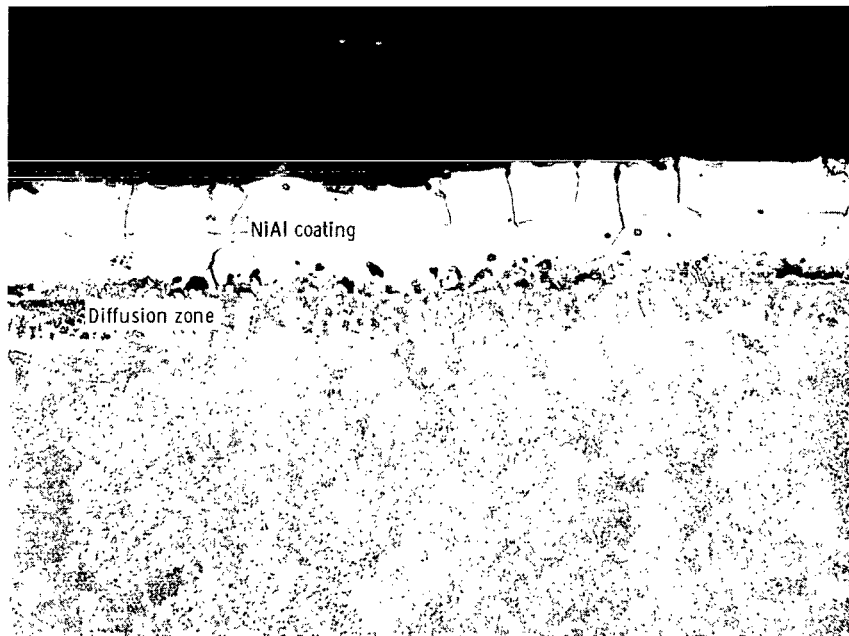


(a) IN 100.

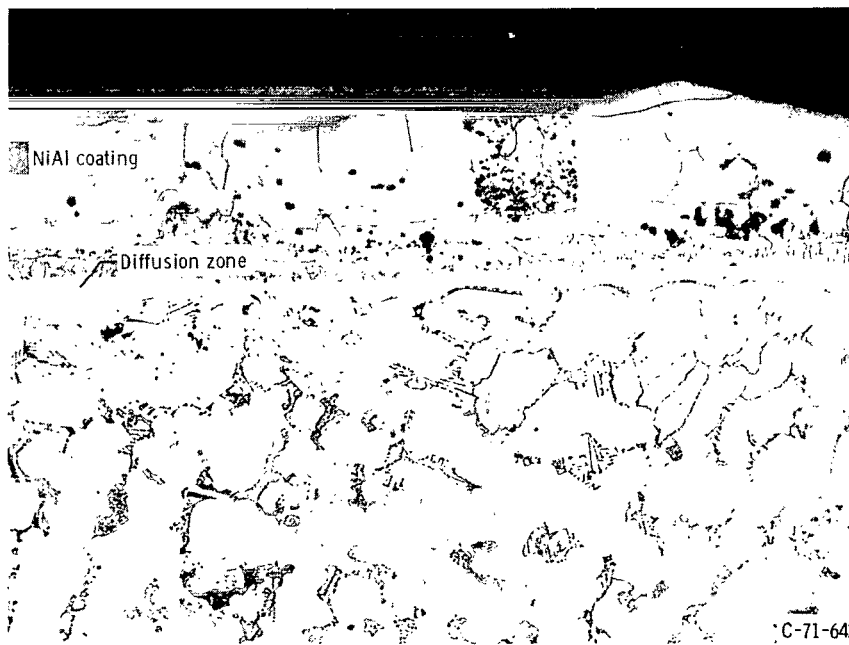


(b) WI-52.

Figure 1. - Nickel slurry coatings pressed and sintered on IN 100 and WI-52 before aluminizing. Etched; X250.



(a) IN 100.



(b) WI-52.

Figure 2. - Aluminized nickel slurry coatings on IN 100 and WI-52. Etched; X250.

