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Oxidation and Hot Corrosion of Superalloys Rene 100 and X-40 at Temperatures of 1475°F to 2000°F

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The oxidation resistance and hot corrosion resistance of two superalloys, Rene 100 and X-40, and an aluminide coating for the protection of one of the superalloys are compared. The comparison is made by using tube-furnace oxidation and hot corrosion tests employing a Mettler TGA apparatus at temperatures of 1475°F to 2200°F. For hot corrosion tests the samples were precoated with Na_2SO_4 prior to heating to the desired temperature and introducing air at a flow rate of 57 cm/min. The oxidation behavior to Rene 100 was poor compared to that of the X-40 alloy, especially at temperatures above 1900°F. The X-40 alloy was found to be more resistant to the initiation of hot corrosion than Rene 100. Both alloys, however, undergo attack at rates consistent with an alloy-induced fluxing mechanism. An aluminide coating on Rene 100 caused the initiation of hot corrosion to be delayed. Attack did occur when cumulative applications of Na_2SO_4 were used The coatings were penetrated by the Na_2SO_4 in localized areas after exposure times of less than 200 hours. The hot corrosion test has validity for mechanisms whereby the degradation process is sustained by reaction products from the alloy. It, therefore, can be used to compare the time to establish such conditions in alloys. Rene 100 is more susceptible to this condition than X-40.

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Introduction

The subject of hot corrosion (deposit-modified, gaseous corrosion) of both nickel and cobalt alloys has received considerable attention in recent years because of the deleterious effects this form of degradation can produce on alloys exposed to combustion gases; e.g., gas turbines, fireside corrosion of boiler tubes, incinerators, etc. Most investigators in the field are in agreement that deposition of alkali sulfates; e.g., Na_2SO_4, which results from salt ingestion into the engine, alkali metals in the fuel, and sulfur gases from combustion of fuels is a normal precursor to hot corrosion. This problem appears to be more serious in marine environments due to the large amount of salts that are ingested with the intake air. Von Doering and Bergman [1], as well as Bornstein and DeCrescente [2], concluded that Na_2SO_4 is formed in turbine engines operating in such environments according to the following reactions:

$$2NaCl + SO_2 = 1/2\ O_2 + H_2O = Na_2SO_4 + 2HCl \tag{1}$$

$$2NaCl + SO_3 + H_2O = Na_2SO_4 + 2\ HCl \tag{2}$$

It has been established that hot corrosion may take place in the temperature range between approximately 1200°F and 1900°F. It is not considered to be a problem above 1900°F where deposits of sulfates are not significant, nor below about 1200°F where the deposits usually are solid. At temperatures between 1200°F and 1500°F a form of degradation called low-temperature hot corrosion is often observed where SO_3 in the gas is required to maintain this attack. Reaction rates far in excess of oxidation rates have been noted in hot corrosion environments at elevated temperatures, however, and salt deposition at lower temperatures, as well as certain phases formed in alloys at the lower temperatures, can affect the high temperature oxidation behavior.

A variety of techniques can be used to study hot corrosion attack of alloys. Three widely used approaches are: (1) laboratory tests in which samples are precoated with Na_2SO_4 and exposed to gases with controlled compositions [3,4], (2) laboratory tests in which samples are immersed in molten salts such as Na_2SO_4 and NaCl (crucible tests) [5], and (3) dynamic rig testing which attempts to establish an environment similar to that existing in gas turbine engines [4,6]. The laboratory tests using deposits must be used with care since these conditions are different from use conditions. Nevertheless, such tests do have comparative value when hot corrosion attack is observed since alloy behavior can be compared and microstructural features documented for defined conditions. The purpose of this investigation was to compare the oxidation and hot corrosion of two superalloys and demonstrate the efficacy of a laboratory test that employed specimens precoated with Na_2NO_4. Also, the hot corrosion resistance of an aluminide coating for the protection of one of the superalloys was assessed.

Materials and Procedures

The superalloys selected were Rene 100 (nickel-base) and X-40 (cobalt-base) which were cast by Howmet Corporation* into bars approximately 152.4 mm long by

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*Howmet Corporation, Austenal Microcast Division, Roy Street, Dover, NJ

12.7 mm in diameter. These alloys were used since both had compositions; e.g., refractory metal concentrations, which could make them susceptible to hot corrosion attack. The chemical compositions of the alloys in weight percent are shown below.

	Cr	W	Ni	C	Fe	S	Zr	P
X-40	25.10	7.70	10.40	0.5	0.27	0.003	0.02	0.01
Rene 100	9.60	--	Bal	0.17	0.15	0.001	0.06	--
	Mn	**Si**	**Co**	**Mo**	**Ti**	**Al**	**V**	**B**
X-40	0.10	0.10	Bal	--	--	--	--	--
Rene 100	0.01	0.04	14.98	3.00	4.32	5.50	1.08	0.013

The protective coating selected for study was CODEP B, a proprietary coating formulated by General Electric Company (contains aluminum, titanium, and carbon) and applied by Walbar Laboratories, Peabody, MA.

Specimens were machined from these bars in the form of discs, 3.18 mm thick by 12.7 mm in diameter, polished through 0/2 emery, rinsed in ethanol, and weighed. Specimens in this condition were used for oxidation studies, whereas specimens used for hot corrosion and coating studies were further prepared as follows: specimens were placed on a hot plate at 480°F and sprayed with a saturated solution of Na_2SO_4 until the desired amount per unit area of 1 mg/cm^2 was obtained. The thin coating of Na_2SO_4 produced in this manner showed good adherence and appeared uniform in thickness.

Etchants used for metallurgical examination of the superalloys were:

Rene 100: 80 cc H_2O, 20 cc HCl, 20 cc H_2O_2 (30%)

X-40: 50 cc H_2O, 50 cc HCl, 20 cc H_2O_2 (3%)

The Mettler TGA apparatus was used for both oxidation and hot corrosion kinetic studies. Two procedures were utilized. In the first, the furnace chamber containing the specimen was evacuated to 5 x 10^{-5} torr prior to flushing with argon and then re-evacuated. Argon was next introduced into the system until a pressure slightly greater than atmospheric was obtained and the furnace heated to the desired temperature at a heating rate of 77°F per minute. When at temperature, air was introduced into the system at a flow rate of 57 cm/min and the weight change automatically and continuously recorded. A more detailed explanation of this procedure, together with the type of apparatus employed, has been described elsewhere [7,8]. The specimen is thus subjected to an argon preheat treatment in this procedure. In the second procedure, the furnace, after being heated to the desired temperature, was lowered over a quartz furnace chamber containing the specimen as air was introduced at a flow rate of 57 cm/min and the weight change recorded automatically and continuously. Specimens used in this procedure were not exposed to an argon preheat treatment. In both procedures the reaction was terminated by replacing the air with argon. The first procedure was used for the oxidation and hot corrosion of both superalloys, whereas the second was followed for the hot corrosion tests only

Results and Discussion

Oxidation

The data obtained for X-40 are presented in Figure 1 where weight changes as a function of time are presented for a number of temperatures between 1475°F to 2200°F. These data, with the exception of the 2200°F test, approximate a parabolic rate law with rate constants in reasonable agreement with those for growth of Cr_2O_3 scales on alloys [9]. Parabolic kinetics are also followed at 2200°F, but the rate constant is significantly less than that for growth of Cr_2O_3 and this may result from the formation of volatile products; i.e., CrO_3, at this temperature.

