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AN EVALUATION OF THREE OXIDATION-RESISTANT ALLOY CLADDINGS FOR IN 100 AND WI 52 SUPERALLOYS

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16. Abstract

This study demonstrated the feasibility of protecting typical turbine alloys from cyclic furnace oxidation by cladding them with oxidation-resistant alloy foils. Foils of Ni-30Cr-1.4Si, Ni-20Cr-4Al-1.2Si, and Fe-25Cr-4Al-1Y were bonded to IN 100 (Ni base) and WI 52 (Co base). Cyclic oxidation exposure to 200 hours was conducted at 1900° and 2000° F (1310 and 1360 K). At 1900° F (1310 K), gravimetric, metallographic, and interdiffusion analyses showed all three claddings capable of providing excellent protection for both substrates. At 2000° F (1360 K), Ni-30Cr-1.4Si and Fe-25Cr-4Al-1Y provided excellent protection for WI 52, while Ni-20Cr-4Al-1.2Si afforded good protection for IN 100.

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SUMMARY

An investigation was made to evaluate the concept of protecting superalloys against high-temperature oxidation by cladding with more-oxidation-resistant alloys. Thin foils of three alloys were bonded to nickel-base (IN 100) and cobalt-base (WI 52) superalloy substrates. The three cladding alloys were Ni-30Cr-1.4Si, Ni-20Cr-4Al-1.2Si, and Fe-25Cr-4Al-1Y. The clad substrates were exposed to cyclic furnace oxidation at 1900° and 2000° F (1310 and 1360 K) for as long as 200 hours. Evaluations were based on gravimetric, metallographic, and cladding-superalloy interdiffusion analyses. The oxide scales were characterized by X-ray fluorescence and diffraction analyses.

The results of this investigation show that all three claddings were highly effective in providing oxidation protection for both superalloys for 200 hours at 1900° F (1310 K). For 200 hours at 2000° F (1360 K), the Ni-20Cr-4Al-1.2Si alloy on IN 100 afforded good oxidation protection. Both the Ni-30Cr-1.4Si and Fe-25Cr-4Al-1Y alloys provided excellent protection for WI 52.

Interdiffusion after oxidation at 2000° F (1360 K) was measured by electron microprobe analysis. In each system at least one element from the superalloy substrate had diffused completely through the cladding within 200 hours. Despite this, the protective ability of the three best cladding substrate systems (Ni-20Cr-1.2Si - IN 100, Ni-30Cr-1.4Si - WI 52, and Fe-25Cr-4Al-1Y - WI 52) was not seriously degraded. However, in the other systems, the oxidation resistance of the cladding alloys was decreased significantly as a result of interdiffusion. Such effects will require more detailed study in any potential application of these cladding alloys to superalloy substrates.

INTRODUCTION

Since the thermal efficiency of gas turbine engines increases as the turbine inlet temperature increases, there has been a continued effort to develop strong, creep-resistant, high-temperature turbine materials. Nickel- and cobalt-base superalloys have been developed which have adequate strength for long-time turbine blade and vane service to average temperatures of about 1800° and 1950° F (1250 and 1340 K), respectively. These superalloys, however, do not have adequate oxidation resistance above about 1700° F (1200 K) for the long times of interest in commercial engines (3000 hr and longer).

Currently, intermetallic aluminide diffusion coatings are being used to protect superalloy turbine components from oxidation attack (refs. 1 and 2). Based on unpublished test data, long-time service of these coatings appears limited to temperatures of about 1800° F (1250 K). The Ni-Cr, Ni-Cr-Al, and Fe-Cr-Al type alloys developed for furnace-heater applications have very good oxidation resistance even after long-time air exposures at temperatures between 2000° and 2300° F (1360 and 1530 K) (refs. 3 to 6). These materials are too weak, however, to resist the stresses generated in advanced turbine engines. Therefore, the present investigation was undertaken to examine the feasibility of combining the high strengths of superalloys with the good oxidation resistance of heater alloys. To do this, 5-mil (0.127-mm) thick foils of Ni-30Cr-1.4Si, Ni-20Cr-4Al-1.2Si, and Fe-25Cr-4Al-1Y (nominal compositions in weight percents) were clad to representative nickel-base (IN 100) turbine blade and cobalt-base (WI 52) turbine vane superalloys by gas-pressure bonding. The six cladding-superalloy systems were subjected to cyclic furnace oxidation at 1900° and 2000° F (1310 and 1360 K) for times as long as 200 hours. Evaluation of the protection ability of the cladding alloys was based primarily on gravimetric analyses, metallographic examination, and cladding-superalloy interdiffusion.

EXPERIMENTAL PROCEDURE

Materials and Specimen Preparation

The superalloys IN 100 (nickel base) and WI 52 (cobalt base) were chosen for this program. These superalloys were cast to size: nominally, 2 by 1 by 0.1 inch (50.8 by 25.4 by 2.54 mm) with 1/16-inch (1.6-mm) radii at the corners. Chemical analysis of each superalloy as determined by an independent laboratory is listed in table I.

The cladding alloys were Ni-30Cr-1.4Si (hereinafter designated Ni-Cr), Ni-20Cr-4Al-1.2Si (hereinafter designated Ni-Cr-Al), and Fe-25Cr-4Al-1Y (hereinafter designated Fe-Cr-Al-Y). All cladding alloys were procured from commercial suppliers in the forms of 0.005-inch (0.127-mm) foil and 0.10-inch (2.5-mm) thick sheet. Each cladding alloy, foil and sheet, was from the same heat. Chemical analyses, as determined by an independent laboratory, of the cladding alloys are given in table II. The alloy suppliers are also listed in table II.

In the present program, the 0.005-inch (0.127-mm) thick foils were used to protect the major surfaces of the superalloys. The 0.1-inch (2.5-mm) sheets were machined into picture frames to protect the edges of the superalloy substrates. They were also machined into test coupons for cladding and testing in order to provide baseline data in the evaluation of the clad superalloys.

The specimens, picture frames, and foil claddings were cleaned (see appendix A) and prepared by assembling the various cladding-substrate components shown in figure 1. The molybdenum diffusion barriers prevented contamination of the nickel-base claddings from the mild-steel cans. Flame-sprayed alumina, however, was deposited on the molybdenum barriers to prevent molybdenum contamination of the Fe-Cr-Al-Y cladding.

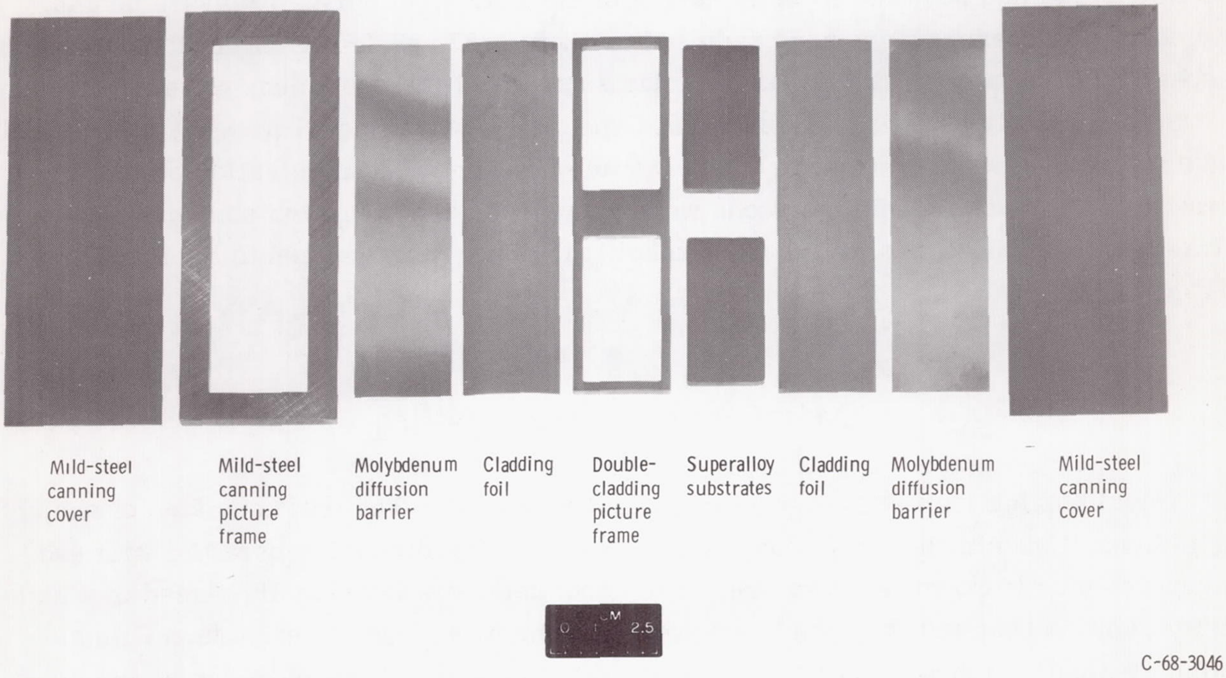


Figure 1. - Components for preparing clad superalloy specimens.

After assembly, the mild-steel canning covers and picture frames were joined by tungsten inert gas welding. The assembly was then vacuum outgassed at 500° F (530 K) for 1 hour and subsequently sealed by electron beam welding at a total pressure of 10^{-5} torr (1.3 mN/m^2). A cross section of an assembly ready for pressure bonding is shown in figure 2.

The sealed assembly was then pressure-bonded for 2 hours at $2000^{\circ}\pm20^{\circ}$ F (1360±11 K) and an isostatic helium pressure of 15 000 psi (1.03 MN/m^2).

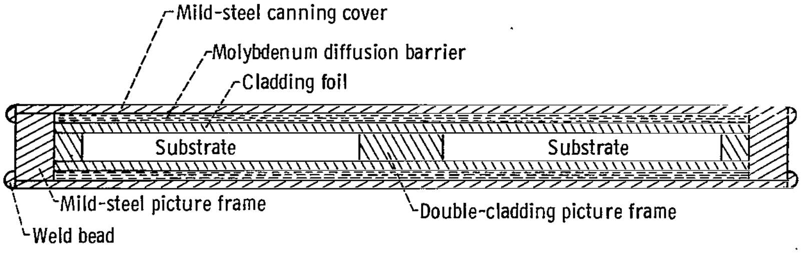


Figure 2. - Cross section of assembly for pressure bonding.

After pressure-bonding, the mild-steel can and molybdenum diffusion barriers were removed by acid leaching. The leached assembly, containing two clad superalloy specimens, was then machined. To ensure complete removal of iron and/or carbon contamination, approximately 1/16 inch (1.6 mm) of cladding was ground off the edges of the specimens that had been in direct contact with the mild steel during pressure bonding. The edges and corners were hand radiused to about 1/32 and 1/16 inch (0.8 and 1.6 mm), respectively. However, the major foil-clad surfaces of the specimens were not machined.

Before oxidation testing, the machined specimens were fine-silica-grit vapor-blasted and acid-cleaned again to remove any last traces of surface contamination or surface scratches. The specimen dimensions were measured; and then each specimen was ultrasonically cleaned in acetone and alcohol, air dried, and weighed to the nearest 0.1 milligram.

Cyclic Furnace Oxidation Testing

A cyclic oxidation test was used in this investigation. The test consisted of subjecting individual specimens for 20-hour exposures at the oxidizing temperature with subsequent cooling to room temperature. Each specimen was individually placed in a high-purity alumina boat and only one specimen at a time was placed in each 40-millimeter diameter alumina tube of a horizontal, eight-tube furnace. After each cycle, the specimens were examined for evidence of spalling, lightly brushed, and then weighed to the nearest 0.1 milligram. To accelerate testing, the tests were conducted at $1900^{\circ}\pm10^{\circ}$ F and $2000^{\circ}\pm10^{\circ}$ F (1310±5 K and 1360±5 K) for accumulated times as long as 200 hours at each temperature or until a weight loss of 3 milligrams per square centimeter, or greater, occurred.

