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AN EVALUATION OF THE CORROSION RESISTANCE
OF SEVERAL HIGH TEMPERATURE ALLOYS
FOR NUCLEAR APPLICATIONS

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AN EVALUATION OF THE CORROSION RESISTANCE
OF SEVERAL HIGH TEMPERATURE ALLOYS
FOR NUCLEAR APPLICATIONS

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November, 1965

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RICHLAND, WASHINGTON

AN EVALUATION OF THE CORROSION RESISTANCE OF SEVERAL HIGH TEMPERATURE ALLOYS FOR NUCLEAR APPLICATIONS

INTRODUCTION

The improvement of thermal operating efficiency of nuclear power reactors makes it necessary to increase their operating temperatures substantially. This places severe limitations in terms of allowable stress and corrosion resistance on many conventional materials now used in pressurized and boiling water reactors. Since nuclear superheat and gas cooled reactors will operate at higher temperatures than either the pressurized or boiling water reactors, new materials must be evaluated for their resistance to corrosive environments and irradiation damage.

The Pacific Northwest Laboratory has been studying the effects of reactor environments upon the properties of reactor structural materials for many years.^(1, 2) As a result of these studies, efforts have been directed toward the selection of alloys whose properties are favorable for application in high-temperature advanced reactors. As part of the alloy screening procedure, studies have been made on several precipitation- and solution-hardenable, high-temperature materials to determine their oxidation resistance in various environments. Further tests are being made on selected alloys to determine the effects of neutron irradiation on their mechanical properties.

SUMMARY AND CONCLUSIONS

The results of these investigations have shown comparatively the oxidation resistance of 10 high-temperature alloys in an oxidizing environment of helium and water vapor. In addition, it has been found that large weight gains are primarily associated with intergranular corrosion and internal oxidation of the alloy. From the data obtained in these investigations, the following general statements can be made.

At 815 °C in an atmosphere of low pressure water vapor, Hastelloy N, Hastelloy X-280, Inconel 600, Inconel 625, and Haynes 25 have more superior corrosion resistance than the other four materials tested. Weight

gains of less than 0.3 mg/cm^2 were observed for these better materials after 300 hr of exposure. Inconel 718, Inconel 702, and Incoloy 800 were more resistant to corrosion than Hastelloy R-235, the least corrosion resistant of all the alloys tested at this temperature.

At 930°C , the same relative grouping was found as in the 815°C data. However, more definite evidence of intergranular attack was found in Haynes 25, Inconel 718, and Inconel 600 at 930°C than at the 815°C test temperature.

At 1038°C , definite and gross intergranular attack and internal oxidation was found in alloys. Hastelloy C, Hastelloy N, Hastelloy X-280, and Haynes 25 exhibit the best resistance to oxidation, and Inconel 600, Inconel 702, Incoloy 800, and Inconel 625 followed in order. At all test temperatures, Hastelloy R-235 showed the worst attack. Inconel 625 exhibited increasing amounts of grain boundary attack and subsequent internal oxidation with increasing test temperature.

For those alloys showing susceptibility to grain boundary attack, careful evaluation of their application to reactor components should be made; especially where such application is for components of thin cross section such as fuel cladding.

MATERIALS

In recent years many alloys have been developed to withstand the effect of high temperature environments. Several nickel-base and cobalt-base precipitation and solution hardenable alloys have been developed to meet the needs of the missile and space, gas turbine, petroleum, and power generation industries. With the nuclear industry demanding higher temperature application of materials, it was felt that materials developed for other industries should be considered before beginning a separate extensive alloy development program.

In addition to an extensive literature survey, contacts were made with several leading materials manufacturers concerning selection of alloys for nuclear application. Materials were selected on the basis of known mechanical properties, phase stability, oxidation and corrosion resistance, fabricability, and availability.

The 10 materials selected for this initial study are listed together with their chemical composition in Table I. Nine of the 10 alloys are classed as nickel-base alloys. The one remaining, Haynes 25, is a cobalt-base alloy selected as a candidate material for use in the out-of-reactor sections of the Advanced Test Reactor (ATR), High Temperature Gas Loop Facility. This facility will be cooled by high temperature (1090 °C), purified, helium gas and will serve as a materials and fuel irradiation test facility.