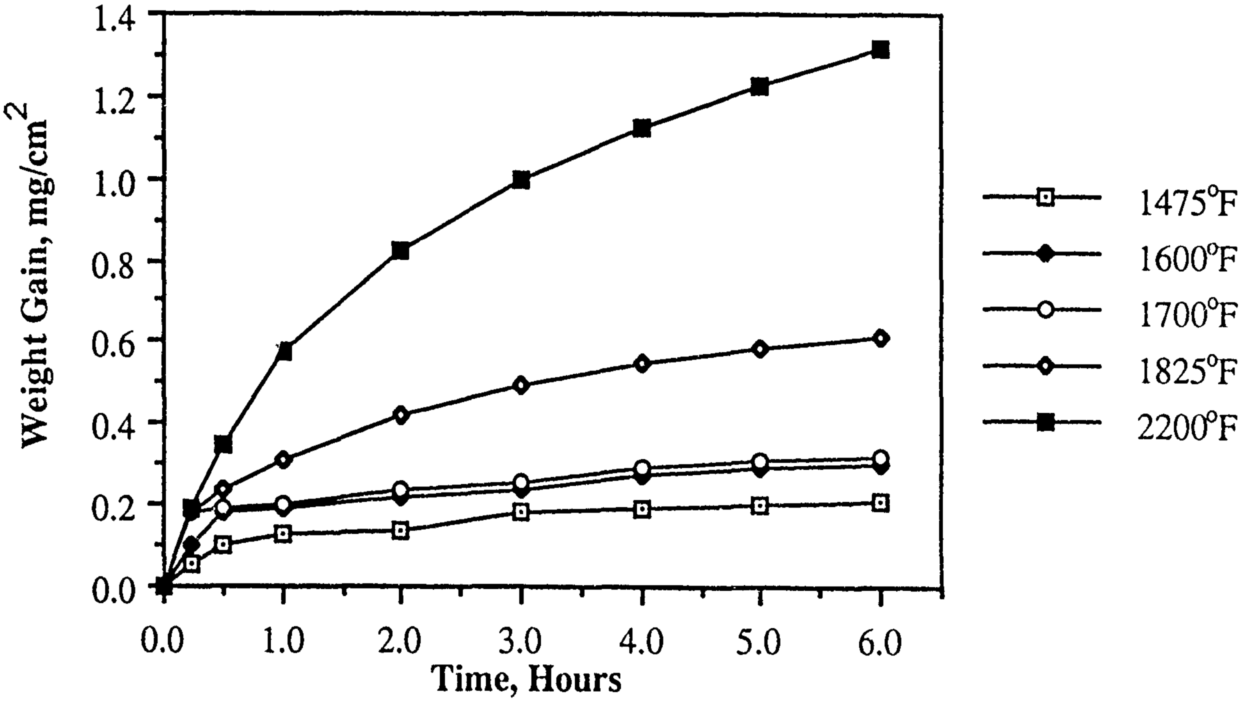


Figure 1. Oxidation of X-40: Air Flow Rat› = 57 cm/min, Pressure = 760 Torr.

The oxidation behavior of Rene 100 (see Figure 2) was poor compared to that for X-40; this was especially the case at the higher temperatures. Examination of Figure 2 shows that the weight gains for Rene 100 after two hours were almost always greater than those for X-40 exposed to the same temperature for six hours. Moreover, the weight change/time plots for Rene 100 had shapes for 2100°F and 2200°F that indicated the oxide scales were cracking at the temperature of oxidation. These data conformed to a parabolic rate law; however, rate constants greater than those for the growth of nickel oxide scales were obtained. For example, at 2200°F a rate constant of 10^{-8} (g^2/m^4-s) was obtained for Rene 100. Inspection of the composition of Rene 100 suggest that Al_2O_3 scales could be formed on this alloy but probably only after extensive amounts of transient oxidation during which oxides such as NiO, Cr_2O_3, CoO, and TiO_2 are formed. The data which has been obtained indicates Al_2O_3 scales may begin to develop but that cracking must render this process ineffective. The alloy, therefore, has poor oxidation resistance compared to X-40, especially at temperatures above 1900°F.

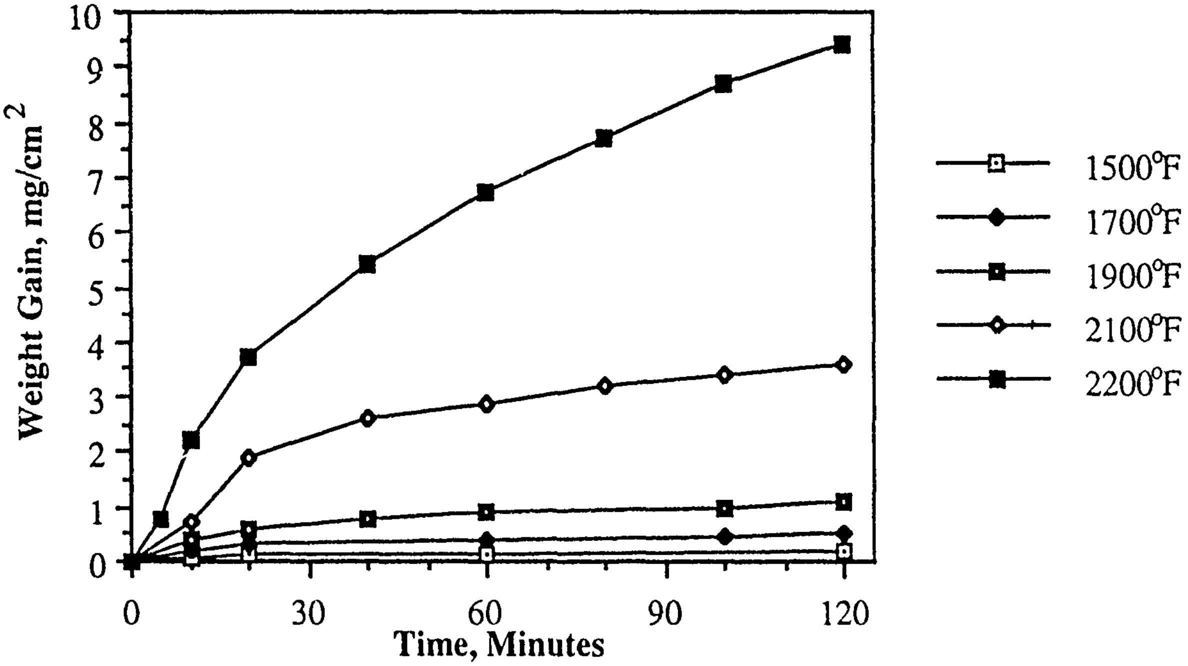


Figure 2. Oxidation of Rene 100: Air Flow Rate = 57 cm/min, Pressure = 760 Torr.

