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**ADVANCED GAS-COOLED NUCLEAR REACTOR
MATERIALS EVALUATION AND DEVELOPMENT
PROGRAM: CORROSION BEHAVIOR OF
EXPERIMENTAL ALLOYS IN CONTROLLED
PURITY HELIUM AT TEMPERATURES IN THE 750-1050°C RANGE**

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Advanced Gas-Cooled Nuclear Reactor Materials
Evaluation and Development Program:

Corrosion Behavior of Experimental Alloys in Controlled
Purity Helium at Temperatures in the 750-1050°C Range

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ABSTRACT

A series of ten experimental alloys (basically Ni-20Cr with addition of one or more of the elements Al, Ti, Si, Nb and Y) has been examined after exposure to controlled purity helium for periods of 1000-6000 hours at temperatures of 750-1050°C. Alloys containing aluminum were particularly susceptible to internal oxidation at the lower temperatures, but at 950°C and above carburization became the dominant corrosive mechanism. The most corrosion resistant alloys were Ni-20Cr and Ni-Cr containing Si, Ti and Nb. The presence of small amounts of yttrium dramatically promoted the occurrence of carburization, even at temperatures as low as 850°C

1. INTRODUCTION

The Department of Energy-sponsored Advanced Gas-Cooled Nuclear Reactor Materials Evaluation and Development Program, managed at General Electric by the Advanced Energy Programs Department forms part of a larger effort designed to utilize the high-temperature gas-cooled reactor (HTGR) as a source of process heat as well as for electrical power. The main focus of the current program is to provide a technical data base on the long-term corrosion resistance, structural stability, and creep properties of potential structural alloys in an environment which simulates the impure helium expected in the HTGR.

In addition to the large number of commercial alloys being evaluated in this program, a group of selected experimental alloys also have been included in the test matrix. The main purpose here is to provide a better understanding of the behavior of the individual elements in complex alloys and to form a basis for a general interpretation of corrosion mechanisms in controlled purity helium. It is hoped that conclusions concerning the effects of individual alloying elements will provide useful guidelines for the future development of new corrosion-resistant alloy compositions.

This report summarizes the results of corrosion tests carried out with the series of experimental alloys at 750, 850, 950 and 1050 C for periods of 1000, 3000 and 6000 hours in the controlled purity helium. Corrosion morphologies produced in these alloys after exposure to the same environment for 1000 hours at 750 and 850 C and for 1000 and 3000 hours at 950 C have been described in previous reports (1,2).

2. EXPERIMENTAL

Ten experimental alloys were evaluated in this part of the program. These alloys, whose nominal compositions are listed in Table I, were basically Ni-20Cr with the addition of one or more of the elements Al, Ti, Si, Nb, and Y.

The alloys were prepared by melting in magnesia crucibles in an argon atmosphere and then casting into 1-in. diameter round bars in copper molds. The metal grades used were 270 nickel, Electrochrome chromium, yttrium chips (Michigan Chemical Co.), niobium chips (EB Grade, Dynamet, Inc.), 99.99% aluminum (Alcoa), titanium sponge (Titanium West Inc.), and silicon (polycrystalline rod, Dow-Corning). The nominal compositions of these materials are listed in Table II. Specimens for the corrosion tests were machined longitudinally from the alloy barstock to give pins 1 in. long x 1/4 in. diameter with a 1/16 in. hole drilled near one end for suspension purposes. The pins were machined using low-stress grinding techniques to produce a surface finish of approximately 16 rms.