TABLE I
CHEMICAL COMPOSITION OF NICKEL- AND IRON-BASE ALLOYS
USED FOR OXIDATION AND CORROSION STUDIES

Alloy	Composition, wt%												
	Cr	Fe	C	Si	Co	Ni	Mn	Mo	Al	Ti	W	Cb	V
Inconel 600	15.46	6.72	0.05	0.28	0.07	Bal	0.32		0.18				
Inconel 625	22.0	3.0	0.05	0.30		61.0	0.15	9.0				4.0	
Inconel 702	15.47	0.50	0.04	0.18		79.73	0.02		3.29	0.69			
Inconel 718	20.0	Bal	0.10	0.75		52.0	0.5	3.0	1.0	1.3		5.0	
Incoloy 800	20.36	47.55	0.04	0.24		30.45	0.90						
Hastelloy C	15.52	5.84	0.07	0.06	2.10	Bal	0.63	15.66			3.54		0.24
Hastelloy N	6.42	3.93	0.07	0.56	0.12	Bal	0.52	16.22			0.06		0.23
Hastelloy R-235	15.47	9.97	0.14	0.08	0.78	Bal	0.04	5.46	2.10	2.59			
Hastelloy X-280	21.73	18.66		0.53	0.11	Bal	0.75	8.65			0.38		
Haynes Alloy 25	19.93	2.47	0.07	0.52	Bal	10.42	1.44				14.91		

Several articles have been written about the physical metallurgy of the materials selected. (3, 4) In general, strength and ductility are achieved by solid solution strengthening or by precipitation strengthening with precipitation of MC, $M_{23}C_{61}$ or γ' phases in the matrix and along grain boundaries. Even in the solution hardenable alloys, such as Hastelloy X-280 and Hastelloy C, precipitates of the M_6C or M_6C' types are found. Nickel base and cobalt base alloys are similar to the austenitic stainless steels in that they are face-centered cubic in structure and generally nonmagnetic.

Microstructures in these alloys are generally quite complicated and usually not fully understood. The solution hardenable alloys Inconel 600, Incoloy 800, Hastelloy X-280, and Hastelloy N are of the simplest variety, and they generally precipitate the M_6C , M_6C' and $M_{23}C_6$ phases in the

650 to 930 °C temperature range. This creates a ductility minimum, which occurs (in most instances) between 730 to 815 °C. Hastelloy C is more complicated; in addition to M_6C , the (Ni_7Mo_6) intermetallic phase precipitates in the 650 to 980 °C range. Heat treating at 1120 °C, followed by air cooling, spheroidizes the precipitates and improves the high temperature corrosion properties.⁽³⁾

Inconel 625, recently developed by the International Nickel Company, Inc., is a "matrix stiffened" alloy containing additions of molybdenum and columbium. Although improved high temperature stress rupture properties are obtained by the columbium and molybdenum additions, the alloy is thermally unstable and will precipitate Ni_3Nb and $M_{23}C_6$ phases.⁽⁴⁾ One advantage of this alloy is the apparent lack of a ductility decrease in the 730 to 815 °C temperature range.

The phase precipitation of the alloys Inconel 702, Inconel 718, and Hastelloy R-235 are dependent upon chemical composition and heat treatment. Inconel 702 and Hastelloy R-235, which contain aluminum and titanium, are strengthened primarily by the precipitation of the $Ni_3(Al, Ti)$ gamma prime phase. Other phases such as $M_{23}C_6$ and M_6C are also present and can be responsible for weld cracking during fabrication. Therefore, heat treatments subsequent to fabrication are necessary.

The heat treatment and chemical composition, specifically the columbium, aluminum, titanium, and carbon, are especially important in developing the properties of Inconel 718.⁽⁵⁾ Variation in these elements can cause the formation and/or solution of the Laves phase, Ni_3Nb and gamma prime strengthening phases, and other microconstituents such as the A_2B , M_6C , or $Nb(C, N)$ phases.

The Haynes 25 alloy is a solid, solution-strengthened, cobalt-base material. Similar to other such alloys, it is known to precipitate M_2W and M_6C intermetallic compounds at 650 to 1050 °C. Its primary advantage, in addition to its relative high strength and corrosion resistance at temperatures to 1100 °C, is its ability to be used in the as-welded condition without subsequent heat treatments. This ability is especially important where field welds are required on large components.