Cyclic oxidation tests were not performed because it was apparent from the isothermal results that Rene 100 would oxidize rapidly and that X-40 was a chromia-former.

Hot Corrosion

The weight change data for hot corrosion experiments must always be examined with care since gaseous products can be formed in addition to condensed phases at the surfaces of specimens. The weight change versus time curves for hot corrosion of X-40 always exhibited an initial rapid increase followed by a long period of very small weight changes (see Figure 3). The weight changes were always significantly larger than those for oxidation, as can be seen by comparing the data presented in Figures 1 and 3. Furthermore, the initial rapid increase in weight due to the presence of Na_2SO_4 became greater as the amount of Na_2SO_4 was increased (see Figure 3).

In Figures 4 and 5 weight change data for X-40 are presented for the two different procedures used in the hot corrosion tests. In the experiments involving the pretreatment with argon, SO_2 was identified (by a starch-iodate test) as an effluent which indicates that the Na_2SO_4 may be decomposing according to the reaction.

$$Na_2SO_4 = Na_2O + SO_2 \; 1/2 \; O_2 \qquad (3)$$

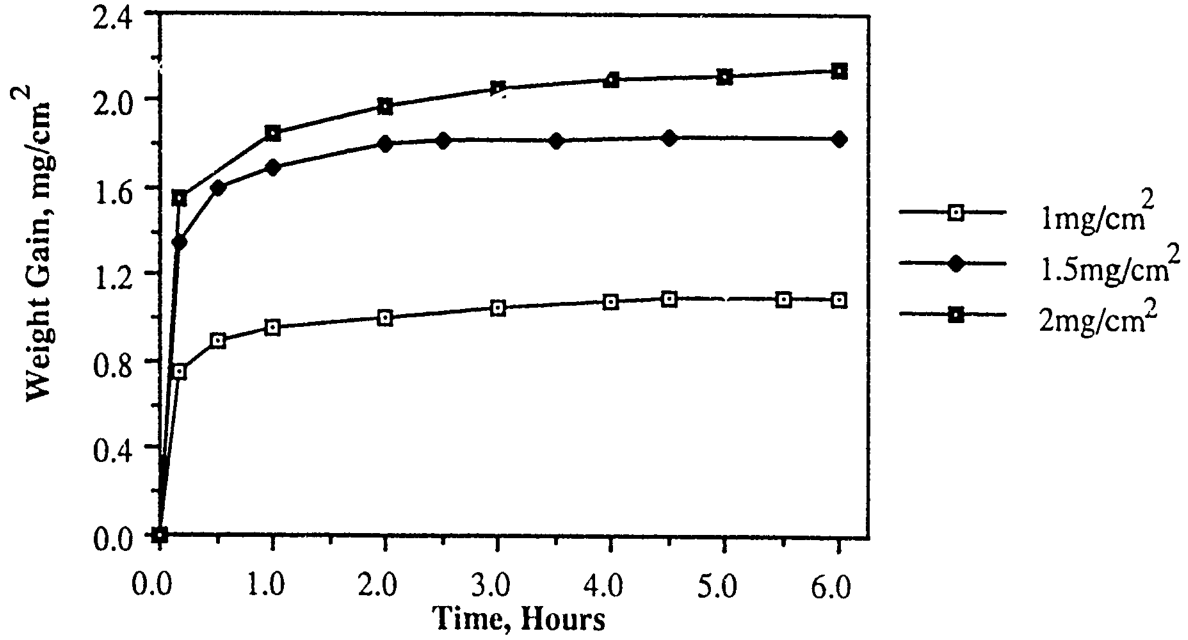


Figure 3. Hot corrosion versus Na_2SO_4 concentration on X-40 at 1600°F. Air Flow Rate = 57 cm/min, Pressure = 760 Torr.

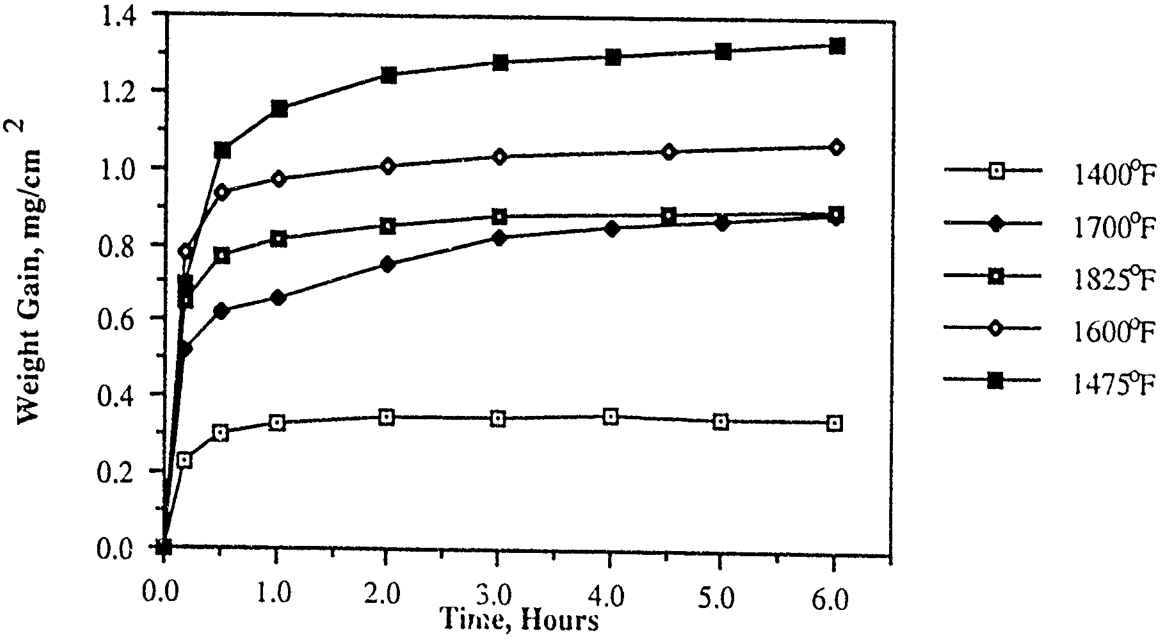


Figure 4. Hot corrosion of X 40 with 1 mg Na_2SO_4/cm^2 Air Flow Rate = 57 cm/min, Pressure = 760 Torr, Ar pretreated.