The specimens were degreased with alcohol and acetone and then weighed in a Mettler H51AR balance to ± 0.01 mg before mounting in the aging and corrosion (A/C) test stands. The test facility in the High-Temperature Reactor Materials Testing Laboratory (HTRMTL) in Building 28, Schenectady, has been described in detail elsewhere (3). The A/C stands consist of eight vertical furnaces equipped with ceramic retort tubes containing a ceramic support assembly from which the test specimens are suspended. The program requires the exposure of specimens at a series of temperatures between 750 and 1050 C to a helium atmosphere containing controlled levels of hydrogen, water vapor, carbon monoxide, carbon dioxide, methane, and nitrogen in the parts-per-million range. The average composition of the atmosphere used in these experiments is shown in Table III. The gas environment is intended to simulate the primary coolant chemistry in a gas turbine high-temperature gas-cooled reactor (HTGR) or a process heat HTGR containing an intermediate heat exchanger.

Following the exposure of the specimens to the controlled purity helium environment at 750-1050°C for 1000-6000 hours, the specimens were cooled, removed from the furnaces, reweighed, and then examined by standard metallographic and electron microprobe techniques to determine the extent and nature of

the corrosive effects. For this purpose photomicrographs in the as-polished condition and after etching with Murakami's reagent (specific for carbides) were prepared for each exposed alloy specimen. Qualitative x-ray maps for the most important metallic components and for carbon were made across polished sections in the vicinity of the surface of each mounted specimen. In addition, a 2-mm thick slice of each exposed pin specimen was subjected to combustion analysis to determine the bulk carbon content.

3. RESULTS AND DISCUSSION

750°C Corrosion Morphology

Weight gains and bulk carbon contents following exposure of the series of experimental alloys to controlled purity helium at 750°C for 3000 and 6000 hours are summarized in Table IV. After the 3000 hour test, most of the alloy specimens showed weight gains similar to, or slightly larger than, those observed previously for 1000 hour exposures at this temperature (1) and carbon contents remained low (0.01-0.06%). The Ni-20Cr-8Al alloy showed localized regions of internal oxidation (Figure 1) with Al-rich filamentous particles (presumably oxide) beneath the surface, but no evidence of carbide precipitation. More uniform corrosion was exhibited by the Ni-20Cr-2Al-8Ti alloy (Figure 2), with a zone of internal oxidation extending to a depth of 20 μ m beneath the surface scale. Microprobe analysis of the surface region (Figure 3) revealed an outer Cr, Ti-rich scale and a zone of internal Al oxide beneath. All other alloys showed minimal evidence of corrosion.

Following the 6000 hour exposure at 750°C evidence for corrosion was noted in several of the alloy specimens. Although Ni-20Cr, Ni-20Cr-2Si, Ni-20Cr-5Si, Ni-20Cr-2Nb and Ni-20Cr-1Y showed little change, Ni-50Cr suffered a substantial weight gain with the development of a thick Cr-rich surface scale (Figure 4). The Ni-20Cr-8Al alloy (Figure 5) exhibited a band of Al-rich internal oxide 50 μ m thick beneath the surface, but showed little evidence of carbide formation. Ni-20Cr-5Ti (Figure 6) had developed a thick (15 μ m) Ti, Cr-rich scale, but no discrete carbide particles. Ni-20Cr-2Al-8Ti (Figure 7) showed a zone of internal oxidation, which microprobe analysis (Figure 8) revealed to consist of a Ti, Cr-rich surface scale

with an Al-rich band beneath extending to a depth of 20 μ m. A few carbide particles may have been present beneath the zone of internal oxidation. The Ni-20Cr-8Al-2Ti alloy, however, was more resistant and showed little evidence of corrosion (Figure 9).