EXPERIMENTAL

The corrosion tests reported herein should be thought of as screening tests for 10 candidate alloys exposed to a corrodant (i. e., low-pressure water vapor) only approximating the environment to which they will be exposed in high-temperature nuclear applications. It was felt that high-temperature screening of candidate alloys for reactor applications would be more meaningful if water vapor were used as the screening corrodant rather than the more commonly used air or oxygen. In many systems, high temperature water or steam will be the corrosive environment. If materials are to be chosen for a reactor system utilizing a coolant other than water or steam (e. g., CO_2 , N_2 , air), the results of the present tests may be only roughly applicable.

Specimen Preparation

Specimens were prepared from a 0.125-in. sheet of each alloy listed in Table I. Specimens of these materials were coupons (0.5 x 1.5 x 0.040 in. in thickness) formed from the sheet material by careful grinding with an abrasive wheel. Final surface finish of the coupons was prepared by light polishing on 150 grit paper. Prior to testing, each coupon was identified, washed, rinsed in dry acetone and alcohol, and weighed. The specimens were mounted upon a quartz rod (Figure 1) for testing.

Test Procedure

The specimens were exposed to an environment of 15 torr, partial pressure of water vapor in Grade A helium. The oxidation, as measured by weight changes, was determined for specimens exposed at 815, 930, and 1038 °C for 100, 200, and 300 hr respectively. The environment was provided by metering the water vapor-helium mixture into a tube furnace at a rate of 1 ft³/hr. This flow rate corresponds to a linear gas velocity past the specimens of about 2 cm/sec.

Three specimens of each material were inserted into the furnace for each temperature condition. To eliminate erroneous data due to gettering of the oxidizing environment at the test section beginning, the specimens were staggered down the length of the test section. At the end of 100 hr, all three specimens were weighed, one retained for metallography, and two reinserted

FIGURE 1
Test Assembly for Determination of Oxidation Resistance
of High Temperature Materials

into the furnace for an additional 100 hr. At the end of this second 100 hr test period, both of the remaining specimens were weighed, one sectioned for metallography, and one replaced for the last 100 hr exposure. After a total of 300 hr exposure, the last remaining specimen was removed, weighed, and prepared for metallography.

Each test coupon was sectioned, polished, and examined in both the unetched and etched condition by standard metallographic procedures to determine the type and depth of maximum and average penetration of the corrosion attack. This information, together with the change-in-weight data, was used to evaluate the relative corrosion resistance of the alloys.

RESULTS OF THE INVESTIGATION

The numerical results from this investigation are given in Table II and are shown graphically in Figures 2, 3, and 4. For specimens exposed at 815 °C (Figure 2), Hastelloy N, Hastelloy X-280, Hastelloy C, Inconel 600, Haynes 25, and Inconel 625 showed the least amount of weight gain, less than 0.3 mg/cm^2 after exposure for 300 hr. Incoloy 800, Inconel 718, and Inconel 702 gained from 0.4 to 0.55 mg/cm after the same length of exposure. The alloy with the greatest weight gain was Hastelloy R-235 at 0.96 mg/cm^2 .

Specimens tested at 930 °C for 100, 200, and 300 hr showed similar results, as at 815 °C (Figure 3). Hastelloy R-235 experienced almost twice the weight gain of any other alloy tested (3.87 mg/cm^2 after an exposure of 300 hr). Inconel 702, Inconel 718, and Incoloy 800 were found to have gained 1.10, 1.62, 1.46 mg/cm^2 , respectively, after 300 hr. As in the 815 °C exposures, the remaining alloys at 930 °C showed the lowest gain in weight (about 1 mg/cm^2 or less).

Some significant differences occurred at the 1038 °C exposure (Figure 4). While Hastelloy R-235 showed an extremely high weight gain, the Inconel 718 showed substantial weight loss. As shown in Table II, these losses amounted to 3.80, 5.07, and 13.84 mg/cm^2 after 100, 200, and 300 hr, respectively. However, specimens of Inconel 625 showed a higher increase in weight gain. Consistent with the test results at 815 and 930 °C, Hastelloy C, Hastelloy N, Hastelloy X-280, and Haynes 25 showed excellent resistance to the test environment.