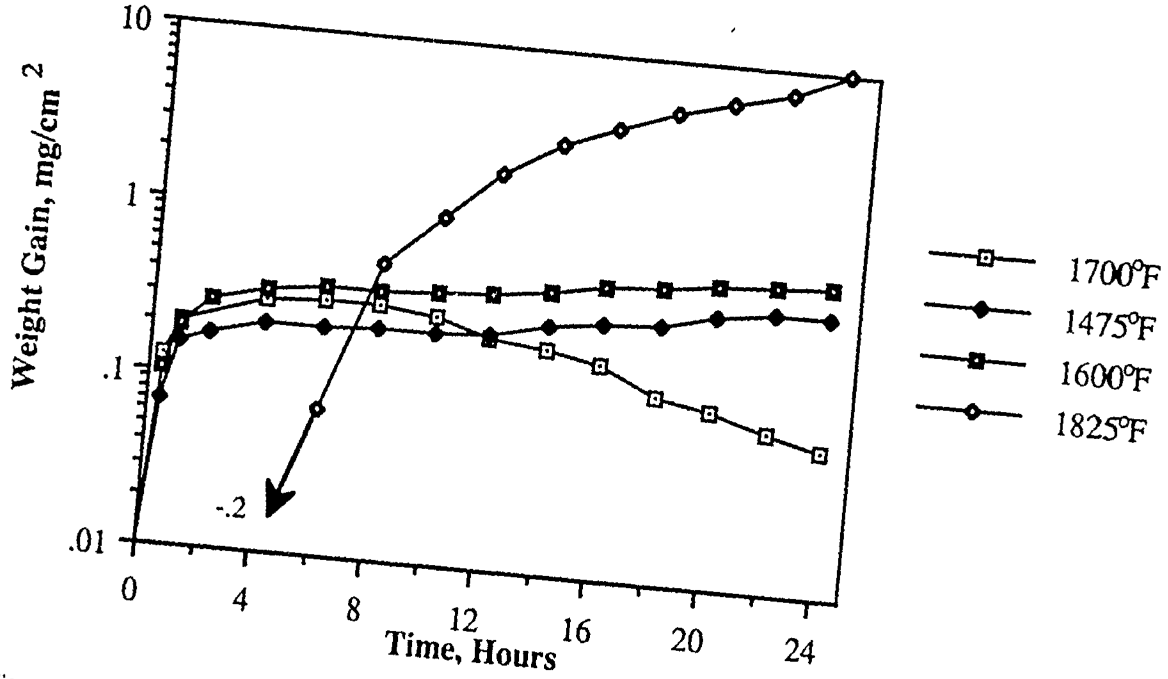


Figure 5. Hot corrosion of X-40 with 2 mg Na_2SO_4/cm^2. Air Flow Rate = 57 cm/min, Pressure = 760 Torr, No Ar pretreatment.

The presence of Na_2O was also determined by a basicity test (Na_2O makes Na_2SO_4 basic in aqueous solutions). All of the weight change data are greater than those for oxidation in the absence of Na_2SO_4 and the deposit, therefore, affected the corrosion process. In the case of the argon pretreatment the acceleration is restricted to the very early stages. The effect of temperature is maximum in the range of 1475°F to 1600°F. These results indicate that during the argon pretreatment some sulfide phases are formed at the surfaces of the X-40 specimens. This probably varies with temperature and becomes less at the higher temperatures due to higher losses of SO_2 via decomposition of the Na_2SO_4. The increased oxidation, therefore, would be caused by the oxidation of the sulfides which subsides as the sulfides are converted to oxides. It is important to emphasize that while the Na_2SO_4 has caused increased attack, this attack is very small compared to what will be observed when alloy induced acidic fluxing begins to become important.

The weight change data obtained for the samples heated in air (see Figure 5) are influenced by the gradual increase in temperature of the specimen (the specimen was found to reach the test temperature after approximately 10 minutes), weight losses due to SO_2 evolution from the Na_2SO_4, and the formation of corrosion products on the surfaces of the specimens. At temperatures between 1475°F to 1700°F the results are not much different from the argon pretreatments exhibiting an initial larger increase in weight followed by a leveling off of the corrosion rates. These data are believed to be less reliable than those obtained with the argon pretreated samples since, in the latter, the weight losses resulting from decomposition of Na_2SO_4 occurred prior to the beginning of the weight change measurements. The results obtained with the X-40 specimen exposed to air and heated to 1825°F, however, show that very severe attack has occurred. In view of the relatively large amount of tungsten in this alloy, it appears that oxides of tungsten must have been formed and reacted with the Na_2SO_4 to produce an acidic melt that causes alloy-induced acidic fluxing to take place [5,9]. Such attack does not occur with the argon pretreatment since the Na_2SO_4 decomposes and is not present during that part of the experiment where air is present.

The hot corrosion of X-40 became more severe at temperatures of 2100°F and 2200°F, as shown in Figure 6. Attack was evident after a few minutes of exposure. The amount of degradation was much more than the amount of Na_2SO_4. Such results show that oxidation products must be sustaining the rapid degradation. This is consistent with alloy-induced acidic fluxing.