850°C Corrosion Morphology

Weight changes and bulk carbon contents observed after exposure of the alloys to controlled purity helium for 3000 hours at 850 C are summarized in Table V. As expected, weight changes were more marked than at 750 C, but carbon contents remained small (0.01-.08%). Most alloys showed identifiable corrosion effects, which were however confined to the growth of a coherent scale in the case of Ni-20Cr (Figure 10), Ni-20Cr-2Nb and Ni-20Cr-1Y. The Ni-Cr-Si alloys gave spalling scales which resulted in net weight losses. Ni-50Cr developed a thick Cr-rich scale (Figure 11) with evidence of carbide particles beneath (Figure 12). Ni-20Cr-8Al (Figure 13) exhibited a broad band of Al internal oxidation 100 μ m thick (Figure 14), beneath which a band of Cr carbide particles extended 180 μ m into the specimen. The carbon content of this alloy was the highest of the series following the 850 C/3000 hour test. The Ni-20Cr-5Ti alloy, on the other hand (Figure 15), had developed a thick Ti, Cr-rich scale, with no evidence of carbide particles beneath (Figure 16). Ni-20Cr-2Al-8Ti (Figure 17) showed a complex corrosion morphology with an outer Ti, Cr-rich scale (Figure 18b,d), a zone of Al-internal oxidation (Figure 18c) 50 μ m thick and fine Cr-rich particles beneath, which could not be clearly identified as carbides (Figure 18 d,e). Ni-20Cr-8Al-2Ti (Figure 19) showed a similar banded morphology, but the Al-rich region (Figure 20b) was thinner in this case and the Cr-rich particles were clearly identified as carbides (Figure 20c,e).

950°C Corrosion Morphology

Corrosion effects produced in the experimental alloy series following exposure to controlled purity helium at 950 C for 1000 and 3000 hours have been described previously (2). Results obtained following 6000 hour exposure at this temperature were consistent with, but generally more pronounced than, those observed at shorter exposure times. Table VI summarizes weight gains and bulk

carbon contents following the 6000 hour test. Ni-20Cr, Ni-20Cr-2Si, Ni-20Cr-5Si, Ni-20Cr-5Ti and Ni-20Cr-2Nb proved again to be quite resistant, whereas Ni-20Cr-8Al and Ni-20Cr-1Y showed very large weight gains and associated carbon contents. Ni-50Cr (Figure 21) showed a substantial weight gain and an appreciable carbon content after the 6000 hour exposure. Microprobe analysis (Figure 22) indicated the absence of a well-defined Cr oxide scale and the presence of carbide particles beneath the surface. Ni-20Cr-8Al showed the largest weight gain and the highest bulk carbon content of the series. A heavily corroded zone had developed near the surface of the specimen (Figure 23). Beneath the outer Cr-rich scale (Figure 24c), a band of elongated Al oxide particles penetrated 85 μ m into the alloy (Figure 24c,d) and a lower band of carbide particles 150 μ m wide was also observed. The Ni-Cr-Al-Ti alloys (Figure 25) also showed a subsurface zone of Al internal oxidation but the distribution of carbide particles was more diffuse with these materials. Also the bulk carbon content was an order of magnitude less with these alloys than with Ni-20Cr-8Al. Finally the Ni-20Cr-1Y, which showed a large weight gain and carbon content, exhibited a diffuse distribution of carbon which did not appear to correlate with the filiform intergranular distribution of Y-rich particles (Figure 26c,d).

1050°C Corrosion Morphology

Exposure to controlled purity helium at 1050°C produced marked corrosive degradation of all the experimental alloy samples, although the extent of carburization (the dominant effect at this temperature) varied considerably among the different specimens. Weight changes and bulk carbon contents after 1000, 3000 and 6000 hours exposure are summarized in Table VII. In general, weight gains correlated well with carbon contents, although Ni-50Cr and Ni-20Cr-5Ti developed flaking scales and the specimens actually lost weight after prolonged exposure. Carbon contents of 1 percent or more were observed for the Ni-20Cr-1Y alloy for exposure times of 1000-6000 hours. Of all the materials tested, this alloy showed the greatest susceptibility to carburization at the higher temperatures. The increase in bulk carbon content with time of exposure at 1050 C is illustrated in Figure 27 for several of the experimental alloy specimens.