Evaluations and Techniques

Weight-change data were obtained on the clad superalloys, the clad cladding alloys, and the superalloys in the unclad condition - the latter two conditions to provide baseline data for comparison with the clad superalloys. The data reported herein are the net weight changes (specimen weight only, not including spall) of the specimens after they were cooled to room temperature and lightly brushed.

Specimens were also metallographically examined (as etched) to determine the structural changes and the extent of interdiffusion, surface recession, and oxide penetration that occurred during cladding and oxidation. The etchants used for each specimen and the corresponding photomicrograph are listed in appendix B.

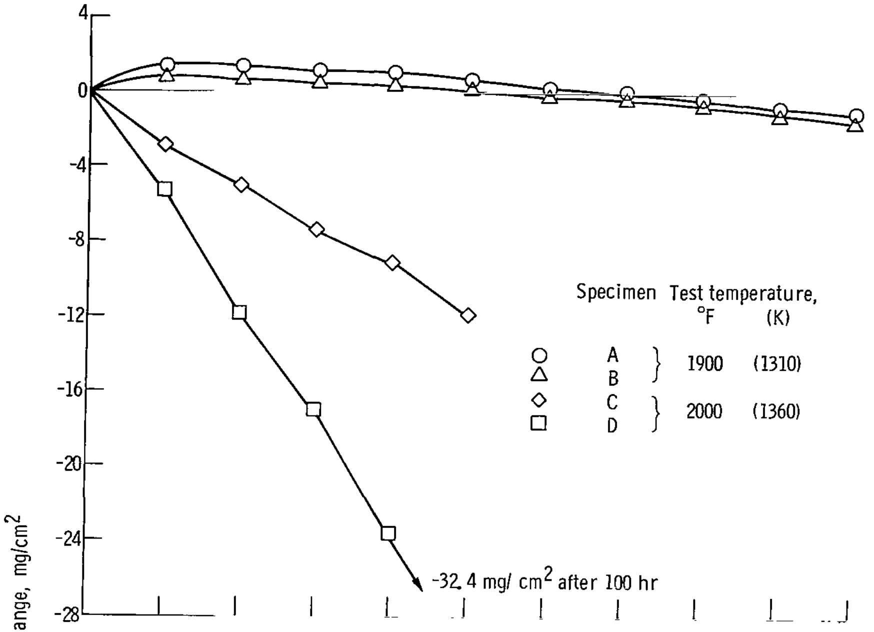
Several supplemental techniques were also used in the evaluations. Knoop microhardness profiles were measured on selected oxidized specimens to indicate if any systems were embrittled during oxidation. X-ray diffraction and X-ray fluorescence analyses were performed on oxidized specimens to examine the structures and compositions of the adhering surface scales. X-ray diffraction was made on either as-scraped or the in-situ scales, while X-ray fluorescence was always made on the in-situ scales.

Selected specimens were examined by electron microprobe analysis to determine more accurately the extent of interdiffusion of major and minor elements. Microprobe examination consisted of two scans, three elements per scan, across a metallographically prepared specimen. One of the major elements was chosen as a control in each scan. Because no variations were observed in minor element concentrations, the electron microprobe data presented herein are only for the major elements.

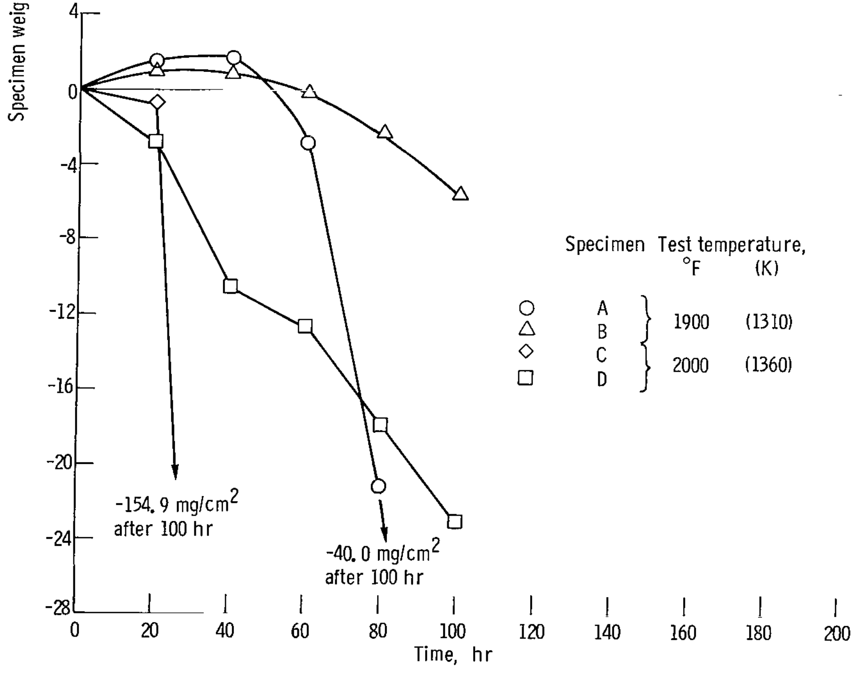
RESULTS AND DISCUSSION

Unclad Superalloys - Cyclic Oxidation

In order to get baseline data for comparison with the clad specimens, duplicate specimens of IN 100 and WI 52 were oxidized at both 1900° and 2000° F (1310 and 1360 K). As shown by the weight-change data for these specimens in figures 3(a) and (b), both alloys oxidize rapidly at 2000° F (specimens C and D). WI 52 also oxidizes at a high rate at 1900° F (1310 K). At this temperature IN 100 has relatively good oxidation resistance based on weight-change data (specimens A and B). Both alloys exhibited considerable oxide spalling, especially at 2000° F (1360 K). Thus, these findings confirm the need for high-temperature protection systems for both superalloys.



(a) Unclad IN 100.

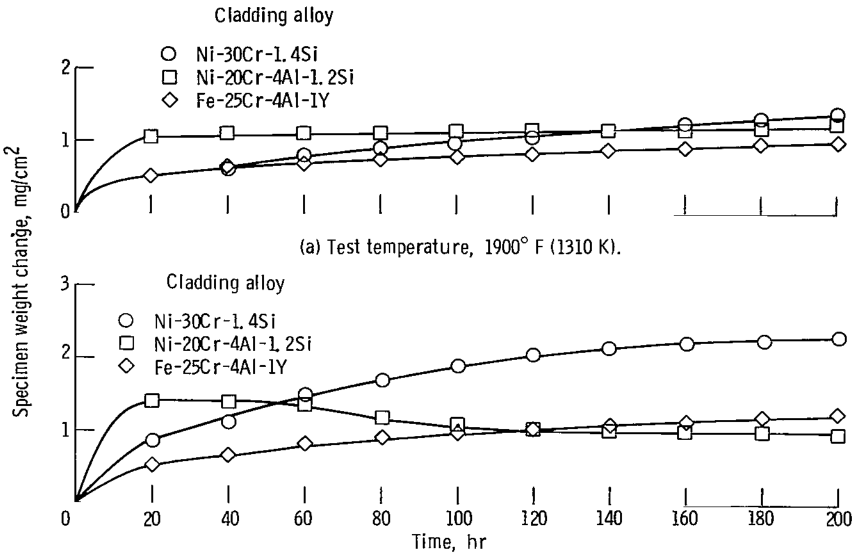


(b) Unclad WI 52.

Figure 3. - Cyclic oxidation of unclad IN 100 and unclad WI 52 at 1900° and 2000° F (1310 and 1360 K). Specimens were cooled to room temperature after each 20-hour exposure at the elevated temperatures.

Cladding Alloys

Cyclic oxidation. - The oxidation behavior of the three cladding foils bonded to substrates of the same composition as the clad (i.e., for example, 5-mil Ni-Cr foils bonded to 0.10-inch Ni-Cr substrates) are shown in figures 4(a) and (b) for tests at 1900° and 2000° F (1310 and 1360 K), respectively. All three alloys appear to have excellent oxidation resistance at 1900° F (1310 K). After 200 hours at this temperature, all alloys gained less than 1.4 milligrams per square centimeter. In addition, no spalling of the oxide scales was observed.

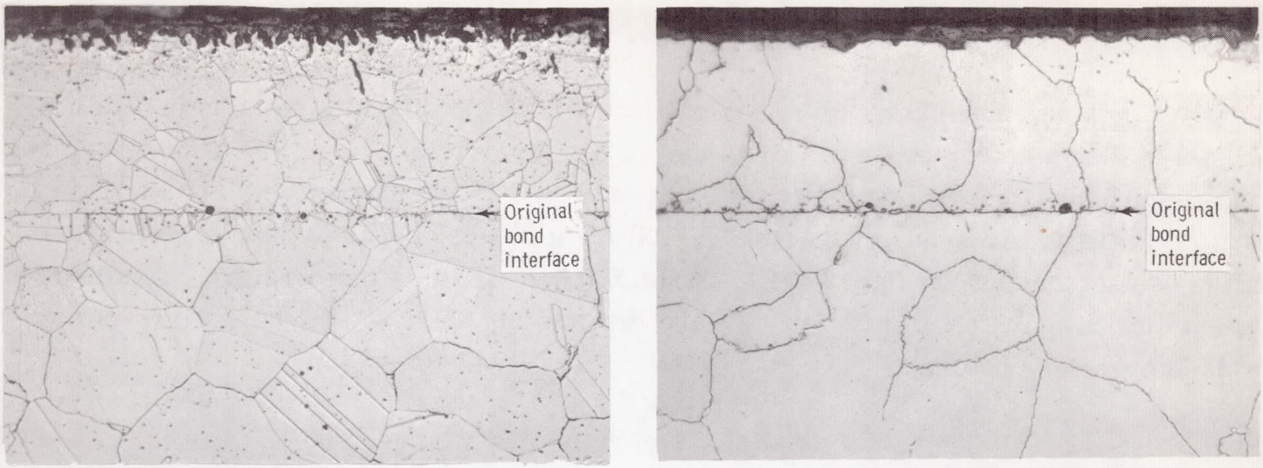


(b) Test temperature, 2000° F (1360 K).

Figure 4. - Cyclic oxidation of clad cladding-alloy substrates at 1900° and 2000° F (1310 and 1360 K). Specimens were cooled to room temperature after each 20-hour exposure at the elevated temperatures.

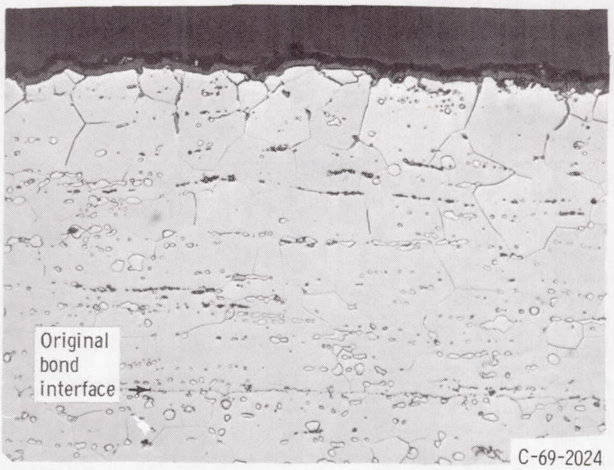
At 2000° F (1360 K), all alloys again appear to have excellent oxidation resistance. However, the Ni-Cr-Al alloy showed some very light spalling at this temperature.

Metallography. - The clad cladding-alloy specimens were metallographically examined after cyclic oxidation. Typical microstructures after oxidation for 200 hours at 2000° F (1360 K) are presented in figure 5 for later comparison with the clad superalloys. All three microstructures show a thin, continuous oxide scale on the surface, with very little oxide penetration along the grain boundaries. The microstructures of the clad cladding alloys oxidized at 1900° F (1310 K) for 200 hours were very similar to those presented in figure 5; the surface oxide thickness and oxide penetrations were the same or slightly less, however.



(a) Clad cladding-alloy substrate, Ni-30Cr-1.4Si.