TABLE II
AVERAGE WEIGHT GAINS FOR ALLOYS EXPOSED TO 15 TORR WATER VAPOR
AT 815, 1030, AND 1150 °C FOR 100, 200, AND 300 hr

Alloy	Test Temperature, °C	Time, hr	Average Weight Change, mg/cm ²	Alloy	Test Temperature, °C	Time, hr	Average Weight Change, mg/cm ²
Inconel 600	815	100	+ 0.1204	Hastelloy C	815	100	+ 0.1029
		200	+ 0.1617			200	+ 0.1194
		300	+ 0.1782			300	+ 0.1382
	930	100	+ 0.3464		930	100	+ 0.2750
		200	+ 0.4952			200	+ 0.3792
		300	+ 0.5552			300	+ 0.4592
	1038	100	+ 0.9433		1038	100	+ 0.5783
		200	+ 1.2931			200	+ 0.6629
		300	+ 1.4600			300	+ 0.7010
Inconel 625	815	100	+ 0.1650	Hastelloy N	815	100	+ 0.0831
		200	+ 0.2244			200	+ 0.1023
		300	+ 0.2660			300	+ 0.1087
	930	100	+ 0.4882		930	100	+ 0.2570
		200	+ 0.8790			200	+ 0.2262
		300	+ 1.1004			300	+ 0.3158
	1038	100	+ 2.4905		1038	100	+ 0.5276
		200	+ 3.3270			200	+ 0.6205
		300	+ 4.196			300	+ 0.7650
Inconel 702	815	100	+ 0.2561	Hastelloy R-235	815	100	+ 0.7130
		200	+ 0.3838			200	+ 0.8013
		300	+ 0.4896			300	+ 0.9643
	930	100	+ 1.1436		930	100	+ 2.1750
		200	+ 1.5732			200	+ 3.0357
		300	+ 1.8232			300	+ 3.5578
	1038	100	+ 1.7439		1038	100	+ 3.8658
		200	+ 1.9241			200	+ 4.7467
		300	+ 1.7900			300	+ 6.9150
Inconel 718	815	100	+ 0.2869	Hastelloy X-28	815	100	+ 0.0840
		200	+ 0.3832			200	+ 0.0993
		300	+ 0.4013			300	+ 0.0995
	930	100	+ 0.9886		930	100	+ 0.3025
		200	+ 1.3872			200	+ 0.4191
		300	+ 1.6162			300	+ 0.4325
	1038	100	- 3.7996		1038	100	+ 0.6701
		200	- 5.0674			200	+ 0.9012
		300	- 13.8400			300	+ 0.908
Incoloy 800	815	100	+ 0.3478	Haynes 25	815	100	+ 0.1798
		200	+ 0.4887			200	+ 0.2147
		300	+ 0.5327			300	+ 0.2461
	930	100	+ 0.8788		930	100	+ 0.5122
		200	+ 1.2563			200	+ 0.7398
		300	+ 1.4634			300	+ 0.7506
	1038	100	+ 1.5598		1038	100	+ 0.8255
		200	+ 1.8669			200	+ 0.9939
		300	+ 2.3700			300	+ 1.0470

FIGURE 2

Oxidation of High Temperature Alloys Exposed to 15 Torr Water Vapor in Helium Flowing at 1 ft³/min at 815 °C

FIGURE 3

Oxidation of High Temperature Alloys Exposed to 15 Torr Water Vapor
in Helium Flowing at 1 ft³/min at 930 °C

FIGURE 4

Oxidation of High Temperature Alloys Exposed to 15 Torr Water Vapor
in Helium Flowing at 1 ft³/min at 1038 °C

Although weight gain data show in a quantitative manner the relative oxidation resistance of the group of alloys, the degree of penetration of the corrosion attack, including intergranular penetration and subscale formation, is more useful in determining the actual damage incurred. Also, metallographic examination aids in determining the type of oxide film formed and the mode of attack by the environment.

Figures 5 through 14 show typical surface attacks for each alloy under each test condition. It is significant to note that those alloys that show good oxidation resistance: namely, Hastelloy C, Hastelloy N, Hastelloy X-280, and Haynes 25, form a tight adherent oxide film on the surface of the specimen with little or no grain boundary penetration or internal oxidation. This is especially true for Hastelloy X-280, Hastelloy C, and Hastelloy N, (Figures 13, 10, and 11, respectively). In Figure 14, some internal oxidation in the Haynes 25 structure can be seen after the exposure at 1038 °C.