(i) 1000 hour exposure

Ni-20Cr showed only minimal corrosion following 1000 hours exposure at 1050 C and the adherent Cr oxide scale remained protective with no evidence of internal carbide formation. On the other hand, Ni-50Cr (Figure 28) failed to develop a protective scale and large blocky Cr carbide precipitates were observed throughout the specimen (Figure 29). In contrast to the results obtained at lower temperatures, the Ni-20Cr-8Al alloy (Figure 30) at 1050 C developed only a thin zone of Al oxide beneath the fractured Cr-rich surface scale (Figure 31b,c). Beneath a subsurface depletion zone, blocky Cr carbide precipitates were however widely distributed throughout the specimen thickness (Figure 31b,d). Similar the Ni-Cr-Al-Ti alloys (Figure 32) showed only a narrow band of Al oxide (Figure 33c) and blocky Cr carbide precipitates widely distributed in the sub-surface region (Figure 33b,d). Ni-20Cr-5Ti, on the other hand, showed only a thin Ti-rich scale (figure 34) which apparently had not been penetrated by carbon as the bulk carbon content was very low (0.024%) and no discrete carbide particles could be detected. Ni-20Cr-2Nb was likewise resistant to corrosion under these conditions.

At lower temperatures the Ni-Cr-Si alloys have been found to be quite resistant to the corrosive effects of controlled purity helium. However, at 1050 C, carburization of these alloys became significant. Figure 35 shows carbide precipitates along grain boundaries in Ni-20Cr-2Si after exposure at 1050 C for 1000 hours. As shown in Figure 36 these intergranular carbides were observable throughout the specimen thickness and microprobe analysis showed them to be Cr carbide rather than Si carbide. Ni-20Cr-5Si (Figure 37) exhibited similar effects with larger blocky carbide particles distributed throughout the alloy specimen.

The most spectacular carburization effects were observed with the Ni-20Cr-1Y alloy (Figure 38) which at 1050 C showed the greatest weight gains and highest bulk carbon contents of the series. The large blocky Cr carbide particles (Figure 39b,d) were found throughout the specimen cross-section (Figure 39e,f). The distribution of Y in this alloy (Figure 39c) was not apparently associated with the carbide particles.

(ii) 3000 hour exposure

Following the 3000 hour exposure at 1050 C even the resistant Ni-20Cr alloy was heavily carburized (Figures 40-41) with blocky Cr carbide particles fairly uniformly distributed throughout the specimen thickness (Figure 41). With Ni-50Cr the carbide particles were larger in size (Figure 42) and interconnected, especially near the surface of the specimen (Figure 43).

The Ni-20Cr-8Al alloy (Figure 44) had developed a surface zone of mixed Al, Cr oxides containing some carbides (Figure 45b,c,d), beneath which a depletion zone about 1000 μ m deep remained free of carbides. At greater depths (Figure 45e-h) large blocky Cr carbide particles were widely distributed. A somewhat similar corrosion morphology was developed in the Ni-Cr-Al-Ti alloys (Figure 46), although in these materials the surface scale was Ti-rich (Figure 47c), with an Al-rich sub-scale (Figure 47d). Cr carbides (Figure 47b,e) were distributed throughout the specimen thickness, but Ti (Figure 47c) appeared not to be associated with the carbide particles.

Ni-20Cr-5Ti exhibited a net weight loss due to the formation of a spalling scale; however, after 3000 hours exposure, the carbon content was high and carbide particles were uniformly dispersed throughout the sample thickness (Figure 48). In this case, the particles appeared to be Ti carbide (Figure 48b,c) rather than Cr carbide.

The Ni-Cr-Si alloys showed massive carbide formation. Ni-20Cr-2Si (Figure 49) developed both inter- and intragranular Cr carbide precipitates (Figure 50a-c) throughout the sample cross section, and with Ni-20Cr-5Si (Figure 51) the blocky Cr carbide precipitates had coalesced to form continuous arrays of particles, often of bizarre script-like morphology (Figure 52).