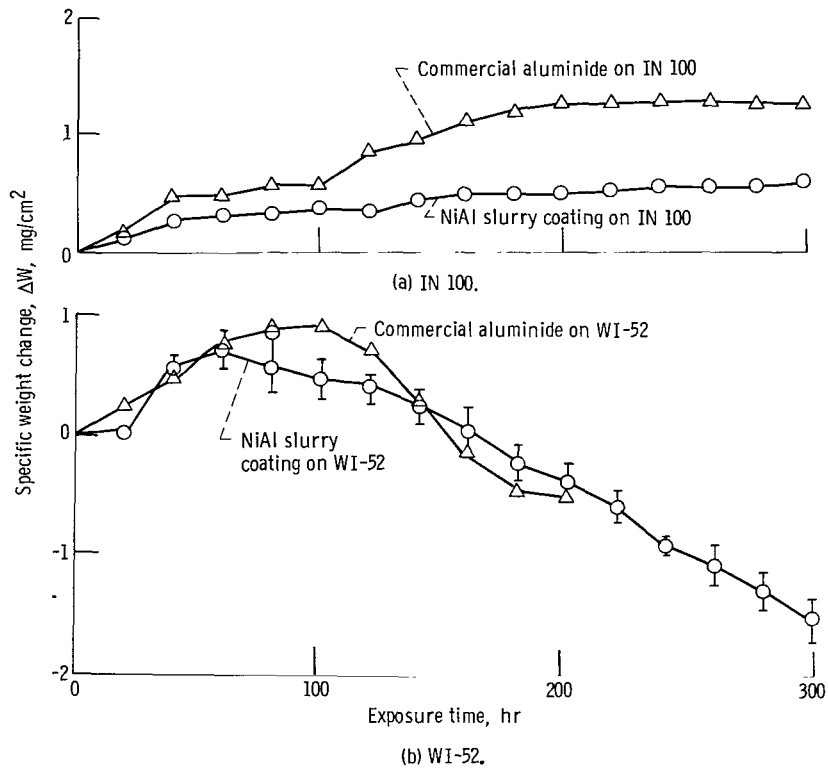
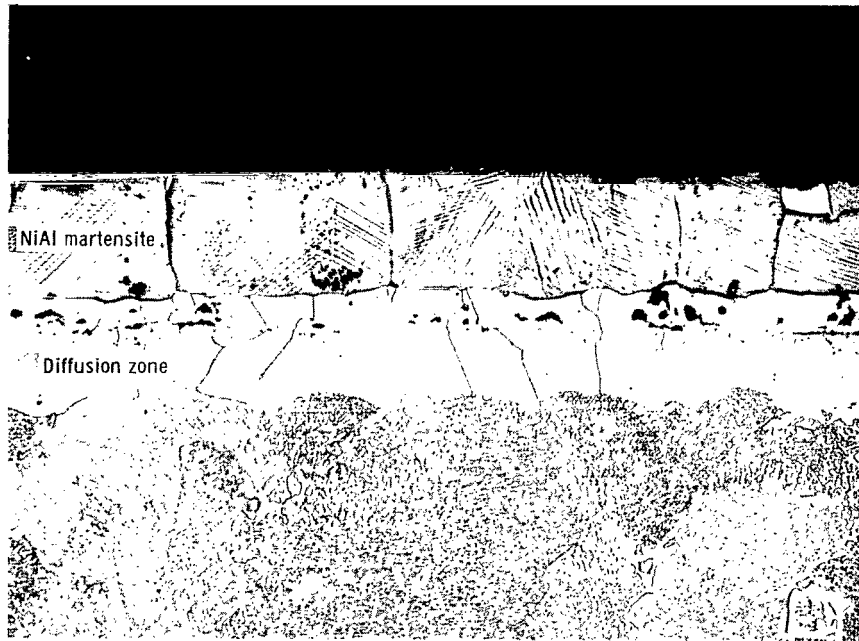
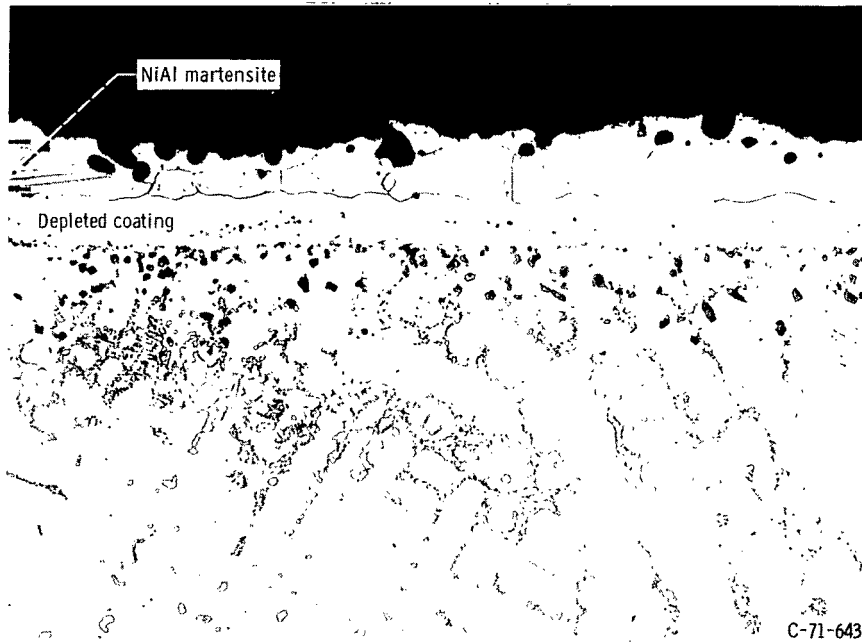