(b) Clad cladding-alloy substrate, Ni-20Cr-4Al-1.2Si.



(c) Clad cladding-alloy substrate, Fe-25Cr-4Al-1Y.

Figure 5. - Microstructures of clad cladding-alloy substrates after ten 20-hour cycles (200 hr total) in air at 2000° F (1360 K). Etched; X250.

X-ray fluorescence (XRF) and diffraction (XRD). - In order to compare the effects of interdiffusion of the clads with the superalloy substrates on the scales that are formed and retained on the cladding-superalloy systems, XRF and XRD analyses were conducted on the scales retained on clad cladding alloys after oxidation at both test temperatures. The results of these analyses are listed in table III.

At either temperature, after 200 hours the Ni-Cr specimens showed increases in Cr, Si, Mo, and Mn in the scale, but only Cr_2O_3 was detected by XRD. The Ni-Cr-Al specimens showed high concentrations of only Ni and Cr. At 1900° F (1310 K) only a spinel was detected, but at 2000° F both NiO and a spinel were observed. The spinel is believed to be of the $NiCr_2O_4$ type. These data, however, indicate that the aluminum concentration is very low and that the expected Al_2O_3 scale is absent on the oxidized Ni-Cr-Al specimens. This may be related to the low aluminum content of the starting alloy. After testing at both temperatures, XRF showed that the scales on the oxidized Fe-Cr-Al-Y specimens contained high amounts of Fe, Cr, Al, and Y, while XRD showed Al_2O_3 and solid solutions of $(Cr, Fe)_2O_3$ as major phases.

Clad IN 100 Systems

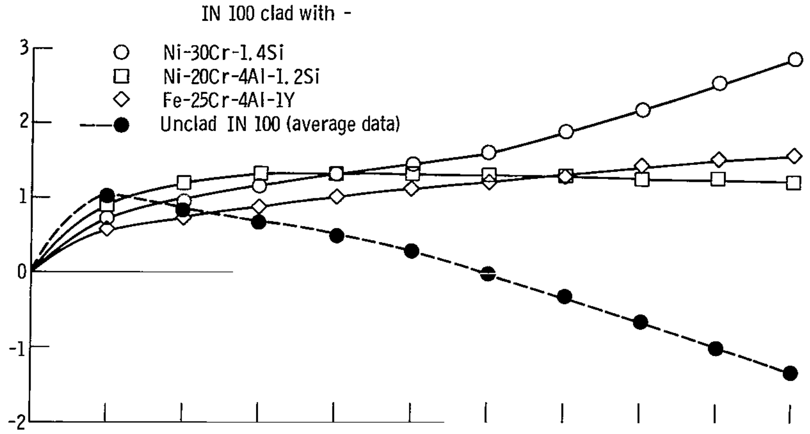
Cyclic oxidation. - Cyclic oxidation weight-change data for IN 100 clad with the three cladding alloys are compared with similar data for bare IN 100 at 1900° and 2000° F (1310 and 1360 K) in figures 6(a) and (b), respectively. In general, for a 200-hour period, the three claddings provided a modest increase in oxidation resistance over bare IN 100 when tested at 1900° F (1310 K). At 2000° F (1360 K), however, the claddings provided substantially better oxidation resistance than the bare IN 100.

Unlike the oxides that developed on the bare IN 100 specimens, the oxides formed on the Fe-Cr-Al-Y and Ni-Cr-Al claddings were adherent at 1900° F (1310 K). Although the Ni-Cr-clad IN 100 specimens continually gained weight through the 200-hour test, very light spalling occurred after 120 hours of testing at that temperature. At 2000° F (1360 K), spalling occurred during the tests of all three cladding - IN 100 systems. Again, spalling was most pronounced in the Ni-Cr-clad IN 100 specimens, and the erratic weight-change behavior is traceable to this spalling. Even with spalling, the oxidation of the Fe-Cr-Al-Y-clad specimens was sufficiently rapid to result in a net weight gain of 3.6 milligrams per square centimeter after 200 hours. In the Ni-Cr-Al - IN 100 system, apparent spalling ceased after 140 hours.

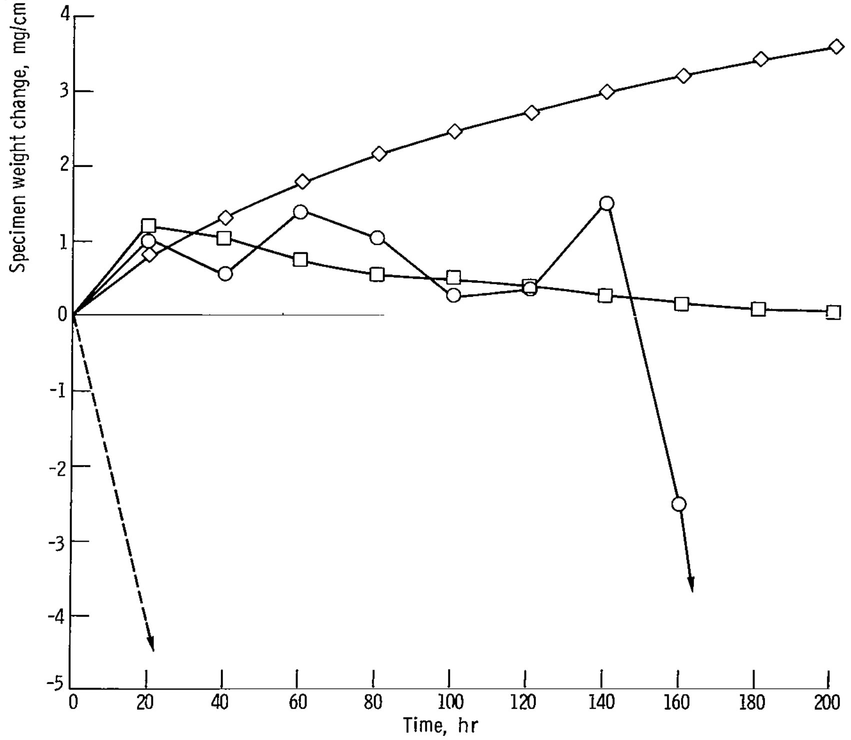
Table IV compares the 200-hour weight-change data (at both temperatures) of the clad IN 100, of the clad cladding-alloy specimens, and of bare IN 100. Differences in weight gain between clad IN 100 and clad claddings are believed to be traceable to the effect of interdiffusion between the cladding and the superalloy substrate. Table IV shows that the oxidation behavior of the Ni-Cr alloy is affected by the IN 100 at both temperatures, and that the Fe-Cr-Al-Y alloy has lessened oxidation resistance at 2000° F (1360 K) but is hardly affected at 1900° F (1310 K).

Metallography. - Photomicrographs of clad IN 100 specimens are shown in figure 7 after cyclic oxidation at both temperatures. Microhardness measurements, taken only on the specimens tested at 2000° F (1360 K), are listed adjacent to the appropriate photomicrographs at the approximate depths at which they were determined. Figure 7 also provides an as-bonded microstructure for each system for comparison purposes.

Figure 7(a) represents the microstructures of Ni-Cr-clad IN 100 after the various exposures. At both temperatures, gross surface scaling and oxide penetration had occurred, and the surfaces are very irregular. In addition, at the higher temperature, internal oxidation produced an oxide dispersion throughout the cladding. Also, at

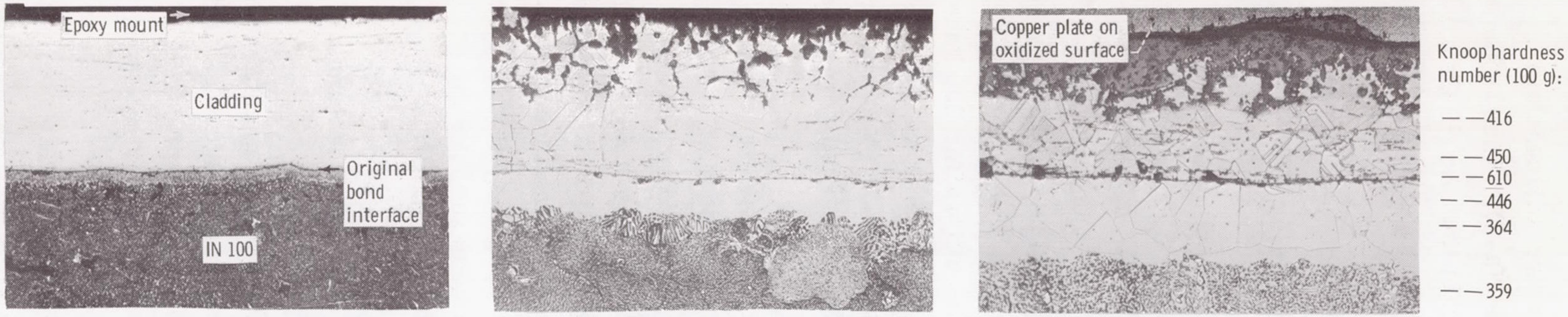


(a) Test temperature, 1900° F (1310 K).



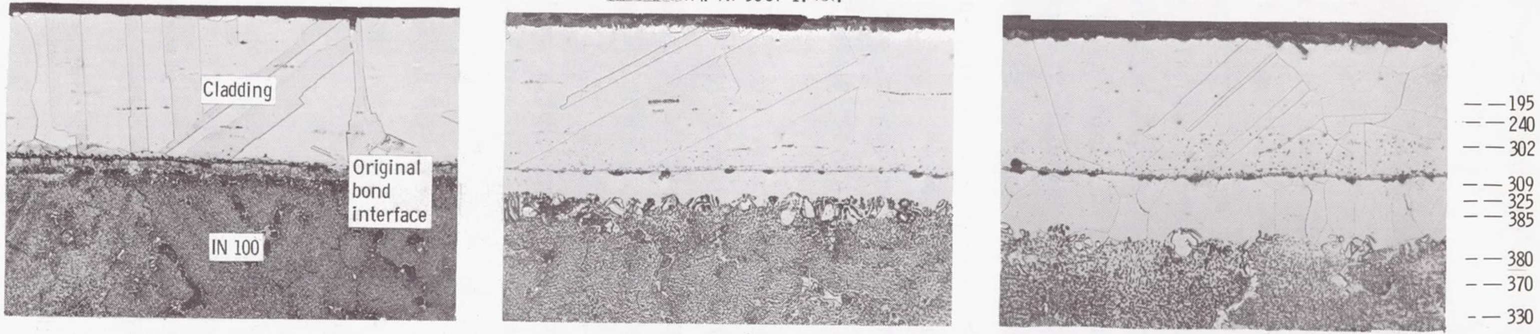
(b) Test temperature, 2000° F (1360 K).

Figure 6. - Cyclic oxidation of clad IN 100 at 1900° and 2000° F (1310 and 1360 K). Cladding thickness, 5 mils (0.127 mm). Specimens were cooled to room temperature after each 20-hour exposure at the elevated temperatures.