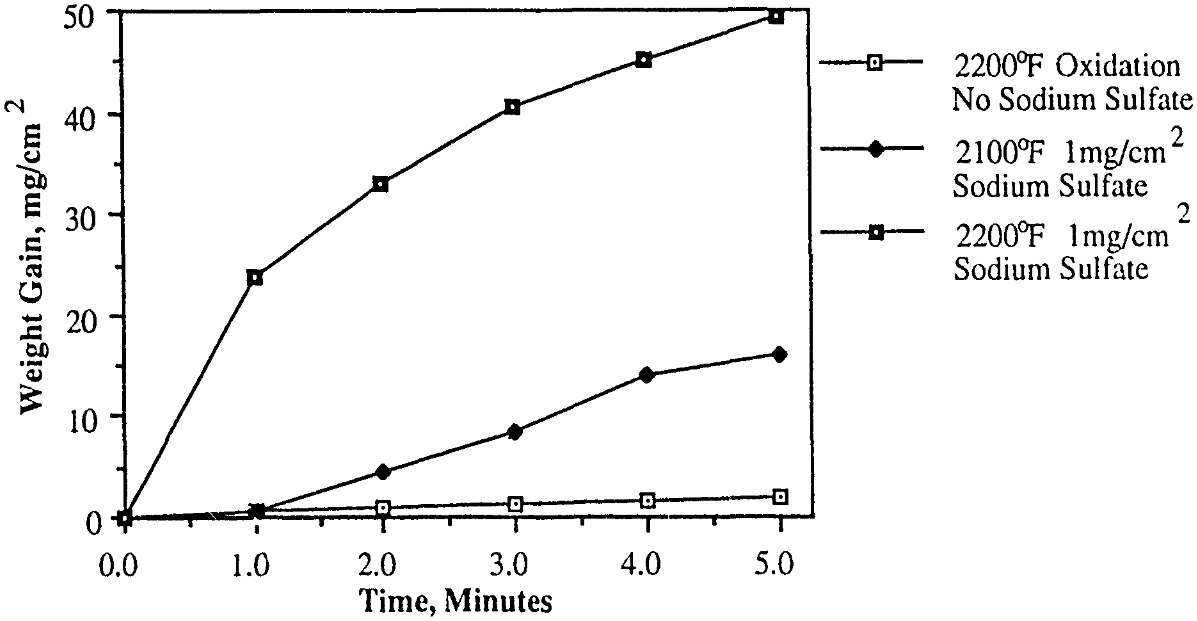


Figure 6. Comparison of oxidation and hot corrosion of X-40 at elevated temperatures

Results obtained from hot corrosion tests with Rene 100 are presented in Figure 7. Very severe attack is obvious upon comparison of the hot corrosion data with that for oxidation. The effects are especially severe at temperatures of 1800°F and above where the data again suggest degradation via an alloy-induced acidic fluxing process originating from the molybdenum and vanadium in Rene 100. Experiments were also performed where Na_2SO_4-coated specimens were exposed at 1500°F for two hours and then re-exposed at 2100°F without further addition of Na_2SO_4. The results are compared to hot corrosion and oxidation data obtained at 2200°F in Figure 8. It is evident that the exposure at 1500°F has caused the hot corrosion at 2100°F to be more severe. This probably has occurred by decreasing the time required to initiate the alloy-induced acidic fluxing at 2100°F and, hence, weight changes even greater than hot corrosion at 2200°F are observed.

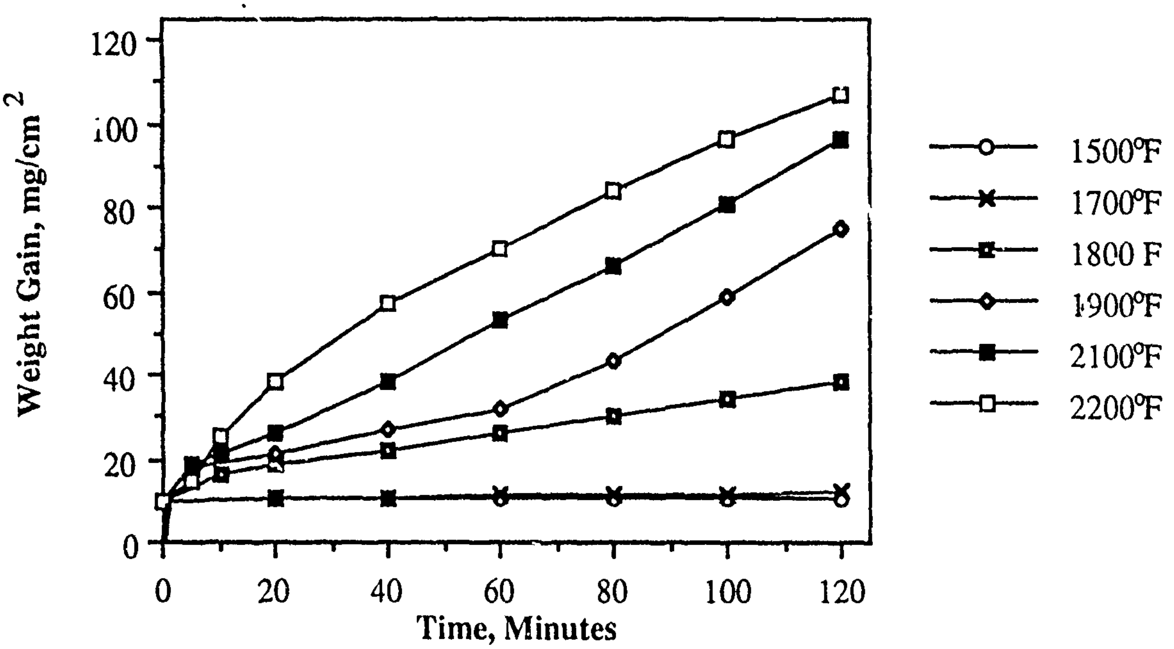


Figure 7. Hot corrosion of Rene 100 with 1 mg Na_2SO_4/cm^2: Air Flow Rate = 57 cm/min, Pressure = 760 Torr