Figure 3. - Comparison of cyclic furnace oxidation behavior of NiAl slurry coating and commercial aluminide coating on IN 100 and WI-52. Tested at 1093° C in 20-hr cycles.



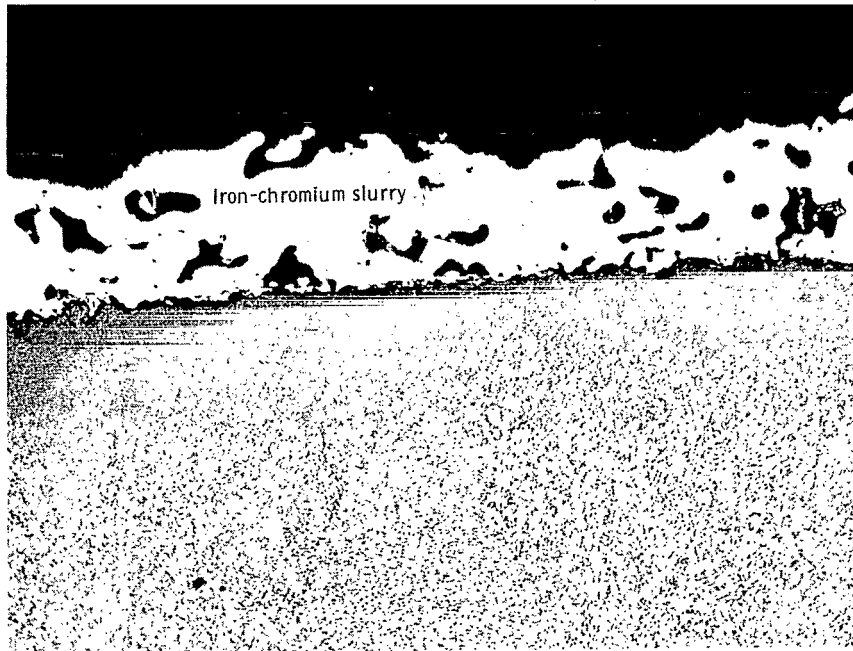


(a) IN 100.