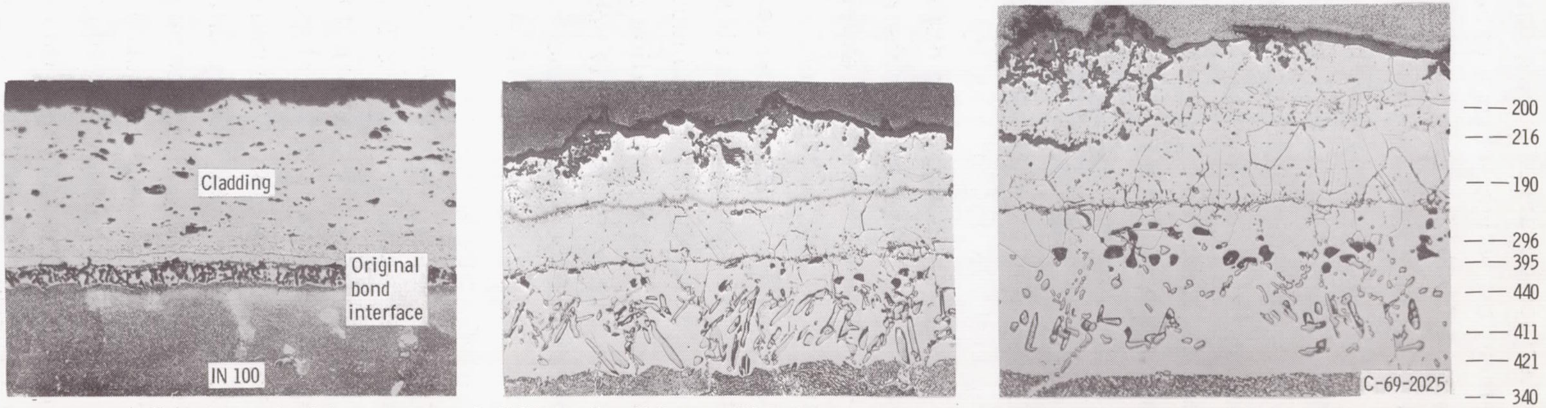
(a-1) As pressure bonded. (a-2) After cyclic oxidation at 1900° F (1310 K) for 200 hours. (a-3) After cyclic oxidation at 2000° F (1360 K) for 160 hours.

(a) Cladding, Ni-30Cr-1.4Si.



(b-1) As pressure bonded. (b-2) After cyclic oxidation at 1900° F (1310 K) for 200 hours. (b-3) After cyclic oxidation at 2000° F (1360 K) for 200 hours.

(b) Cladding, Ni-20Cr-4Al-1.2Si.



(c-1) As pressure bonded. (c-2) After cyclic oxidation at 1900° F (1310 K) for 200 hours. (c-3) After cyclic oxidation at 2000° F (1360 K) for 200 hours.

(c) Cladding, Fe-25Cr-4Al-1Y.

Figure 7. - Microstructures of clad IN 100 before and after cyclic oxidation. Etched; X250.

2000° F (1360 K), diffusion hardening of the substrate and the cladding adjacent to the bond interface occurred during oxidation, as can be seen in figure 7(a-3). These results supplement the weight-change data presented in figure 6 and indicate that the Ni-Cr alloy without a diffusion barrier would be a rather poor choice of a cladding for oxidation protection of IN 100, at least at these test temperatures and times.

In contrast, as shown in figure 7(b), Ni-Cr-Al appeared to provide good protection to IN 100. Very little surface oxidation and oxide penetration occurred at either temperature. Although some voids (probably Kirkendall voids) were formed along the bond interface during oxidation, the thermal cycling during testing caused no cladding-substrate separation. Also, no apparent evidence of internal oxidation was noted in any of the oxidized specimens. However, as is readily apparent in figure 7(b-3), in 200 hours at 2000° F (1360 K) diffusion of cladding constituents into the IN 100 and substrate depletion due to diffusion into the cladding occurred to a significant depth. This is reflected by the region of gamma - gamma prime destabilization in the IN 100. At 1900° F (1310 K), the zone in the IN 100 affected by diffusion is only about one-half as thick after 200 hours of testing. Even though some diffusional changes occurred in the substrate, the relatively unaffected cladding microstructures and the low weight gains indicate that the Ni-Cr-Al cladding can afford excellent furnace protection to IN 100 at both temperatures for times in excess of 200 hours.

Figure 7(c) shows that, based on metallography, Fe-Cr-Al-Y claddings on IN 100 are intermediate in protection ability between the poor Ni-Cr and the good Ni-Cr-Al claddings. A fairly thin, continuous, protective scale formed on the claddings at both temperatures. Oxide penetration of the claddings, in both tests, appeared to be somewhat localized. This was especially noticeable at 2000° F (1360 K) (fig. 7(c-3)). Also, internal oxidation of the cladding and substrate hardening occurred mainly at the higher temperature. Diffusion voids were evident after tests at both temperatures. Destabilization of the gamma - gamma prime structure occurred in IN 100 to a considerable depth at both temperatures. An unknown phase developed in the destabilized region. Therefore, because of the extensive interdiffusion and destabilization that occurs at these temperatures, the Fe-Cr-Al-Y cladding does not appear promising for IN 100 unless a suitable diffusion barrier could be developed.

As a further means of comparing the three claddings on IN 100, measurements were made of the amount of cladding thickness unaffected by penetration of surface oxides after oxidation. These data are presented in table V. Since the precise thickness of each as-bonded cladding was not measured before oxidation testing, the comparison is only qualitative. However, the Ni-Cr-Al cladding appears to be the least affected. After 200 hours of testing at 2000° F (1360 K), this cladding still has 4.3 mils (0.109 mm) of unaffected cladding remaining. Table V also contains measurements of the visually observable depths of diffusion-affected substrate. In all three systems, oxidation testing increased the depth of substrate affected by diffusion by from three to six times over that in the as-bonded condition. Although these increases are substantial, it is encouraging that the depth of the diffusion zone was less than 2 mils (0.051 mm) in 200 hours at 1900° F (1310 K) and was only 2.6 mils (0.066 mm) at 2000° F (1360 K) for the best system, Ni-Cr-Al - IN 100.

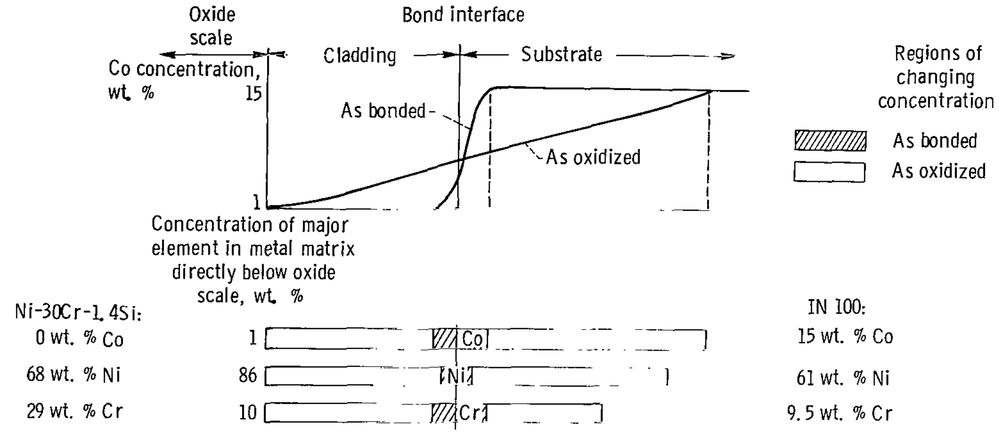
X-ray fluorescence (XRF) and diffraction (XRD). - XRF and XRD results obtained on the oxide scales adhering to the clad IN 100 specimens after testing at both temperatures are presented in table VI.

Comparison of the XRF data obtained on these specimens with those obtained on the clad cladding-alloy specimens (table III) at equivalent oxidation temperatures indicates that Ti and perhaps Mo diffuse readily from the IN 100 through the Ni-Cr cladding during oxidation at both temperatures. Except for these two elements, the radiation intensities of the other elements detected on the Ni-Cr-clad IN 100 specimens oxidized at 1900° F (1310 K) for 200 hours and at 2000° F (1360 K) for 60 hours were similar to those observed on the oxidized Ni-Cr-clad Ni-Cr specimens shown in table III. It could therefore be surmised that the scales would consist of the same oxides in these cases. Both systems did show the presence of Cr_2O_3 as a major phase.

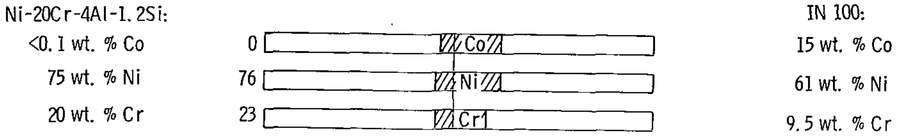
In the case of Ni-Cr-Al cladding on IN 100, XRF showed that Mo diffuses rapidly through the cladding from the substrate at both temperatures. After only 40 hours at 2000° F (1360 K), Al and Si concentrated in the scales, while after 200 hours Co and Ti had also concentrated in the oxide scales. Although some light spalling occurred at 2000° F (1360 K), these XRF data should generally reflect scale composition. The major elements detected at both temperatures were Ni and Cr. At 2000° F, Al was also a major scale element. Based on these intensities, the spinels detected by XRD probably are $NiCr_2O_4$ after 1900° F (1310 K) exposure and $Ni(Cr, Al)_2O_4$ after 2000° F (1360 K) exposure. Nickel oxide was also present as a major phase only after the higher-temperature exposure. These findings are similar to those found on the Ni-Cr-Al - Ni-Cr-Al systems presented in table III under the same conditions.

Comparison of the XRF data for the Fe-Cr-Al-Y - Fe-Cr-Al-Y and the Fe-Cr-Al-Y - IN 100 systems indicates that Ni, Co, Ti, and Mo diffuse readily through the cladding from IN 100 during oxidation testing at both temperatures. Furthermore, on the clad IN 100, especially at 2000° F (1360 K), the surface concentration of Al decreased during oxidation. XRD also showed no Al_2O_3 after oxidation at either temperature, in contrast to the major Al_2O_3 phase detected on the clad cladding-alloy specimen (see tables III and VI). Since spalling occurred during 2000° F (1360 K) oxidation, it is possible that Al_2O_3 may have spalled off the clad IN 100 and therefore was not detected. However, no spalling was observed to explain the loss of Al in the 1900° F tests. Diffusion into the substrate may be the major reason for these decreases. Since a spinel was detected in the oxide scale, some of the remaining Al may also be present in this spinel, probably of the (Ni, Co) (Cr, Al, Fe)$_2$O$_4$-type based on the XRF data.

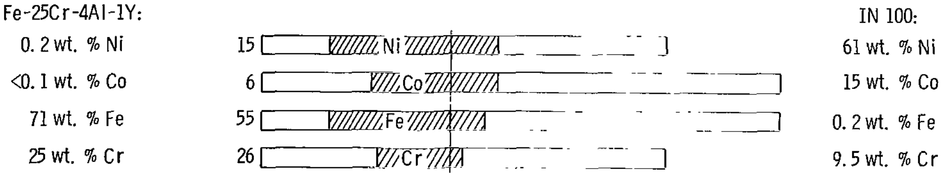
Electron microprobe (EMP). - EMP results on clad IN 100 specimens, as-bonded and as-oxidized at 2000° F (1360 K), are summarized in figure 8. The cross-hatched bars represent the extent of diffusion in the as-bonded condition. The clear bars represent the extent of diffusion after oxidation. The numbers at the left end of each clear bar represent the approximate concentration of each major element in the cladding just below the oxide scale. The results show that for the Fe-Cr-Al-Y - IN 100 system extensive interdiffusion occurred during bonding and oxidation; in the other two systems the extent of interdiffusion was considerable only after oxidation. In all three claddings,



(a) Before and after 160-hour oxidation.



(b) Before and after 200-hour oxidation.



(c) Before and after 200-hour oxidation.

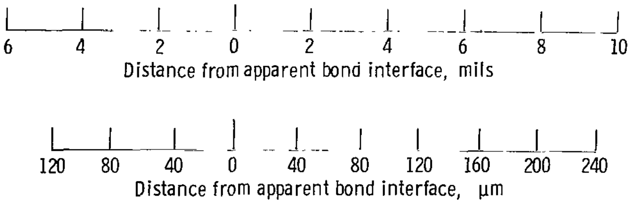


Figure 8. - Approximate extent of interdiffusion of major elements in clad IN 100 systems after bonding and oxidizing at 2000° F (1360 K).