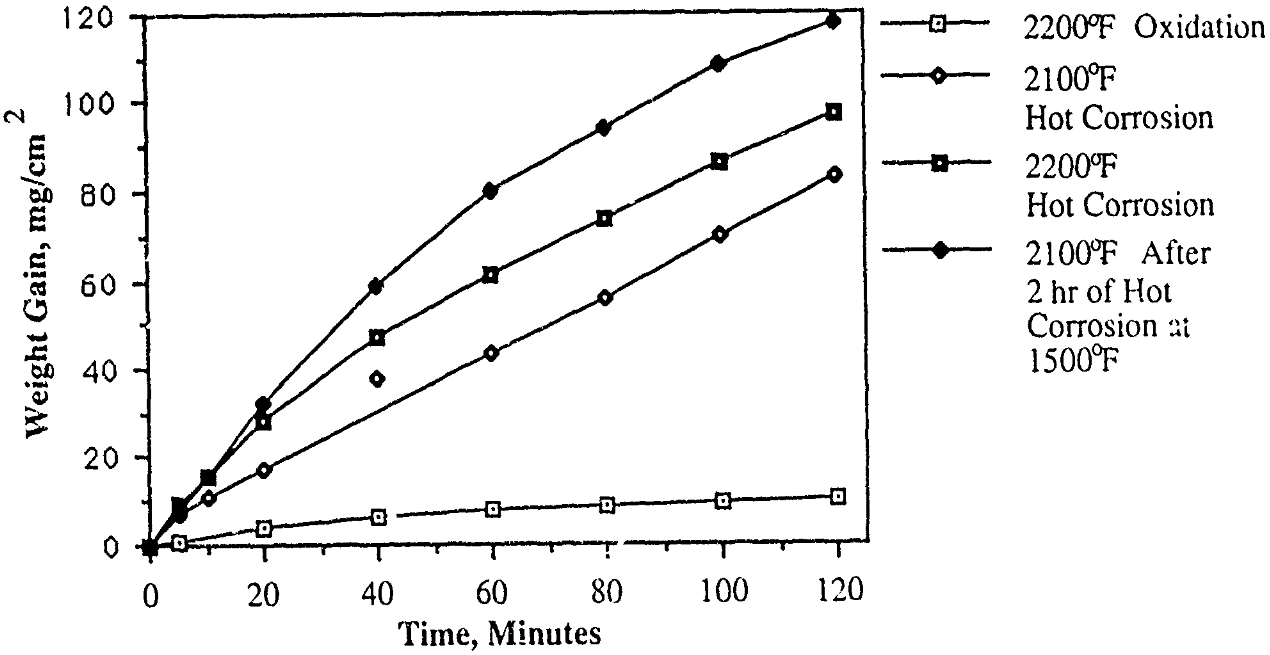
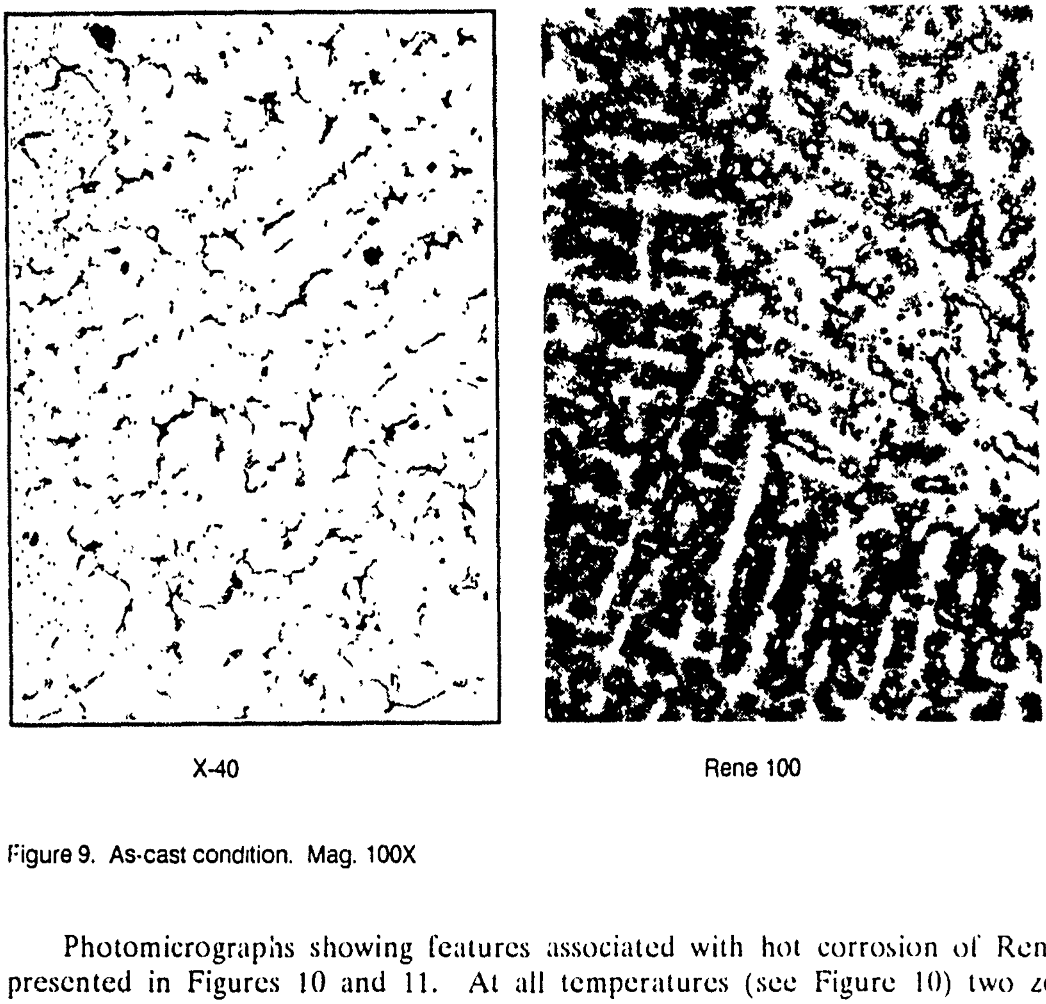


Figure 8. Oxidation versus hot corrosion of Rene 100 Air Flow Rate = 57 cm/min, Pressure = 760 Torr

Metallographic Analyses of Degraded Specimens

Results obtained from metallographic examination of specimens exposed to the hot corrosion tests were consistent with the weight change data. The microstructural features of the as-fabricated alloys are presented in Figure 9. Both alloys exhibit large grains composed of cored dendrites which are more readily seen in the nickel-base alloy. Carbides were evident as an interdendritic network distributed throughout both alloys. The major carbide in this network is believed to be $Cr_{23}C_6$ in X-40 [10], and also probably in Rene 100 also.



- Large grains of cored dendrites
- Inter dendritic network of carbides, probably $Cr_{23}C_6$