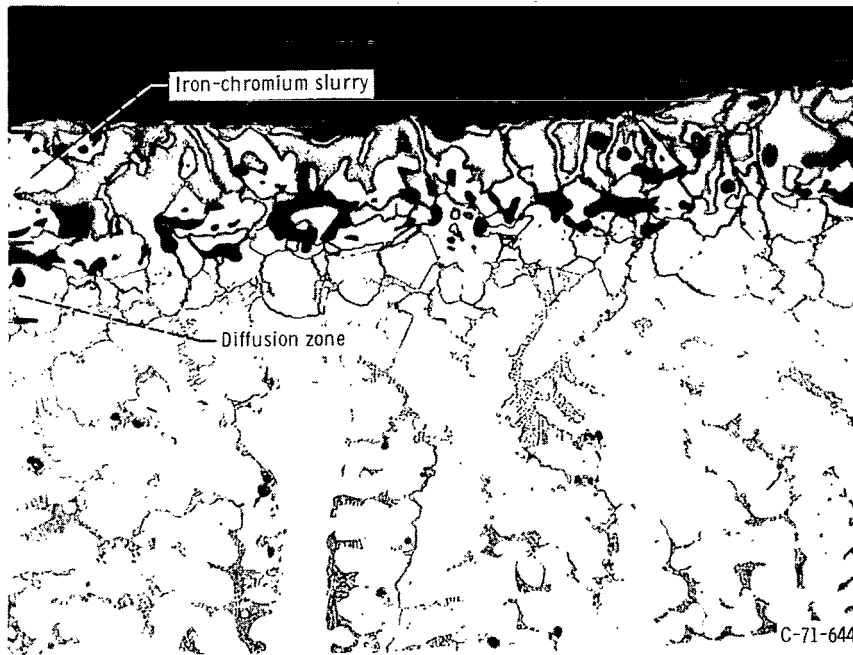


(b) WI-52.

Figure 4. - Oxidized NiAl slurry coatings on IN 100 and WI-52; tested at 1093° C for 300 hr in 20-hr cycles. Etched; X250.

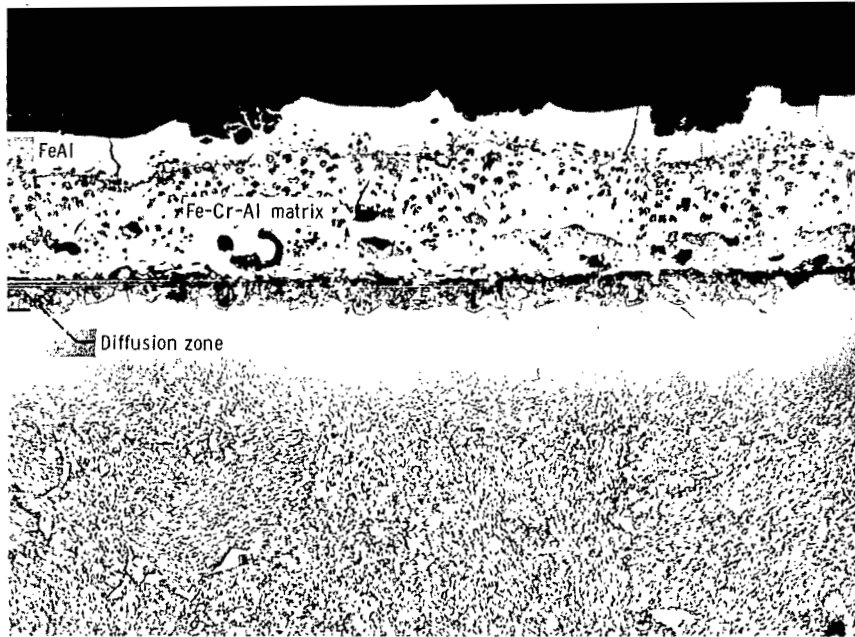


(a) IN 100.