As expected from the very large weight gain and carbon content, the Ni-20Cr-1Y alloy (Figure 53) showed massive inter- and intragranular Cr carbide precipitation (Figure 54) throughout the specimen thickness. Finally, Ni-20Cr-2Nb (Figure 55), which was one of the most resistant materials at lower temperatures,

developed a fine dispersion of inter- and intragranular Cr carbide particles throughout the specimen cross-section (Figure 56). The spherical Nb-rich inclusions (Figure 56c) did not appear to be associated with the more elongated carbide particles.

(iii) 6000 hour exposure

Exposure to controlled purity helium at 1050 C for 6000 hours produced massive carbide precipitation in all the experimental alloys investigated. Figure 57 shows Cr carbide particles enveloping grain boundaries and also finer intragranular carbides near the surface of Ni-20Cr. Figure 58 shows the appearance of larger carbide particles near the center of this specimen. With Ni-50Cr (Figure 59) a gradient in carbide particle size from the surface to the interior was observed. Ni-20Cr-8Al (Figure 60) developed a fractured surface scale of mixed Al, Cr oxides and blocky carbide particles throughout the specimen thickness. Carbide precipitates were also widely distributed in the Ni-Cr-Al-Ti alloys (Figures 61-62), in Ni-20Cr-5Ti (Figure 63) and in the Ni-Cr-Si alloys (Figures 64-65). Ni-20Cr-1Y (Figure 66) showed the largest bulk carbon content (1.3%) with large blocky interconnected Cr carbide precipitates throughout the specimen (Figure 67). Ni-20Cr-2Nb (Figure 68) exhibited a fine dispersion of Cr carbide particles precipitated intergranularly as envelopes along grain boundaries.

4. CONCLUSIONS

At 750 and 850°C, several of the experimental alloys investigated were quite resistant to corrosion by controlled purity helium. The best materials were Ni-20Cr, Ni-20Cr-2Si, Ni-20Cr-5Si, Ni-20Cr-5Ti, Ni-20Cr-1Y and Ni-20Cr-2Nb. Ni-20Cr-8Al and the Ni-Cr-Al-Ti alloys were susceptible to internal oxidation of the Al under these conditions, with Ni-20Cr-2Al-8Ti being somewhat inferior to Ni-20Cr-8Al-2Ti.

At the higher temperatures the presence of Y caused a dramatic increase in the susceptibility of the Ni-20Cr alloy to carburization. Thus, above 850 C, the Ni-20Cr-1Y alloy showed a massive uptake of carbon with the formation of Cr carbide precipitates throughout the specimen cross-section.

At 950°C all the alloys showed carbide precipitation to some extent, although Ni-20Cr, Ni-20Cr-2Nb, Ni-20Cr-5Ti and the Ni-Cr-Si alloys were more resistant than the others.

At 1050 C all the alloys were massively carburized within 1000 hours. The onset of carburization as a function of temperature and alloy composition is illustrated in Figure 69 for 3000 hour exposure times. The permeation of carbon into the alloys was apparently inhibited by the alloying elements Nb, Ti and Si and accelerated by Y. However none of the ternary alloys showed a marked improvement over Ni-20Cr which remained one of the best alloy materials at temperatures below 950 C.

5. ACKNOWLEDGMENT

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6. REFERENCES

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2. D. W. McKee, "Advanced Gas-Cooled Nuclear Reactor Materials Evaluation and Development Program: Corrosion Behavior of Experimental Alloys in Controlled Purity Helium at 950 C", General Electric Report, 81CRD036, March 1981.
3. "Advanced Gas-Cooled Nuclear Reactor Materials Evaluation and Development Program Progress Report for Period January 1, 1977-March 31, 1977", C00-2975-10, April 25, 1977.