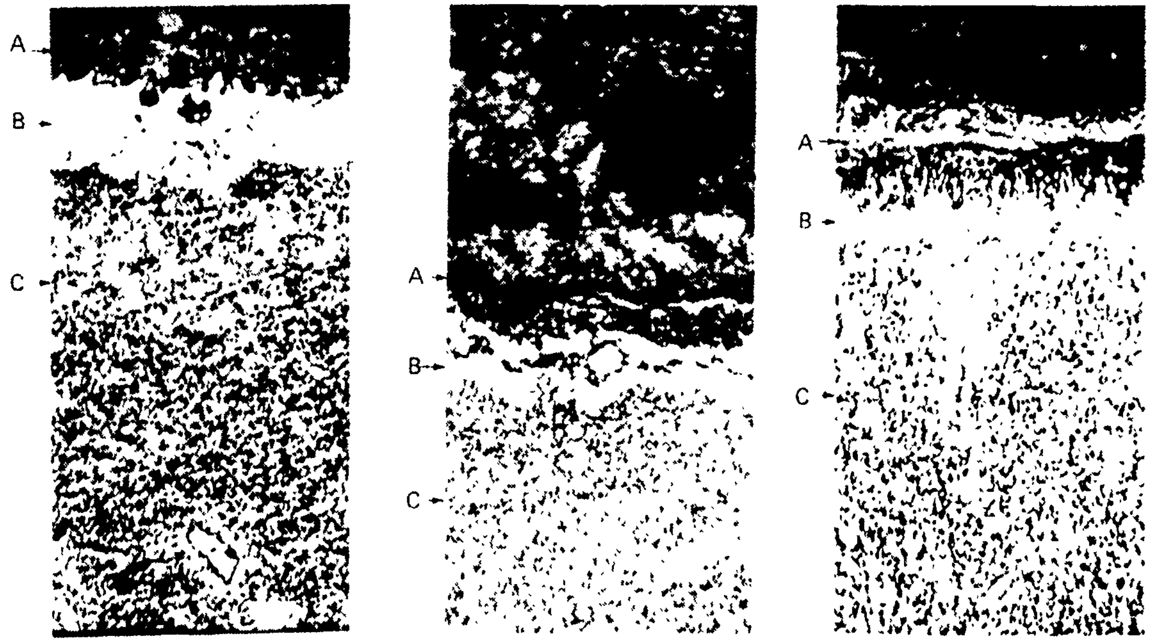
X-40

Rene 100

Figure 9. As-cast condition. Mag. 100X

Photomicrographs showing features associated with hot corrosion of Rene 100 are presented in Figures 10 and 11. At all temperatures (see Figure 10) two zones were evident. The outermost layer (A) was found to consist chiefly of oxides. The major phase in this zone appeared to be NiO; however, the proportions of $NiCr_2O_4$ and $NiMoO_4$ increased as the second zone (B) was approached. This latter zone consisted of the substrate depleted of chromium (white) and a grey sulfide phase. This sulfide phase is presumed to be mainly chromium sulfides based upon results obtained by Seybolt [11] and Hamilton [12] on nickel-base alloys with similar compositions. Chromium has a large affinity for sulfur, and when sulfur is present in the environment, chromium sulfides are usually formed in the depleted zone which develops due to oxidation as well as the formation of the chromium sulfides. Very often preferential oxidation of the sulfide is evident at 1700°F and 1900°F (see Figure 10)

At 2100°F and 2200°F oxidation of the depleted zone occurs by a rather uniform frontal attack where particles of alloy (white) are encompassed with oxide (see Figures 10 and 11). Such morphologies are consistent with alloy-induced acidic fluxing caused by oxidation of the molybdenum and vanadium in the Rene 100.



1700°F 1900°F 2100°F

Figure 10. Hot corrosion of Rene 100 after two hours Mag. 1000X

Legend
A. NiO, $NiO_4 \bullet Cr_2O_3$, $NiMoO_4$
B. Chromium depleted zone (white) containing Cr sulfides (grey)
C. Substrate

Microstructures developed during the hot corrosion of X-40 at 1700°F and 1825°F are presented in Figure 12. An external oxide scale (A) is evident. X-ray diffraction analysis of this zone indicated the outermost portion to be composed of NiO and cobalt oxides. The inner portion of this zone also contained these oxides, as well as large amounts of spinel phases; namely, $NiCr_2O_4$ and $CoCr_2O_4$. Preferential oxidation of the X-40 was also evident. This appeared to occur at alloy grain boundaries where sulfides had been formed in the alloy (see Figure 12). No positive identification of sulfides in the oxide scale was obtained by X-ray analyses. This is consistent with the sulfides in the substrate becoming oxidized [13]. Sulfide formation followed by oxidation is a common feature for some forms of hot corrosion. This type of preferential attack causes increased oxidation and can eventually result in the onset of alloy-induced acidic fluxing, whereby tungsten in the X-40 becomes oxidized and combines with the Na_2SO_4 to form an acidic deposit.

The limited coating test results show that penetration of the coating occurs after exposure periods of less than 200 hours at temperatures of 1600°F and above. Therefore, the Rene 100/CODEP B system is not suitable for application where conditions that cause hot corrosion attack are present.

Conclusions

1. The oxidation behavior of Rene 100 was poor compared to that of the X-40 alloy, especially at temperatures above 1900°F.

2. The superalloy X-40 was found to be more resistant to the initiation of hot corrosion than Rene 100. Both alloys, however, eventually undergo attack at rates consistent with the alloy-induced acidic fluxing mechanism.

3. Both alloys degrade by similar mechanisms. During the initiation stage, sulfides are formed within the alloys which eventually become preferentially oxidized. Such preferential oxidation of sulfide results in more rapid oxidation, and also in the formation of refractory metal oxides involving the elements tungsten, molybdenum, and vanadium. Reaction of the refractory metal oxides with Na_2SO_4 leads to the development of acidic melts that cause catastrophic degradation of these two alloys.

4. Exposure at temperatures as low as 1500°F shortened the initiation stage at higher temperatures. Sulfides were formed at the lower temperature and they rapidly oxidized at the higher temperatures.

5. Repeated applications of Na_2SO_4 resulted in the initiation of hot corrosion attack after shorter exposure times than for single applications.

6. An aluminide coating, CODEP B, on Rene 100 caused the initiation of hot corrosion to be delayed. Attack did occur, however, when cumulative applications of Na_2SO_4 were used. The coatings were penetrated by the Na_2SO_4 in localized areas after exposure times of less than 200 hours.

7. The test which has been used induces degradation for a specific set of conditions and, therefore, care must be exercised in extrapolating the results to other conditions. This test has validity for hot corrosion mechanisms whereby the degradation process is sustained by reaction products from the alloy. It, therefore, can be used to compare the time to establish such conditions in alloys. Rene 100 is more susceptible to this condition than X-40. When alloys do not undergo alloy-induced acidic fluxing, gas composition, deposit composition, and amount of the deposit play very important roles in the amount and type of degradation that occurs. The hot corrosion test used in this report would not be appropriate under those conditions.

The alloy X-40 is more resistant to hot corrosion than Rene 100, probably due to its higher chromium concentration and the cracking of the oxide scale on the Rene 100 alloy. Both alloys, however, exhibit features that show the propagation mode of hot corrosion has been reached after relatively short initiation stages; this is especially the case at temperatures above 1800°F. It is, therefore, apparent that these alloys should not be used uncoated under conditions where hot corrosion attack can be expected.

Hot Corrosion of Coated Rene 100

Since Rene 100 was so susceptible to hot corrosion, specimens of Rene 100 were aluminized by using the CODEP B process. A scanning electron photomicrograph of the surface of a coated specimen is presented in Figure 13. Several nodules are evident above the surface of the coating. These nodules appear to be small clusters of pack powder. X-ray diffraction analysis confirmed this coating to be nickel-aluminide (NiAl).