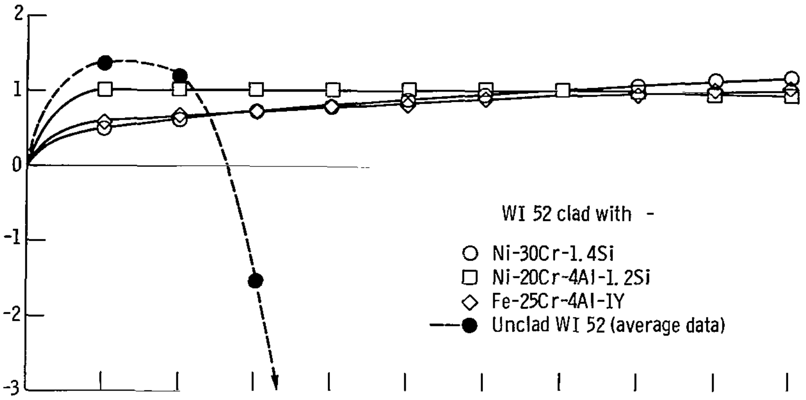
nominally 0.005-inch (0.127-mm) thick, during oxidation at least one substrate element had diffused throughout the thickness in 200 hours or less. This can be compared to superalloy element diffusion into the cladding of only 0.6, 0.5, and 3.1 mils (0.015, 0.013, and 0.079 mm) for Ni-Cr-clad, Ni-Cr-Al-clad, and Fe-Cr-Al-Y-clad IN 100 systems, respectively, after bonding. The zone in the IN 100, which either was depleted of elements by their diffusion into the cladding and/or which was enriched in elements from the cladding, varied in depth from 0.8 to 8.6 mils (0.020 and 0.218 mm) after oxidation. Compared to the data on the metallographically determined depths of diffusion-affected substrate in table V, the EMP data show diffusion depths about $1\frac{1}{2}$ to 2 times greater than those observable metallographically. Of the three systems, however, the Ni-Cr-Al cladding, which was the most protective, also affected the IN 100 the least; that is, only 5.2 mils (0.132 mm) of diffusion after 200-hour oxidation at 2000° F (1360 K).

Clad WI 52 Systems

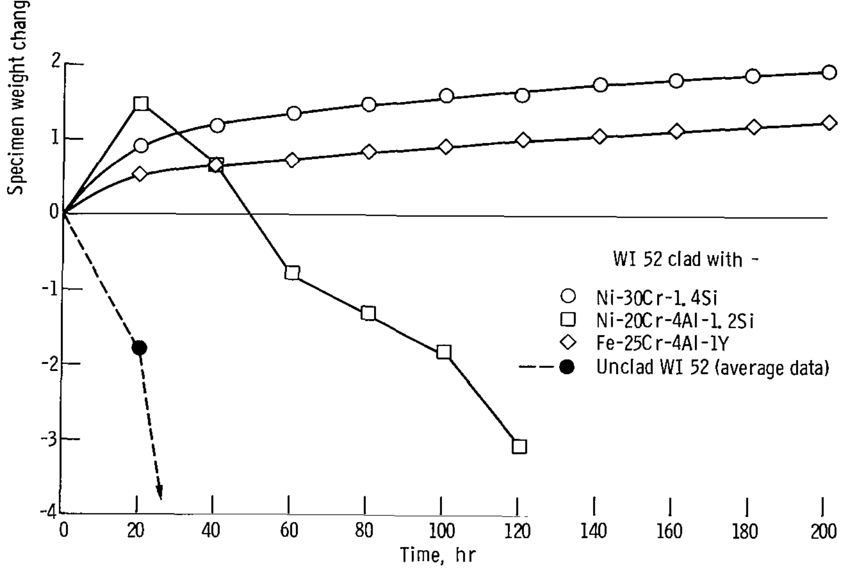
Cyclic oxidation. - Cyclic oxidation weight-change data for WI 52 clad with the three claddings are compared with similar data for bare WI 52 at both temperatures in figures 9(a) and (b). All three claddings markedly improved the poor oxidation resistance of WI 52 at 1900° F (1310 K). Figure 9(a) shows that the weight changes of the clad WI 52 specimens at this temperature were positive throughout the 200-hour tests, and no spalling was observed. The unclad WI 52, however, began to spall heavily after only 60 hours at this temperature.

The benefits of cladding are even more obvious at 2000° F (1360 K). Here two of the three cladding alloys provided 200-hour oxidation protection for WI 52. These were the Ni-Cr and the Fe-Cr-Al-Y claddings. They showed only modest weight gains and no spalling and, thus, appear to have potential for longer time use. The Ni-Cr-Al cladding, however, began spalling heavily after only 20 hours and lost 3.1 milligrams per square centimeter after 120 hours.

Table VII compares the 200-hour oxidation weight changes for these systems with the results of testing the clad cladding alloys and unclad WI 52 at both temperatures. At 1900° F (1310 K), the WI 52 has very little or no effect on the oxidation behavior of any of the three claddings. At 2000° F, WI 52 has a detrimental effect on the oxidation resistance of the Ni-Cr-Al.



(a) Test temperature, 1900° F (1310 K).

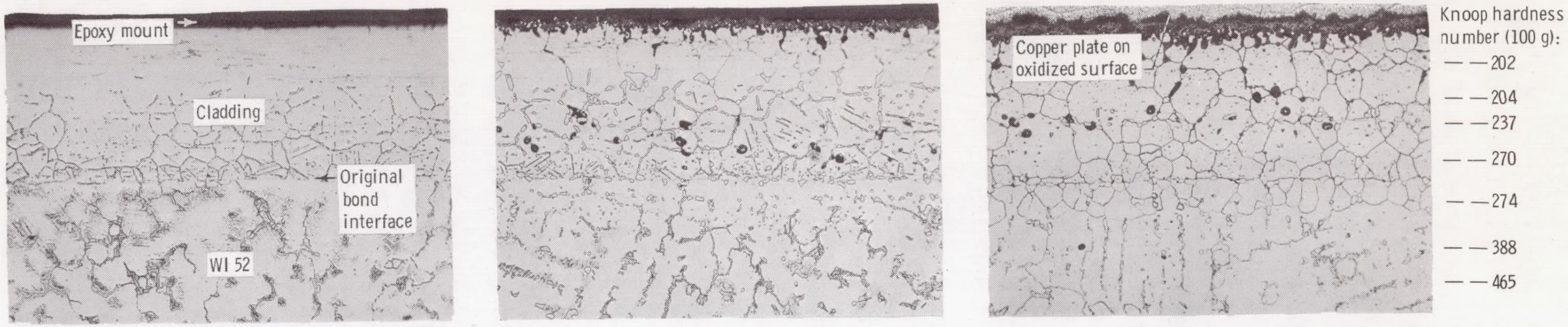


(b) Test temperature, 2000° F (1360 K).

Figure 9. - Cyclic oxidation of clad WI 52 at 1900° and 2000° F (1310 and 1360 K). Cladding thickness, 5 mils (0.127 mm). Specimens were cooled to room temperature after each 20-hour exposure at the elevated temperatures.

Metallography. - Figure 10 shows the microstructures of the clad WI 52 systems before and after oxidation testing at both temperatures.

As shown in figure 10(a), the Ni-Cr cladding is protective of WI 52 at both test temperatures. Little surface oxidation and oxide penetration occurred in either case, but some diffusion voids were observed at both temperatures. Internal oxidation was limited to the higher test temperature. The microstructures indicate that outward diffusion from the WI 52 into the clad was rapid. During the 2-hour bonding treatment at 2000° F (1360 K), diffusion extended from the substrate along the cladding grain boundaries into about two-thirds of the 0.005-inch (0.127-mm) thick cladding. Electron microprobe analysis of the grain boundary phase showed high chromium and low cobalt.

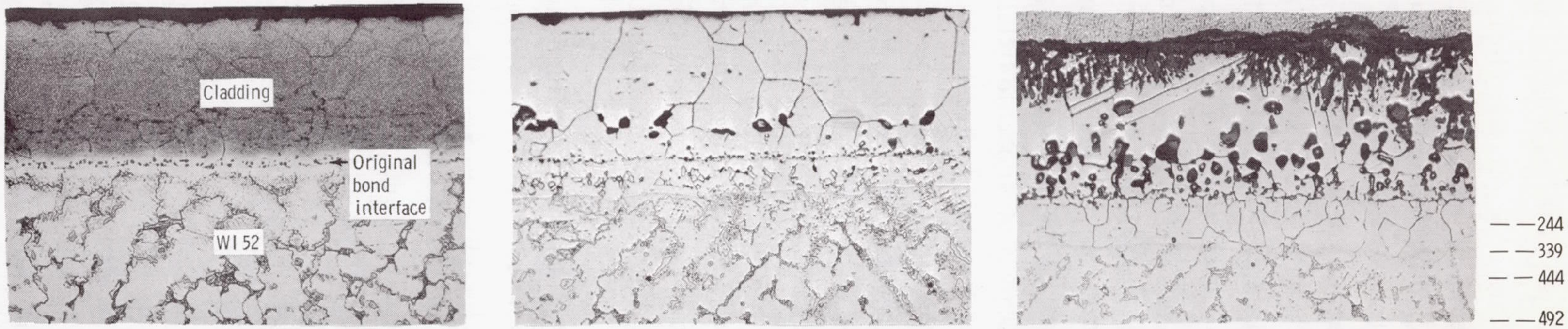


(a-1) As pressure bonded.

(a-2) After cyclic oxidation at 1900° F (1310 K) for 200 hours.

(a-3) After cyclic oxidation at 2000° F (1360 K) for 200 hours.

(a) Cladding, Ni-30Cr-1.4Si.

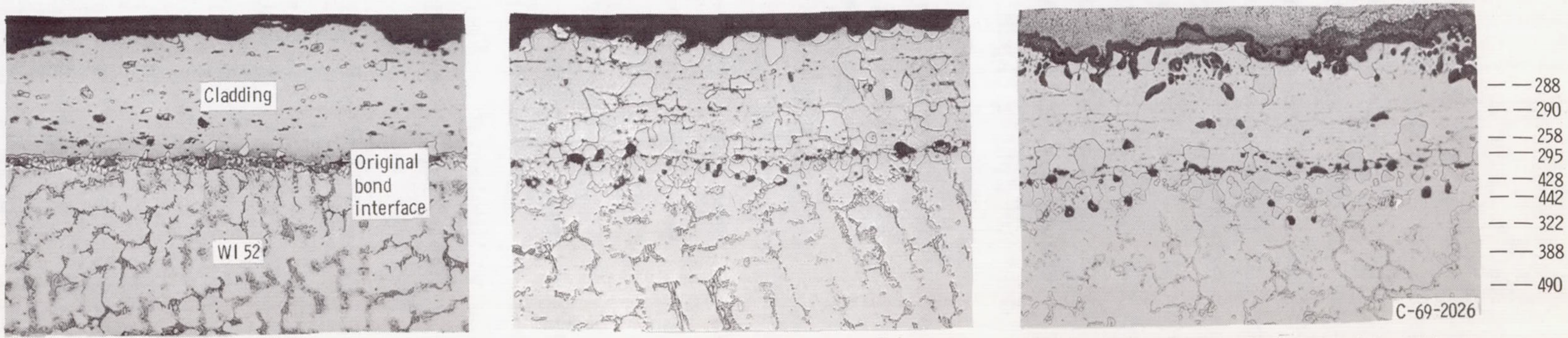


(b-1) As pressure bonded.

(b-2) After cyclic oxidation at 1900° F (1310 K) for 200 hours.

(b-3) After cyclic oxidation at 2000° F (1360 K) for 120 hours.

(b) Cladding, Ni-20Cr-4Al-1.2Si.



(c-1) As pressure bonded.

(c-2) After cyclic oxidation at 1900° F (1310 K) for 200 hours.

(c-3) After cyclic oxidation at 2000° F (1360 K) for 200 hours.

(c) Cladding, Fe-25Cr-4Al-1Y.

Figure 10. - Microstructures of clad WI 52 before and after cyclic oxidation. Etched; X250.

Based on this analysis and on the phase morphology, this phase is probably a chromium carbide. The carbide apparently formed by carbon diffusion from WI 52. It agglomerated during oxidation at the lower temperature but either went into solution or was oxidized at 2000° F (1360 K). Figure 10(a-3) shows no significant hardening occurring in this system during oxidation. Thus, the Ni-Cr cladding offers excellent oxidation protection for WI 52 for at least 200 hours at both temperatures.