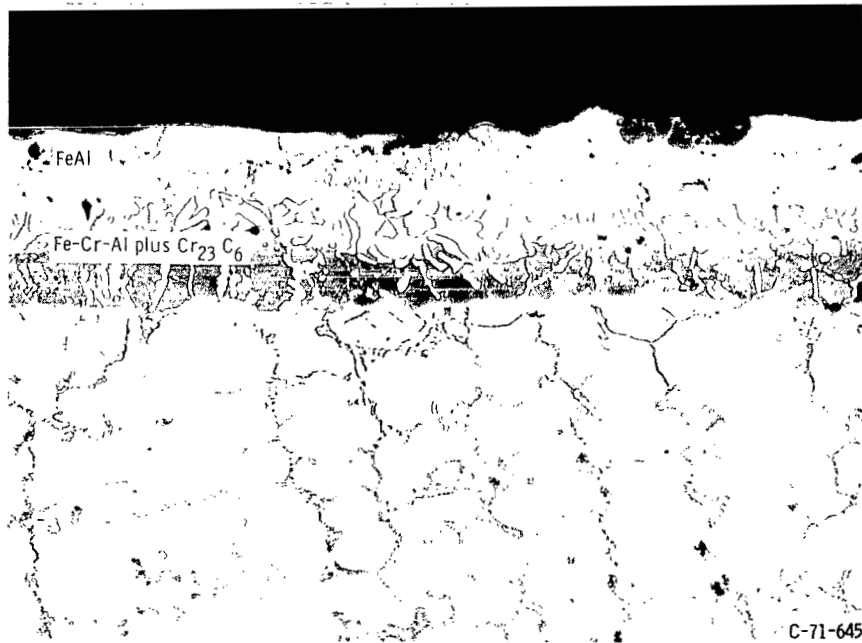


(b) WI-52.

Figure 5. - Iron - 30-percent-chromium powder mixture slurry, pressed and sintered on IN 100 and WI-52 before aluminizing. Etched; X250.



(a) IN 100.



(b) WI-52.

Figure 6. - Aluminized iron-chromium coatings on IN 100 and WI-52. Etched; X250.