TABLE I
EXPERIMENTAL ALLOY COMPOSITIONS
(Nominal)

Ni-20Cr
Ni-50Cr
Ni-20Cr-8Al
Ni-20Cr-8Al-2Ti
Ni-20Cr-2Al-8Ti
Ni-20Cr-2Si
Ni-20Cr-5Si
Ni-20Cr-5Ti
Ni-20Cr-1Y
Ni-20Cr-2Nb

Table II

NOMINAL COMPOSITIONS OF ALLOY
CONSTITUTENTS

Ni	C	113 ppm	Cr	< 10 ppm
	Fe	10	S	< 5
	Cu	20	Si	< 10
	Co	10	Al	< 10
	Mn	< 10		
Cr	Si	0.019 wt%	Al	0.007 wt%
	C	0.015	Fe	0.35
	S	0.006		
Y	Yb	10 ppm	Fe	30 ppm
	Er	5	Al	5
	Dy	56	Ca	10
	Tb	38	Ni	75
	La	30	Cu	10
	Si	10	Ta	1000
Nb	Al	< 20 ppm	Ta	191 ppm
	C	< 30	W	18
	Ca	< 20	Zr	< 100
	Cd	< 5	V	< 20
	Co	< 10	Ti	< 40
	Cr	< 20	Sn	< 10
	Cu	< 40	Si	< 50
	Fe	< 50	Pb	< 20
	Hf	< 50	Ni	< 20
	Mg	< 20	Mn	< 20
Al	Si	0.006 wt%		
	Fe	0.002		
	Ga	0.004		
Ti	C	0.013 wt%	Si	0.003 wt%
	N	0.01	O	0.05
	Fe	0.03		
Si	B	< 0.3 ppb		

Table III

CONTROLLED PURITY HELIUM GAS
COMPOSITION

Impurity	Nominal μ -atm	Normal Operating Ranges μ -atm
Hydrogen	400	325-475
Water Vapor	2	1-3
Carbon Monoxide	40	30-50
Carbon Dioxide	0.2	0.05-.35
Methane	20	15-25
Nitrogen	6	2-10
Oxygen	$<10^{-14}$ *	<0.1 **

*Equilibrium Oxygen Level

**Below the level detectable by the loop gas chromatograph ($\leq .1\mu$ -atm)

Table IV

WEIGHT CHANGES AND CARBON CONTENTS OF ALLOYS
AFTER 750°C CORROSION TESTS

Alloy	3000 Hour Exposure		6000 Hour Exposure	
	Wt. change mg/cm ²	C wt%	Wt. change mg/cm ²	C wt%
Ni-20Cr	+0.10	0.026	+0.14	0.039
Ni-50Cr	+0.38	0.060	+1.06	0.041
Ni-20Cr-8Al	+0.63	0.026	+2.92	0.104
Ni-20Cr-8Al-2Ti	+0.06	0.020	+0.31	0.010
Ni-20Cr-2Al-8Ti	+1.31	0.033	+1.92	0.059
Ni-20Cr-2Si	+0.13	0.016	+0.17	0.030
Ni-20Cr-5Si	+0.08	0.029	+0.20	0.026
Ni-20Cr-5Ti	+1.17	0.031	+1.66	0.022
Ni-20Cr-1Y	+0.25	0.047	+0.42	0.036
Ni-20Cr-2Nb	+0.20	0.017	+0.18	0.032

Table V

WEIGHT CHANGES AND CARBON CONTENTS OF ALLOYS

AFTER 850°C CORROSION TESTS

Alloy	<u>3000 Hour Exposure</u>	
	Wt. change mg/cm ²	C wt%
Ni-20Cr	+0.62	0.006
Ni-50Cr	+0.44	0.047
Ni-20Cr-8Al	+5.72	0.077
Ni-20Cr-8Al-2Ti	+2.01	0.048
Ni-20Cr-2Al-8Ti	+3.14	0.042
Ni-20Cr-2Si	-1.38	0.014
Ni-20Cr-5Si	-2.02	0.017
Ni-20Cr-5Ti	+2.67	0.014
Ni-20Cr-1Y	+1.00	0.008
Ni-20Cr-2Nb	+0.57	0.008