Figure 10(b-3) shows that the surface of the Ni-Cr-Al - WI 52 system had a thin scale with no apparent oxide penetration after 200 hours at 1900° F (1310 K). A similar microstructure was observed after 40 hours at 2000° F (1360 K). After 120 hours of oxidation testing at 2000° F (1360 K), however, severe oxide penetration was evident (fig. 10(b-3)), and an increased number of diffusion voids and internal oxides were also observed. Because of the voids and internal oxides, no microhardness measurements were made on the cladding. Since voids occurred at both temperatures and since the spalling of the oxide scale was very heavy at the higher temperature, Ni-20Cr-4Al-1.2Si cladding does not appear capable of providing adequate oxidation protection for WI 52 at these temperatures and times without a suitable diffusion barrier.

As shown in figure 10(c), however, Fe-Cr-Al-Y does appear to provide good oxidation protection for WI 52 at both temperatures. On all specimens, the surface is rough, due to contact with the flame-sprayed Al_2O_3 diffusion barrier employed during cladding. Figure 10(c-3) shows that oxide penetration did not occur during the 200-hour oxidation test at 1900° F (1310 K). Some oxide penetration, however, is evident in the specimen tested at the higher temperature. In addition, some hardening of the region on the substrate side of the bond interface occurred during the 200-hour exposure at 2000° F (1360K). At both temperatures, a rather prevalent second phase (not the YFe_9 phase) in figure 10(c-1) developed in the cladding and also below the original bond interface. This second phase was not present in the clad cladding-alloy specimens after oxidation (see fig. 5(c)), and therefore, must have resulted from interdiffusion of the cladding and the substrate.

Table VIII lists the approximate thickness of claddings unaffected by penetration of surface oxides after cyclic oxidation of clad WI 52 specimens. The data indicate that all three claddings afford good oxidation protection for WI 52 for 200 hours at 1900° F (1310 K). At least 4.0 mils (0.101 mm) of the three claddings were unaffected by oxide penetration.

Some metallographic measurements on the depth of substrate affected by diffusion from and/or into the claddings are also listed in table VIII. Oxidation testing at 1900° F (1310 K) for 200 hours increased the diffusion depth only from 0.3 to 0.5 mils (0.008 to 0.013 mm) in the Ni-Cr - WI 52 system. Furthermore, less than 2 mils (0.051 mm) of substrate was affected by interdiffusion in all three systems after 200 hours at 1900° F (1310 K) and in the Ni-Cr - WI 52 and Fe-25Cr-4Al-1Y - WI 52 systems after 200 hours at 2000° F (1360K). The depth of diffusion-affected substrate in the Fe-Cr-Al-Y - WI 52 system after 200 hours at 1900° and 2000° F (1310 and 1360 K) increased only from 1.5 to 1.6 mils (0.038 to 0.041 mm). This small increase indicates less temperature sensitivity than shown by the Ni-Cr - WI 52 system (0.5 to 1.4 mils (0.013 to 0.036 mm)).

X-ray fluorescence (XRF) and diffraction (XRD). - Table IX lists the XRF and XRD results obtained on the scales retained on clad WI 52 after cyclic oxidation at both temperatures.

The XRF data for the oxidized Ni-Cr-clad WI 52 show that Cb was higher and Si lower at both temperatures, as compared to the oxidized Ni-Cr-clad Ni-Cr specimens (table III). After 200 hours at 2000° F (1360 K), Co and Al had higher and lower concentrations, respectively, in the oxide scales on the clad superalloy than on the clad cladding alloy. Since the XRF data show that both Ni and Cr intensities were high on the oxidized clad WI 52 specimens, the spinel phase detected by XRD on these specimens is probably $NiCr_2O_4$. The spinel is only a minor phase after 60 hours at 2000° F (1360 K); after 200 hours at 2000° F (1360 K), no spinel was detected. The XRD results also show that Cr_2O_3 was a major constituent of the scales on both the Ni-Cr - WI 52 and the Ni-Cr - Ni-Cr specimens.

In the Ni-Cr-Al - WI 52 system, the XRF data indicate that Al, Co, and Cb concentrated in the scales at both temperatures and Si concentrated at the higher temperature. The XRF data also show high radiation intensities for Ni and Cr on the two oxidized Ni-Cr-clad WI 52 specimens and for Al on the specimen tested for 40 hours at 2000° F (1360 K). Based on the intensity data, the spinels detected by XRD are thought to be predominantly $NiCr_2O_4$ at 1900° F (1310 K) and $NiAl_2O_4$ after 40 hours at 2000° F (1360 K). The XRD results, however, show that while Cr_2O_3 was detected as a minor constituent on the clad WI 52 specimens, it was not detected in the scales of the Ni-Cr-Al-clad Ni-Cr-Al specimens at either temperature. Heavy spallation after 120 hours at 2000° F (1360 K) makes scale composition comparison fruitless.

In the Fe-Cr-Al-Y - WI 52 system, the XRF results, when compared to the Fe-Cr-Al-Y-clad Fe-Cr-Al-Y specimens in table III, show that Co and W diffuse readily through the cladding to the scale from the WI 52 during oxidation at both temperatures. The comparison also suggests that Mn diffuses readily through the cladding at the higher temperature. The XRF data show that, after oxidation, high radiation intensities were obtained for the same elements (Cr, Al, and Fe) on both clad cladding-alloy and clad WI 52 specimens. The XRD data show that the same phases, Al_2O_3 and $(Cr, Fe)_2O_3$, were present on these specimens.

Electron microprobe (EMP). - A summary of the EMP concentration-penetration data for clad WI 52 specimens before and after oxidation at 2000° F (1360 K) is presented in figure 11. The data demonstrate that considerable interdiffusion occurred during oxidation in all three systems. The 5-mil (0.127-mm) thick claddings were completely affected by interdiffusion after oxidation. Diffusion affected the substrates to depths of

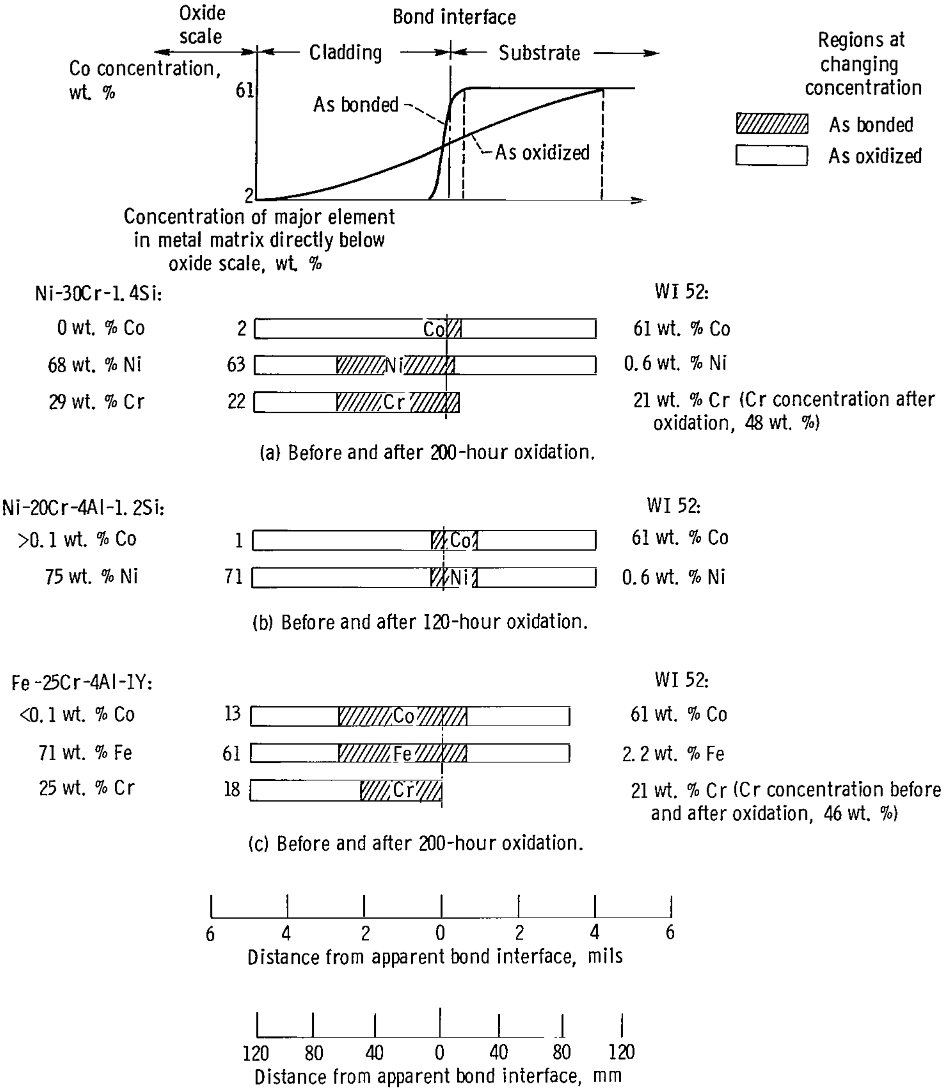


Figure 11. - Approximate extent of interdiffusion of major elements in clad WI 52 systems after bonding and oxidizing at 2000° F (1360 K).

4 mils (0.102 mm). Figure 11 shows that the Cr concentration before and/or after oxidation is a maximum at, or adjacent to, the original bond interface in both the Ni-Cr - WI 52 and Fe-Cr-Al-Y - WI 52 systems. This may have a bearing on the excellent oxidation resistance of these two systems.

SUMMARY OF RESULTS

An investigation was undertaken to explore the feasibility of using oxidation-resistant cladding alloys to protect superalloys during cyclic furnace oxidation at 1900° and 2000° F

(1310 and 1360 K). Foils, 5 mils (approx. 0.127 mm) thick, from three commercial heat-resistant alloys (Ni-30Cr-1.4Si, Ni-20Cr-4Al-1.2Si, and Fe-25Cr-4Al-1Y) were gas-pressure-bonded to IN 100 and WI 52 substrates (nickel- and cobalt-base superalloys, respectively). To provide a basis of comparison, the cladding foils were also gas-pressure-bonded to substrates of the same composition as the foils.

The results of this investigation are summarized in table X. All three cladding alloys were highly effective in providing oxidation protection for both superalloys at 1900° F (1310 K). At 2000° F (1360 K), however, the same claddings on different superalloys produced markedly different results. The Ni-20Cr-4Al-1.2Si afforded good protection for IN 100, while Ni-30Cr-1.4Si and Fe-25Cr-4Al-1Y alloys provided equally excellent protection for WI 52. The other three systems showed either high weight losses due to oxide scale spalling or extensive interdiffusion during oxidation at 2000° F (1360 K).

Extensive interdiffusion between the cladding alloy and the superalloy substrates was observed after oxidation at 2000° F (1360 K). However, despite this interdiffusion, the protective ability of the claddings in three of the six systems was not seriously degraded. These three systems were Ni-20Cr-4Al-1.2Si - IN 100, Ni-30Cr-1.4Si - WI 52, and Fe-25Cr-4Al-1Y - WI 52. In addition, diffusion zones within the substrates of these three most oxidation-resistant systems at 2000° F (1360 K) were less than 5.5 mils (0.140 mm) in depth after 200 hours. For very long exposures at these temperatures, it is believed that diffusion barriers could extend protective life in these oxidation-resistant systems, as well as in those systems whose poor oxidation resistance is traceable to interdiffusion.

Therefore, it is believed that low-strength, heater-type alloys are potentially feasible as claddings for protecting the high-strength, low-oxidation-resistant nickel- and cobalt-base superalloys. At least, the Ni-Cr-Al-type alloys appear to warrant further study for protecting nickel-base superalloys, while Ni-Cr- and Fe-Cr-Al-Y-type alloys are definitely worthy of further study as claddings for cobalt-base superalloys. Advanced evaluation under conditions more closely simulating those in an advanced gas turbine engine appear warranted.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, June 26, 1969,
129-03-05-03-22.