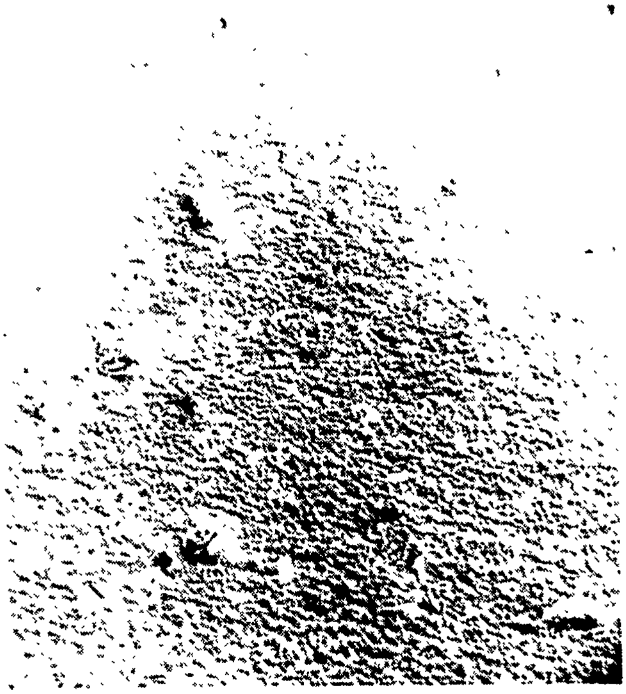


Figure 13. As-coated Rene 100/CODEP B system. XRDA shows nickel aluminide, NiAl nodules, and small clusters of pack powder. Mag. 45X

Hot corrosion of CODEP B-coated Rene 100 was examined at 1600°F and 1800°F using single, as well as multiple, additions of Na_2SO_4 and the second procedure for heating the specimens. Only single applications of Na_2SO_4 were used at 2200°F.

At 1600°F both single and multiple additions of Na_2SO_4 resulted in visible coating failure after 50 hours at temperature. The attack usually was such that the coating was penetrated in localized areas (see Figure 14). After penetration of the coating substrate attack became evident as indicated by large amounts of corrosion products (see Figure 15).

Figure 14. Hot corrosion of Rene 100/CODEP B system after two applications of Na_2SO_4 for 50 hours at 1600°F. Coating failure in localized areas. Mag. 7X

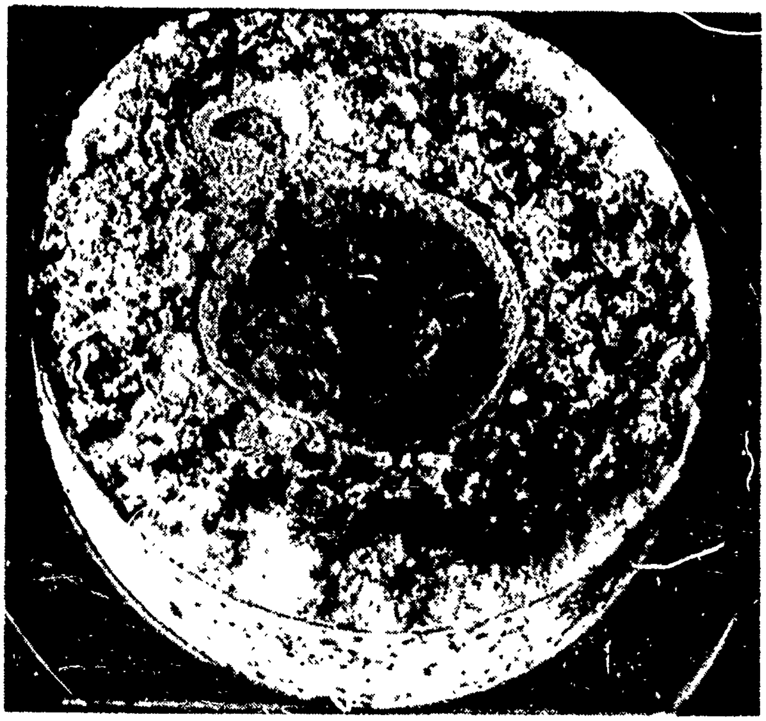
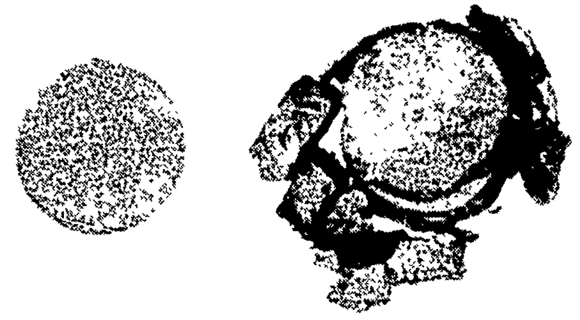


Figure 15. Hot corrosion of Rene 100/CODEP B system after 900 hours at 1600°F Large amounts of substrate corrosion products. Mag 7X

At 1800°F the coated specimen with one application of Na_2SO_4 was terminated after 250 hours. No visual evidence of failure was apparent and only a small weight gain was observed (2.4 mg/cm^2). When cumulative additions of Na_2SO_4 were used at 25-hour intervals, visual evidence of penetration of the coating became apparent after

175 hours. The localized nature of the attack was not as obvious as at 1600°F. Total failure of the coating occurred after 200 hours of exposure. Photographs comparing the degradation of coated Rene 100 in tests using single and multiple applications of Na_2SO_4 are presented in Figures 16a and 16b, respectively.

Figure 16. Hot corrosion of Rene 100/CODEP B system at 1800°F. Mag. 2X



Single Application of Na_2SO_4 **Multiple Applications of Na_2SO_4**

a b

At 2200°F, the coated system was exposed for 35 hours with one application of Na_2SO_4. The nodules observed in the as-coated condition became covered with a dark blue product (see Figure 17). It appeared that coating failure was initiating at these sites.

Figure 17 Hot corrosion of Rene 100/CODEP B system after 35 hours at 2200°F Single application of Na_2SO_4 Mag 7X

The limited coating test results show that penetration of the coating occurs after exposure periods of less than 200 hours at temperatures of 1600°F and above. Therefore, the Rene 100/CODEP B system is not suitable for application where conditions that cause hot corrosion attack are present.

Conclusions

1. The oxidation behavior of Rene 100 was poor compared to that of the X-40 alloy, especially at temperatures above 1900°F.

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7. The test which has been used induces degradation for a specific set of conditions and, therefore, care must be exercised in extrapolating the results to other conditions. This test has validity for hot corrosion mechanisms whereby the degradation process is sustained by reaction products from the alloy. It, therefore, can be used to compare the time to establish such conditions in alloys. Rene 100 is more susceptible to this condition than X-40. When alloys do not undergo alloy-induced acidic fluxing, gas composition, deposit composition, and amount of the deposit play very important roles in the amount and type of degradation that occurs. The hot corrosion test used in this report would not be appropriate under those conditions.

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