Table VI

WEIGHT CHANGES AND CARBON CONTENTS OF ALLOYS

AFTER 950°C CORROSION TESTS

Alloy	6000 Hour Exposure	
	Wt. change mg/cm ²	C wt%
Ni-20Cr	+1.09	0.002
Ni-50Cr	+12.74	0.092
Ni-20Cr-8Al	+17.25	0.898
Ni-20Cr-8Al-2Ti	+1.23	0.061
Ni-20Cr-2Al-8Ti	+0.37	0.044
Ni-20Cr-2Si	+0.60	0.032
Ni-20Cr-5Si	+1.15	0.075
Ni-20Cr-5Ti	+0.69	0.043
Ni-20Cr-1Y	+8.33	0.569
Ni-20Cr-2Nb	+0.48	0.032

Table VII

WEIGHT CHANGES AND CARBON CONTENTS OF ALLOYS AFTER 1050°C CORROSION TESTS

Alloy	<u>1000 Hour Exposure</u>		<u>3000 Hour Exposure</u>		<u>6000 Hour Exposure</u>	
	Wt. change mg/cm ²	C wt. %	Wt. change mg/cm ²	C wt. %	Wt. change mg/cm ²	C wt. %
Ni-20Cr	+1.77	0.067	+5.78	0.420	+7.36	0.551
Ni-50Cr	+0.67	0.076	+0.56	0.305	-0.99	0.359
Ni-20Cr-8Al	+5.85	0.126	+6.11	0.331	+5.73	0.358
Ni-20Cr-8Al-2Ti	+1.04	0.056	+2.35	0.178	+2.92	0.210
Ni-20Cr-2Al-8Ti	+0.22	0.046	+2.24	0.172	+6.74	0.495
Ni-20Cr-2Si	+2.00	0.078	+7.91	0.577	+6.93	0.453
Ni-20Cr-5Si	+1.99	0.083	+10.97	0.771	+2.48	0.215
Ni-20Cr-5Ti	-0.96	0.024	-3.28	0.303	-5.73	0.366
Ni-20Cr-1Y	+11.61	0.916	+22.24	1.40	+27.48	1.3
Ni-20Cr-2Nb	+1.81	0.065	+9.42	0.595	+4.46	0.314

Figure 1 Microstructure of Ni-20Cr-8Al after exposure for 3000 hours at 750 C. Etched 200x

Figure 2 Microstructure of Ni-20Cr-2Al-8Ti after exposure for 3000 hours at 750 C. Etched 200x.

a

b

c

d

Figure 3

Microprobe analysis of Ni-20Cr-2Al-8Ti after 3000 hours at 750 C exposure. (a) Back scattered image, (b) Cr (c) Ti (d) Al. 500x

a

b

c

Figure 4

Microprobe analysis of Ni-50Cr after 6000 hours at 750 C exposure.
(a) Back scattered image, (b) Cr (c) C 500x

a

b

c

d

Figure 5 Microprobe analysis of Ni-22Cr-8Al after 6000 hours at 750 C exposure.
(a) Back scattered image (b) Cr (c) Al (d) C. 500x

a

b

c

d

Figure 6 Microprobe analysis of Ni-20Cr-5Ti after 6000 hours at 750 C exposure.
(a) Back scattered image, (b) Cr (c) Ti (d) C. 500x

Figure 7 Microstructure of Ni-20Cr-2Al-8Ti after exposure for 6000 hours
at 750 C. Etched 200x

a

b

c

d

Figure 8 Microprobe analysis of Ni-20Cr-2Al-8Ti after 6000 hours at 750 C exposure. (a) Cr (b) Ti (c) Al (d) C. 500x

Figure 9 Microstructure of Ni-20Cr-8Al-2Ti after exposure for 6000 hours
at 750 C. Etched 200x

Figure 10 Microstructure of Ni-20Cr after exposure for 3000 hours at 850
C. Etched 500x

Figure 11 Microstructure of Ni-50Cr after exposure for 3000 hours at 850
C. Etched 500x

a

b

Figure 12 Microprobe analysis of Ni-50Cr after 3000 hours at 850 C exposure.
(a) Back scattered image, (b) C. 400x