APPENDIX A

CLEANING PROCEDURE FOR BONDING COMPONENTS BEFORE GAS-PRESSURE BONDING

The cladding components were ultrasonically degreased in trichloroethylene at 250° F (390 K), chemically cleaned in 60-volume-percent H_2O + 20-volume-percent HF + 20-volume-percent HNO_3 at 135° F (330 K) for 5 minutes, washed in detergent, rinsed in water, and then ultrasonically cleaned first in acetone and then in alcohol.

The superalloy substrates were vapor-blasted with 100-mesh silica abrasive, washed in detergent, rinsed in water, and then ultrasonically cleaned first in acetone and then in alcohol.

The canning and diffusion barrier components were ultrasonically degreased in trichloroethylene at 250° F (390 K) and then ultrasonically cleaned first in acetone and then in alcohol.

APPENDIX B

ETCHANTS FOR METALLOGRAPHIC EXAMINATION

The etchant used for each specimen and the corresponding photomicrograph are listed in the following table:

Figure	Etchant
5(a)	Marble's etch
5(b)	Marble's etch
5(c)	50 ml acetic acid + 50 ml nitric acid
7(a-1)	20 ml water + 50 ml glycerine + 30 ml hydrochloric acid + 10 ml nitric acid + 1 ml hydrofluoric acid
7(a-2)	90 ml alcohol + 10 ml hydrochloric acid + 2 drops hydrogen peroxide
7(a-3)	20 ml water + 30 ml hydrochloric acid + 50 ml glycerine + 10 ml nitric acid + 1 ml hydrofluoric acid
7(b-1)	90 ml alcohol + 10 ml hydrochloric acid + 2 drops hydrogen peroxide
7(b-2)	20 ml water + 50 ml glycerine + 30 ml hydrochloric acid + 10 ml nitric acid + 1 ml hydrofluoric acid.
7(b-3)	20 ml water + 30 ml hydrochloric acid + 50 ml glycerine + 10 ml nitric acid + 1 ml hydrofluoric acid
7(c-1)	20 ml water + 30 ml hydrochloric acid + 50 ml glycerine + 10 ml nitric acid + 1 ml hydrofluoric acid
7(c-2)	90 ml alcohol + 10 ml hydrochloric acid + 2 drops hydrogen peroxide
7(c-3)	20 ml water + 30 ml hydrochloric acid + 50 ml glycerine + 10 ml nitric acid + 1 ml hydrofluoric acid
10(a-1)	90 ml alcohol + 10 ml hydrochloric acid + 2 drops hydrogen peroxide
10(a-2)	90 ml alcohol + 10 ml hydrochloric acid + 2 drops hydrogen peroxide
10(a-3)	20 ml water + 30 ml hydrochloric acid + 50 ml glycerine + 10 ml nitric acid + 1 ml hydrofluoric acid
10(b-1)	20 ml water + 30 ml hydrochloric + 50 ml glycerine + 10 ml nitric acid + 1 ml hydrofluoric acid
10(b-2)	90 ml alcohol + 10 ml hydrochloric acid + 2 drops hydrogen peroxide
10(b-3)	20 ml water + 30 ml hydrochloric acid + 50 ml glycerine + 10 ml nitric acid + 1 ml hydrofluoric acid
10(c-1)	20 ml water + 30 ml hydrochloric acid + 50 ml glycerine + 10 ml nitric acid + 1 ml hydrofluoric acid
10(c-2)	Cladding: 75 ml glycerine + 25 ml aqua regia Substrate: 90 ml alcohol + 10 ml hydrochloric acid + 2 drops hydrogen peroxide
10(c-3)	20 ml water + 30 ml hydrochloric acid + 50 ml glycerine + 10 ml nitric acid + 1 ml hydrofluoric acid

REFERENCES

1. Jackson, C. M.; and Hall, A. M.: Surface Treatments for Nickel and Nickel-Base Alloys. NASA TM X-53448, Apr. 20, 1966.
2. Nichols, E. S.; Burger, J. A.; and Hanink, D. K.: Comparative Evaluation of Protective Coatings for High Temperature Alloys. Paper 843E, SAE, Apr. 1964.
3. Williams, D. N.; Ernst, R. H.; MacMillan, C. A.; English, J. J.; and Bartlett, E. S.: Development of Protective Coatings for Chromium-Base Alloys. Battelle Memorial Inst. (NASA CR-54619), Mar. 5, 1968.
4. Monson, L. A.; and Pollock, W. I.: Development of Coatings for Protection of Dispersion Strengthened Nickel from Oxidation. Part I. Oxidation Studies, Coating Development and Coating Analysis. E. I. duPont de Nemours and Co., Inc. (AFML-TR-66-47, pt. 1, DDC No. AD-808519), Mar. 1966.
5. Stephan, H. R.; and Collins, J. F.: Iron-Chromium-Aluminum-Yttrium Alloy for Electrical Heating Elements. Rep. GEMP-440, General Electric Co., Oct. 18, 1966.
6. Collins, J. F.; and McConnaughey, N.: Iron-Chromium-Aluminum-Yttrium Alloys for Cladding Applications to 2400° F. Rep. APEX-681, General Electric Co., June 12, 1961.

TABLE I. - CHEMICAL ANALYSIS OF SUPERALLOY SUBSTRATES

IN 100	
Element	Content, wt. %
Major constituents	
Ni	61.14
Co	15.37
Cr	9.50
Al	5.33
Ti	4.26
Mo	3.17
Minor constituents	
V	0.82
Fe	.20
C	.166
Cb	.10
Zr	.087
Si	.08
Ta	[a]<.05
W	[a]<.05
Zn	[a]<.03
B	.0119
O	.0117
P	.0113
S	.0080
N	.0060
Mn	.003
Mg	[a]<.001
Cu	[a]<.0005
H	.00013

WI 52	
Element	Content, wt. %
Major constituents	
Co	61.24
Cr	21.42
W	11.09
Fe	2.24
Cb	1.87
Minor constituents	
Si	0.95
Ni	.55
C	.45
Mn	.40
Cu	.03
S	.0180
O	.0169
P	.0095
N	.0054
Ta	.0047
Ca	.003
B	.0029
Mg	.001
Ce	.001
Al	[a]<.001
Y	[a]<.0005
H	.00012

[a] Not detected, less than the limits of detection.

TABLE II. - CHEMICAL ANALYSIS AND SUPPLIERS OF CLADDING ALLOYS

Element	Content, wt.%		
	Ni-30Cr-1.4Si	Ni-20Cr-4Al-1.2Si	Fe-25Cr-4Al-1Y
Ni	Balance	Balance	0.15
Cr	29.63	19.54	24.82
Fe	.15	.7	Balance
Al	.3	3.51	4.12
Si	1.43	1.22	.18
Y	------	------	.58
Mn	.01	.4	------
Ca	.02	.005	[a]<.003
Co	.001	.03	.007
Cu	.001	.005	.005
Mg	.001	.001	------
V	.0015	------	.02
W	[a]<.05	[a]<.05	[a]<.05
Zr	.04	.01	[a]<.001
C	.028	.076	.0058
O	.010	.035	.027
N	.084	.15	.021
H	.00044	.0010	.00051
S	.0068	.0027	.0048
P	.0026	.0021	.0057
Supplier	Wilbur B. Driver Company	Driver-Harris Company	Nuclear Materials and Propulsion Operation, General Electric Company

[a] Not detected, less than the limits of detection.

TABLE III. - X-RAY FLUORESCENCE AND DIFFRACTION ANALYSES OF

System	Oxidation			X-ray fluorescence analysis - intensity of detected,[a] net counts					
	Temperature		Time, hr	NiK_α	CrK_α	AlK_α	FeK_α	CoK_α	SiK_α
	°F	K							
Ni-30Cr-1.4Si alloy	(b)		(b)	1758.2×10^3	229.6×10^3	3.4×10^3	7.1×10^3	-------	4.5×10^3
Ni-30Cr-1.4Si-clad Ni-30Cr-1.4Si	1900	1310	200	336.2×10^3	341.7×10^3	2.5×10^3	3.8×10^3	-------	8.8×10^3
	2000	1360	200	198.0	463.0	4.4	5.0	-------	13.9
Ni-20Cr-4Al-1.2Si alloy	(b)		(b)	1958.2×10^3	155.7×10^3	18.0×10^3	73.3×10^3	-------	3.3×10^3
Ni-20Cr-4Al-1.2Si-clad Ni-20Cr-4Al-1.2Si	1900	1310	200	1647.1×10^3	197.6×10^3	2.6×10^3	18.3×10^3	-------	1.9×10^3
	2000	1360	200	1539.6	202.7	3.7	18.2	-------	1.3
Fe-25Cr-4Al-1Y alloy	(b)		(b)	3.7×10^3	215.2×10^3	13.4×10^3	849.1×10^3	3.7×10^3	-------
Fe-25Cr-4Al-1Y-clad Fe-25Cr-4Al-1Y	1900	1310	200	6.5×10^3	137.6×10^3	342.5×10^3	620.1×10^3	3.0×10^3	-------
	2000	1360	200	8.8	93.8	427.8	504.1	2.9	-------

[a] Counts measured for constant time of 100 seconds.

[b] As-received cladding foil (not pressure bonded).

[c] Lattice parameter, a_o.

SURFACES OF CLADDING ALLOYS BEFORE AND AFTER CYCLIC OXIDATION

characteristic radiation of elements per minute						X-ray diffraction analysis of retained oxidation scale		Degree of visible spalling during oxidation
WL_α	MnK_α	TiK_α	MoK_α	CbK_α	YK_α	Major constituents	Minor constituents	
----	1.9×10^3	-----	3.2×10^3	-------	--------	--------------------------	----------	----
----	6.1×10^3	-----	4.0×10^3	-------	--------	Cr_2O_3	----------	None
----	7.3	-----	7.0	-------	--------	Cr_2O_3	----------	None
----	63.6×10^3	-----	-------	-------	--------	--------------------------	----------	----
----	16.5×10^3	-----	-------	-------	--------	Spinel (a_0 = 8.35±0.02Å)^c	----------	None
----	15.9	-----	-------	-------	--------	Spinel (a_0 = 8.25±0.02Å) + NiO	----------	Very light
----	3.1×10^3	-----	-------	1.9×10^3	16.9×10^3	--------------------------	----------	----
----	1.6×10^3	-----	-------	3.0×10^3	41.2×10^3	Al_2O_3 + $(Cr, Fe)_2O_3$	----------	None
----	1.3	-----	-------	4.0	53.9	Al_2O_3 + $(Cr, Fe)_2O_3$	----------	None

TABLE IV. - OXIDATION WEIGHT CHANGE OF CLAD CLADDING-ALLOY AND CLAD AND UNCLAD IN 100 SPECIMENS AFTER 200 HOURS

System	Specimen weight change at specified temperature, mg/cm^2	
	1900° F (1310 K)	2000° F (1360 K)
Ni-30Cr-1.4Si-clad Ni-30Cr-1.4Si	+1.3	+2.3
Ni-30Cr-1.4Si-clad IN 100	+2.9	[a]-12.1
Ni-20Cr-4Al-1.2Si-clad Ni-20Cr-4Al-1.2Si	+1.2	+1.0
Ni-20Cr-4Al-1.2Si-clad IN 100	+1.2	+.1
Fe-25Cr-4Al-1Y-clad Fe-25Cr-4Al-1Y	+1.0	+1.2
Fe-25Cr-4Al-1Y-clad IN 100	+1.6	+3.6
Unclad IN 100	-1.3	[b]-22.1

[a] After 180 hours.
[b] After 100 hours.