For alloys showing higher weight gains, a significant amount of intergranular attack and internal oxidation was found. These alloys include Inconel 600, Inconel 625, Inconel 702, Inconel 718, and Incoloy 800. The intergranular mode of attack can be clearly seen in Figures 5 through 9. Each of these alloys exhibited a tight, adherent film after exposure, along with a penetrating intergranular attack. In some cases, as for Inconel 718 exposed at 1038 °C, the attack spread parallel to the surface after penetration (Figure 8).

The depth of penetration for Inconel 702 (Figure 7) was about the same for each exposure temperature. After exposure at 815 °C, a film was formed on the specimen surface with some grain boundary penetration. Photomicrographs of the specimens after exposure at 930 °C showed an increase in film thickness, thickening of the grain boundary reaction product, and also attack parallel to the specimen surface. After 1030 °C exposure, additional oxidation of the material between the film and region of former parallel attack can be seen. This type of behavior is thought to be the result of selective oxidation, caused by a variation of chemical composition between the matrix and grain boundary in the alloy.

The attack on Hastelloy R-235 is shown in Figure 12. The high weight gains that occur for every test condition are no doubt due to the severe intergranular attack (Figure 12). This type of oxidation attack can be observed even after 100 hr at 815 °C. Deep penetration also occurred at the 930 and 1038 °C test conditions. The oxidation attack spread from the grain boundary into the material matrix after penetration has occurred.

FIGURE 5

Oxidation of Inconel 600 in 15 Torr Water Vapor
(500X)

FIGURE 6

Oxidation of Inconel 625 in 15 Torr Water Vapor
(500X)

FIGURE 7

Oxidation of Inconel 702 in 15 Torr Water Vapor
(500X)

Neg. 0652478-8

FIGURE 8

Oxidation of Inconel 718 in 15 Torr Water Vapor
(500X)

Neg. 0652478-6

FIGURE 9

Oxidation of Incoloy 800 in 15 Torr Water Vapor
(500X)

FIGURE 10

Oxidation of Hastelloy C in 15 Torr Water Vapor
(500X)

FIGURE 11

Oxidation of Hastelloy N in 15 Torr Water Vapor
(500X)

Neg. 0652478-7

FIGURE 12

Oxidation of Hastelloy R-235 in 15 Torr Water Vapor
(500X)

FIGURE 13

Oxidation of Hastelloy X-280 in 15 Torr Water Vapor
(500X)

Neg. 0652478-3

FIGURE 14

Oxidation of Haynes 25 in 15 Torr Water Vapor
(500X)

DISCUSSION

The data described in this report indicate that the observed oxidation rates follow a parabolic rate law. While the data were not sufficient to substantiate this observation, other investigators have found that a parabolic law does exist for the same and similar alloys tested in air.^(6, 7, 8) One additional substantiation of a parabolic rate of formation was the tight adherent oxide layer formed upon the tested specimens. No definite evidence of oxide spalling was found in any of the materials. Even the materials that underwent an overall weight loss exhibited smooth, uniform, oxide surfaces.

Examination of the photomicrographs for several alloys reveals an absence of precipitate and a change of appearance in the region directly adjacent to the oxide layer on the surface. These changes may indicate that some alloy depletion has occurred in these materials because of the oxidation. Such alloy depletion has been observed before^(6, 7) and usually precedes the formation of internal oxidation product formation. Other investigators⁽⁷⁾ have determined the kinetics of internal oxidation and found it to follow a parabolic rate of growth.

In addition to oxidation processes, the weight change data may reflect the effects of volatilization of high temperature alloys in flowing gas at elevated temperatures. The weight loss of Inconel 718 exposed to the 1038 °C test environment may be caused by oxide volatilization. Also, the decrease in weight gain of the Inconel 702 may also reflect this phenomena of oxide volatilization. Haeckl (in his work on Inconel 702) found definite evidence of volatilization from specimens exposed to air at 1093 °C.⁽⁶⁾

In this study, attempts were not made to determine the kinetics of the oxidation processes observed. Instead, a comparison of oxidation resistance was made for the materials exposed to a common oxidizing environment, and high temperature water vapor was used as an oxidant. Further investigation should be made to determine not only the oxidation kinetics and products formed from these alloys but also the effects of the oxidation upon the mechanical properties of the materials; in particular, the creep, stress to rupture, and cyclic-fatigue properties.

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