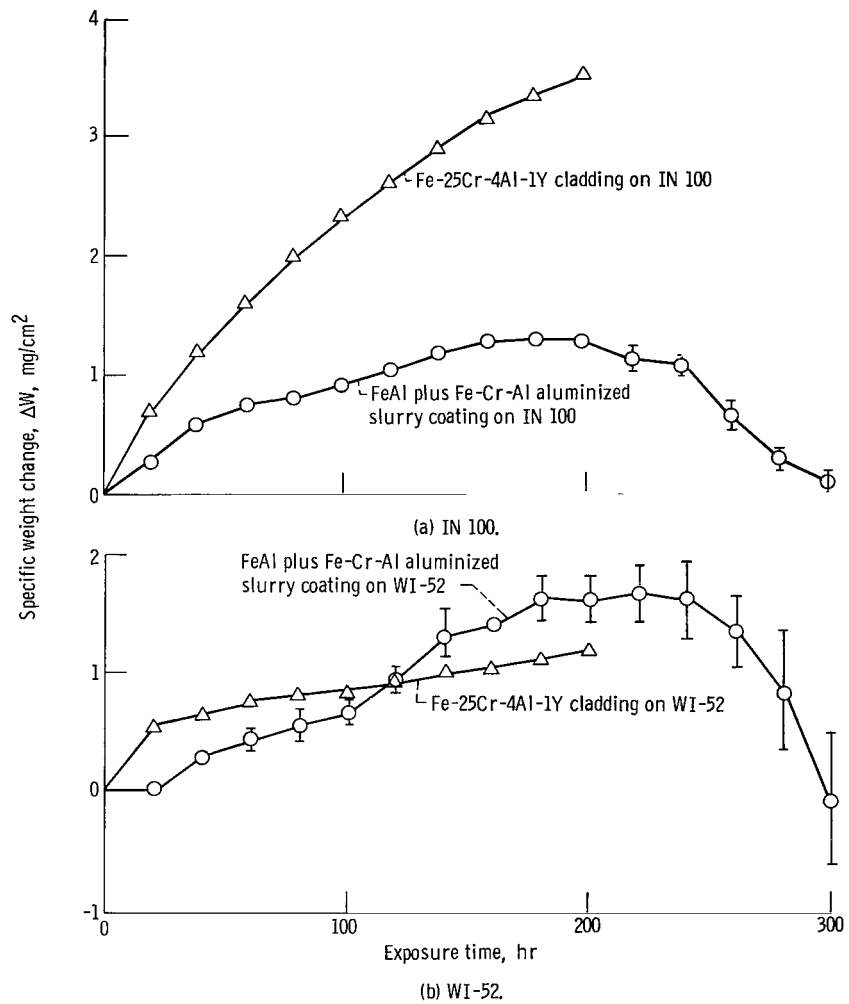
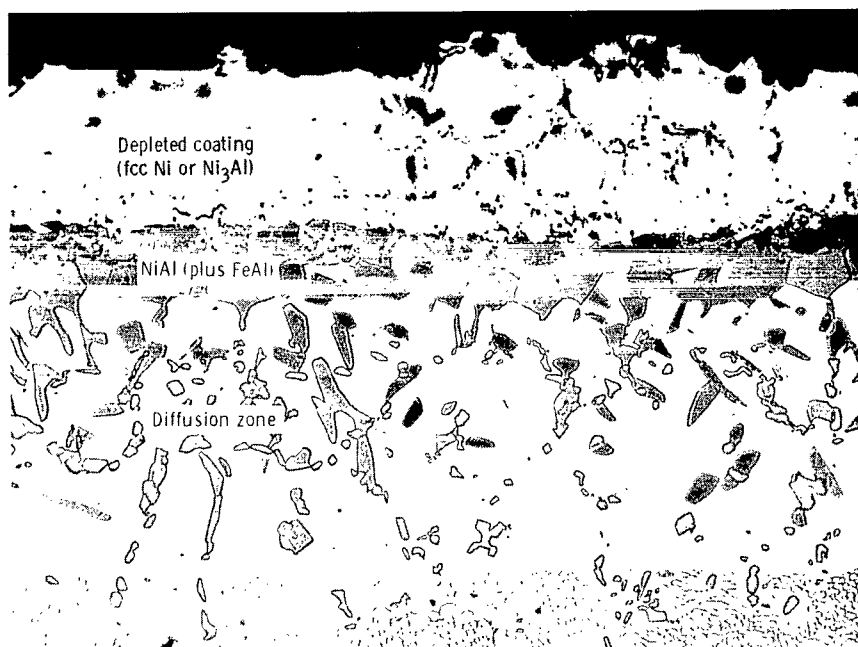
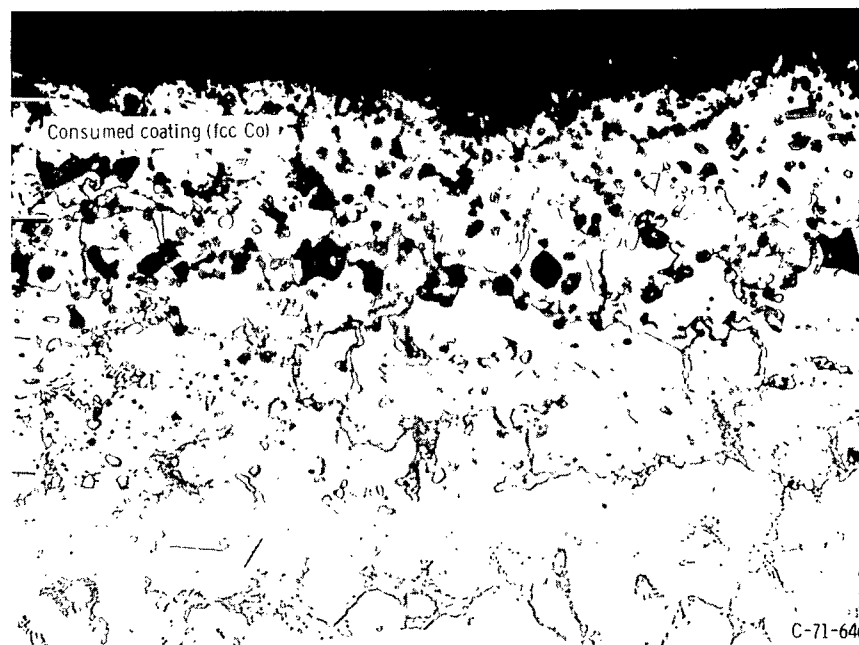


Figure 7. - Comparison of cyclic furnace oxidation behavior of FeAl plus Fe-Cr-Al aluminized slurry coating and Fe-25Cr-4Al-1Y cladding on IN 100 and WI-52. Tested at 1093° C in 20-hr cycles.



(a) IN 100.



(b) WI-52.

Figure 8. - Oxidized FeAl plus Fe-Cr-Al aluminized slurry coatings on IN 100 and WI-52; tested at 1093° C for 300 hr in 20-hr cycles. Etched; X250.