TABLE V. - RESULTS OF METALLOGRAPHIC EXAMINATION OF CLAD IN 100 SPECIMENS OXIDIZED AT 1900° AND 2000° F (1310 AND 1360 K)

System	Oxidation			Approximate thickness of unaffected cladding[a]		After oxidation at specified temperature and time		After pressure bonding[c]	
	Temperature		Time, hr	mils	mm	Approximate visual depth of diffusion within substrate[b]			
	°F	K				mils	mm	mils	mm
Ni-30Cr-1.4Si	As-received cladding			5.0±0.5	0.127±0.013	---	-----	---	-----
Ni-30Cr-1.4Si-clad IN 100	(c)	(c)	---	---	-----	---	-----	0.5	0.013
	1900	1310	200	2.7	0.069	2.5	0.064	---	-----
	2000	1360	60	3.4	.086	1.9	.048	---	-----
	2000	1360	160	2.3	.058	3.0	.076	---	-----
Ni-20Cr-4Al-1.2Si	As-received cladding			5.0±0.5	0.127±0.013	---	-----	---	-----
Ni-20Cr-4Al-1.2Si-clad IN 100	(c)	(c)	---	---	-----	---	-----	0.5	0.013
	1900	1310	200	4.7	0.119	1.7	0.043	---	-----
	2000	1360	40	4.8	.122	1.5	.038	---	-----
	2000	1360	200	4.3	.109	2.6	.066	---	-----
Fe-25Cr-4Al-1Y	As-received cladding			5.0±0.8	0.127±0.020	---	-----	---	-----
Fe-25Cr-4Al-1Y-clad IN 100	(c)	(c)	---	---	-----	---	-----	0.9	0.023
	1900	1310	200	3.2	0.081	3.8	0.097	---	-----
	2000	1360	200	3.0	.076	5.7	.145	---	-----

[a] Average of three measurements. Includes internal oxidation and voids, but not penetration of surface oxides.
[b] Average of three measurements.
[c] As pressure bonded at 2000° F (1360 K), 15 000 psi (1.03 MN/m^2), and 2 hr.

TABLE VI. - X-RAY FLUORESCENCE AND DIFFRACTION ANALYSES OF RETAINED SCALES ON CLAD IN 100 SPECIMENS AFTER CYCLIC OXIDATION

System	Oxidation			X-ray fluorescence analysis - elements showing an appreciable increase (or decrease) in intensity at surface[a]	X-ray diffraction analysis		Degree of visible spalling during oxidation
	Temperature		Time, hr		Major constituents	Minor constituents	
	°F	K					
Ni-30Cr-1.4Si-clad IN 100	1900	1310	200	Ti, Mo	Cr_2O_3	--------------------	Very light
	2000	1360	60	Ti	Cr_2O_3	--------------------	Medium
	2000	1360	160	(b)	(b)	(b)	Very heavy
Ni-20Cr-4Al-1.2Si-clad IN 100	1900	1310	200	Mo	Spinel (a_0 = 8.32±0.02Å)[c]	--------------------	None
	2000	1360	40	Mo, Al, Si	Spinel (a_0 = 8.30±0.02Å) + NiO	--------------------	Very light
	2000	1360	200	Mo, Al, Si, Co, Ti	Spinel (a_0 = 8.20±0.02Å) + NiO	--------------------	Light
Fe-25Cr-4Al-1Y-clad IN 100	1900	1310	200	Ni, Co, Ti, Mo	$(Cr, Fe)_2O_3$	Spinel (a_0 = 8.25±0.02Å)	None
	2000	1360	200	Ni, Co, Ti, Mo, (Al)	$(Cr, Fe)_2O_3$	--------------------	Light to medium

---

[a] Intensity change of ≥ +200 percent (≤ -66 percent) compared to data obtained on clad cladding-alloy specimens oxidized at same temperatures. (Decreasing elements are in parentheses.)

[b] Results considered to be meaningless since heavy spalling occurred during oxidation.

[c] Lattice parameter, a_0.

TABLE VII. - OXIDATION WEIGHT CHANGE OF CLAD CLADDING-ALLOY AND CLAD AND UNCLAD WI 52 SPECIMENS AFTER 200 HOURS

System	Specimen weight change at specified temperature, mg/cm^2	
	1900° F (1310 K)	2000° F (1360 K)
Ni-30Cr-1.4Si-clad Ni-30Cr-1.4Si	+1.3	+2.3
Ni-30Cr-1.4Si-clad WI 52	+1.2	+1.9
Ni-20Cr-4Al-1.2Si-clad Ni-20Cr-4Al-1.2Si	+1.2	+1.0
Ni-20Cr-4Al-1.2Si-clad WI 52	+.9	[a]-3.1
Fe-25Cr-4Al-1Y-clad Fe-25Cr-4Al-1Y	+1.0	+1.2
Fe-25Cr-4Al-1Y-clad WI 52	+1.0	+1.2
Unclad WI 52	[b]-22.9	[b]-89.0

[a] After 120 hr.

[b] After 100 hr.

TABLE VIII. - RESULTS OF METALLOGRAPHIC EXAMINATION OF CLAD WI 52 SPECIMENS OXIDIZED AT 1900° AND 2000° F (1310 AND 1360 K)

System	Oxidation			Approximate thickness of unaffected cladding[a]		After oxidation at specified temperature and time		After pressure bonding[c]	
	Temperature		Time, hr			Approximate visual depth of diffusion within substrate[b]			
	°F	K		mils	mm	mils	mm	mils	mm
Ni-30Cr-1.4Si	As-received cladding			5.0±0.5	0.127±0.013	---	-----	---	-----
Ni-30Cr-1.4Si-clad WI 52	(c)	(c)	---	---	-----	---	-----	0.3	0.008
	1900	1310	200	4.4	0.112	0.5	0.013	---	-----
	2000	1360	60	4.3	.109	.8	.020	---	-----
	2000	1360	200	4.0	.101	1.4	.036	---	-----
Ni-20Cr-4Al-1.2Si	As-received cladding			5.0±0.5	0.127±0.013	---	-----	---	-----
Ni-20Cr-4Al-1.2Si-clad WI 52	(c)	(c)	---	---	-----	---	-----	0.4	0.010
	1900	1310	200	4.5	0.114	1.3	0.033	---	-----
	2000	1360	40	4.6	.117	1.4	.036	---	-----
	2000	1360	120	3.1	.079	1.7	.043	---	-----
Fe-25Cr-4Al-1Y	As-received cladding			5.0±0.8	0.127±0.020	---	-----	---	-----
Fe-25Cr-4Al-1Y-clad WI 52	(c)	(c)	---	---	-----	---	-----	0.5	0.013
	1900	1310	200	4.0	0.101	1.5	0.038	---	-----
	2000	1360	200	2.9	.074	1.6	.041	---	-----

[a] Average of three measurements. Includes internal oxidation and voids, but not penetration of surface oxides.

[b] Average of three measurements.

[c] As pressure bonded at 2000° F (1360 K), 15 000 psi (1.03 MN/m^2), and 2 hr.

TABLE IX. - X-RAY FLUORESCENCE AND DIFFRACTION ANALYSES OF RETAINED SCALES ON CLAD WI 52 SPECIMENS AFTER CYCLIC OXIDATION

System	Oxidation			X-ray fluorescence analysis - elements showing an appreciable increase (or decrease) in intensity at surface[a]	X-ray diffraction analysis		Degree of visible spalling during oxidation
	Temperature		Time, hr		Major constituents	Minor constituents	
	°F	K					
Ni-30Cr-1.4Si-clad WI 52	1900	1310	200	Cb, (Si)	Cr_2O_3 + spinel (a_o = 8.35±0.02Å)[b]	--------------------	None
	2000	1360	60	Ni, Cb, (Si), (Al)	Cr_2O_3	Spinel (a_o = 8.30±0.02Å)	None
	2000	1360	200	Co, Cb, (Si), (Al)	Cr_2O_3	--------------------	None
Ni-20Cr-4Al-1.2Si-clad WI 52	1900	1310	200	Al, Co, Cb	Spinel (a_o = 8.40±0.02Å)	Cr_2O_3	None
	2000	1360	40	Al, Co, Cb, Si	Spinel (a_o = 8.15±0.02Å) + NiO	Cr_2O_3	Medium
	2000	1360	120	(c)	(c)	(c)	Very heavy
Fe-25Cr-4Al-1Y-clad WI 52	1900	1310	200	Co, W	Al_2O_3 + $(Cr, Fe)_2O_3$	--------------------	None
	2000	1360	200	Co, W, Mn	Al_2O_3 + $(Cr, Fe)_2O_3$	--------------------	None

[a] Intensity change of ≥ +200 percent (≤ -66 percent) compared to data obtained on clad cladding-alloy specimens oxidized at the same temperature. (Decreasing elements are in parentheses.)

[b] Lattice parameter, a_o.

[c] Results were considered meaningless since heavy spalling occurred during oxidation.

TABLE X. - SUMMARY OF RESULTS OF CYCLIC OXIDATION OF UNCLAD AND CLAD SUPERALLOYS

System[a]	Oxidation time, hr	Specimen weight change, mg/cm^2	Spalling resistance up to specified oxidation time	Metallographic examination					Extent of interdiffusion as determined by electron microprobe examination		
				Cladding thickness unaffected by oxidation[b]		Internal oxidation	Approximate diffusion-zone thickness[c]		Cladding	Substrate depth	
				mils	mm		mils	mm		mils	mm
1900° F (1310 K) Oxidation											
Unclad IN 100	200	[d]-1.3	Fair	---	-----	------------------	---	-----	----------	---	-----
Ni-30Cr-1.4Si-clad IN 100	↓	+2.9	Very good	2.7	0.069	None	2.5	0.064	----------	---	-----
Ni-20Cr-4Al-1.2Si-clad IN 100		+1.2	Excellent	4.7	.119	None	1.7	.043	----------	---	-----
Fe-25Cr-4Al-1Y-clad IN 100		+1.6	Excellent	3.2	.081	None	3.8	.097	----------	---	-----
Unclad WI 52	100	[d]-22.9	Very poor	---	-----	------------------	---	-----	----------	---	-----
Ni-30Cr-1.4Si-clad WI 52	200	+1.2	Excellent	4.4	.112	None	0.5	.013	----------	---	-----
Ni-20Cr-4Al-1.2Si-clad WI 52	200	+.9	Excellent	4.5	.114	None	1.3	.033	----------	---	-----
Fe-25Cr-4Al-1Y-clad WI 52	200	+1.0	Excellent	4.0	.101	Very slight in cladding	1.5	.038	----------	---	-----
2000° F (1360 K) Oxidation											
Unclad IN 100	100	[d]-22.1	Very poor	---	-----	------------------	---	-----	----------	---	-----
Ni-30Cr-1.4Si-clad IN 100	160	-2.2	Very poor	2.3	0.058	Throughout cladding	3.0	0.076	Throughout	6.5	0.165
Ni-20Cr-4Al-1.2Si-clad IN 100	200	+.1	Good	4.3	.109	None	2.6	.066	Throughout	5.2	.132
Fe-25Cr-4Al-1Y-clad IN 100	200	+3.6	Fair	3.0	.076	Throughout cladding	5.7	.145	Throughout	8.6	.218
Unclad WI 52	100	[d]-89.0	Very poor	---	-----	------------------	---	-----	----------	---	-----
Ni-30Cr-1.4Si-clad WI 52	200	+1.4	Excellent	4.0	.101	Throughout cladding	1.4	.036	Throughout	3.9	.099
Ni-20Cr-4Al-1.2Si-clad WI 52	120	-3.1	Very poor	3.1	.079	Throughout cladding	1.7	.043	Throughout	4.0	.101
Fe-25Cr-4Al-1Y-clad WI 52	200	+1.3	Excellent	2.9	.074	Throughout cladding	1.6	.041	Throughout	3.3	.084

[a] Cladding thickness on major surfaces of superalloy substrates was nominally 0.005 inch (0.127 mm).

[b] Average of three measurements. Includes internal oxidation and voids, but not penetration of surface oxides.

[c] Average of three visual measurements of the zone on the substrate side of bond interface.

[d] Average of two specimens.

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