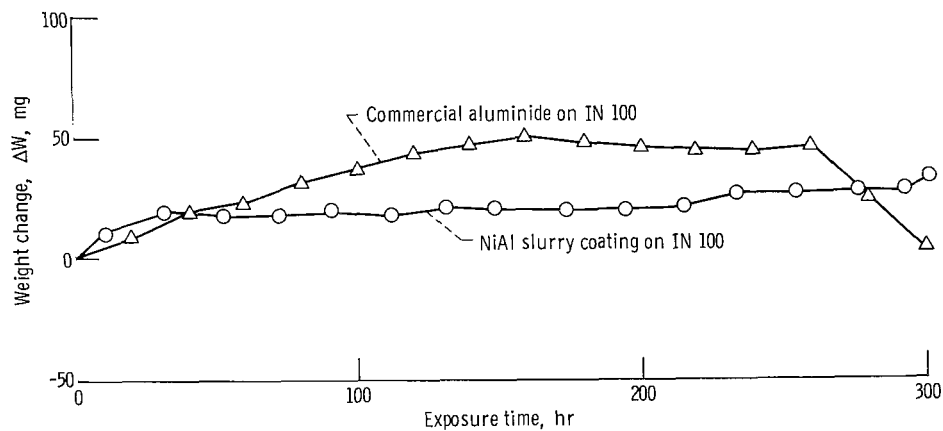
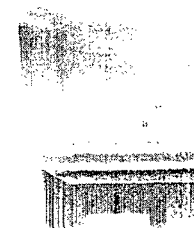


Figure 9. - Comparison of oxidation behavior in Mach 1 burner rig tests of NiAl aluminized slurry coating and commercial aluminide coating on IN 100. Tested at 1093° C in 1-hr cycles.



C-71-647

Figure 10. - NiAl slurry-coated IN 100 burner rig specimen tested at 1093° C for 300 hr in 1-hr cycles. Light areas at A<sub>1</sub> and A<sub>2</sub> identified as α-Al<sub>2</sub>O<sub>3</sub> plus spinel ( $a_0 = 8.05 \times 10^{-10}$  m; 8.05 Å); blister at B identified as NiO plus spinel ( $a_0 = 8.20 \times 10^{-10}$  m; 8.20 Å).

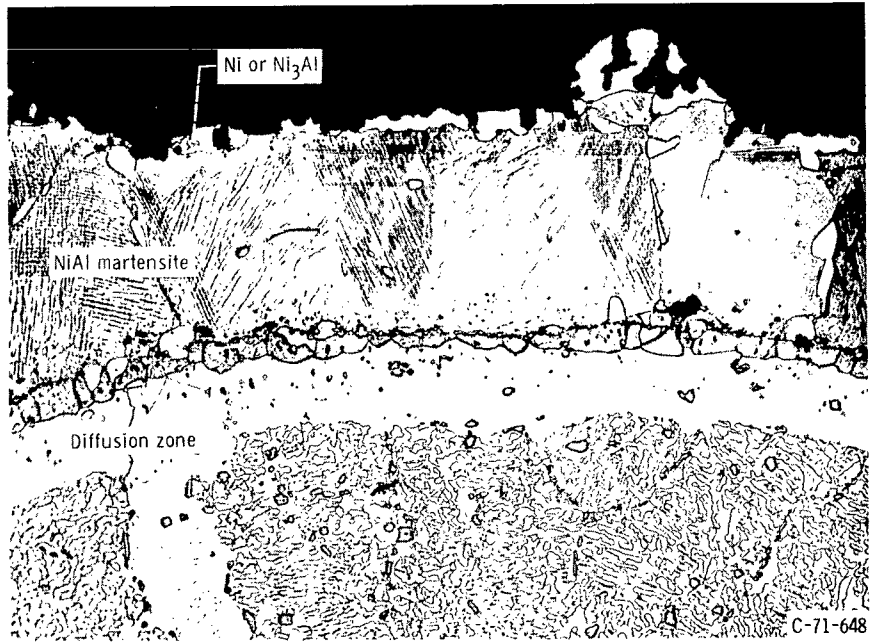


Figure 11. - NiAl slurry-coated burner rig specimen tested at (1093° C) for 300 hr in 1-hr cycles. Leading edge; etched; X250.

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