Figure 13 Microstructure of Ni-20Cr-8Al after exposure for 3000 hours at 850 C. Etched 250x.

a

b

c

d

re 14 Microprobe analysis of Ni-20Cr-8Al after 3000 hours at 850 C exposure.
(a) Back scattered image (b) Al (c) Cr (d) C. 400x

Figure 15 Microstructure of Ni-20Cr-5Ti after exposure for 3000 hours at 850 C. As polished 500x

a

b

c

d

Figure 16 Microprobe analysis of Ni-20Cr-5Ti after 3000 hours at 850 C exposure.
 (a) Back scattered image (b) Cr (c) Ti (d) C. 400x

Figure 17 Microstructure of Ni-20Cr-2Al-8Ti after exposure for 3000 hours
at 850 C. Etched 500x

a

b

c

d

e

Figure 18

Microprobe analysis of Ni-20Cr-2Al-8Ti after 3000 hours at 850 C exposure. (a) Back scattered image, (b) Ti (c) Al (d) Cr (e) C. 400x

Figure 19 Microstructure of Ni-20Cr-8Al-2Ti after exposure for 3000 hours
at 850 C. Etched 500x

a

b

c

d

e

Figure 20

Microprobe analysis of Ni-20Cr-8Al-2Ti after 3000 hours at 850 C exposure. (a) Back scattered image (b) Al (c) Cr (d) Ti (e) C. 400x

Figure 21 Microstructure of Ni-50Cr after 6000 hours exposure at 950 C.
Etched 200x.

a

b

c

Figure 22 Microprobe analysis of Ni-50Cr after 6000 hours at 950 C exposure.
(a) Back scattered image, (b) Cr (c) C. 400x

Figure 23 Microstructure of Ni-20Cr-8Al after exposure for 6000 hours at 950 C. Etched 200x.

a

b

c

d

Figure 24 Microprobe analysis of Ni-2Cr-8Al after 6000 hours at 950 C exposure. (a) Back scattered image, (b) Al (c) Cr (d) C. 500x

a

b

c

d

e

Figure 25

Microprobe analysis of Ni-2Cr-8Al-2Ti after 6000 hours at 950 C exposure. (a) Back scattered image (b) Al (c) Cr (d) Ti (e) C. 500x

a

b

c

d

Figure 26 Microprobe analysis of Ni-20Cr-1Y after 6000 hours at 950 C exposure.
(a) Back scattered image, (b) Cr (c) Y (d) C. 500x

Figure 27 Change in bulk carbon content as a function of time at 1050 C.

Figure 28 Microstructure of Ni-50Cr after exposure for 1000 hours at 1050 C. Etched 500x

a

b

c

Figure 29 Microprobe analysis of Ni-50Cr after 1000 hours at 1050 C exposure.
(a) Back scattered image (b) Cr (c) C. 400x

Figure 30 Microstructure of Ni-2Cr-8Al after exposure for 1000 hours at 1050 C. Etched 250x

a

b

c

d

Figure 31 Microprobe analysis of Ni-20Cr-8Al after 1000 hours at 1050 C exposure (a) Back scattered image (b) Cr (c) Al (d) C. 400x

Figure 32 Microstructure of Ni-20Cr-8Al-2Ti after exposure for 1000 hours
at 1050 C. Etched 500x

a

b

c

d

Figure 33 Microprobe analysis of Ni-20Cr-8Al-2Ti after 1000 hours at 1050 C exposure (a) Back scattered image (b) Cr (c) Al (d) C. 400x

a

b

Figure 34 Microprobe analysis of Ni-20Cr-5Ti after 1000 hours at 1050 C exposure.
(a) Back scattered image (b) Ti. 400x

Figure 35 Microstructure of Ni-20Cr-2Si after exposure for 1000 hours at 1050 C. Etched 500x

a

b

c

d

Figure 36 Microprobe analysis of Ni-20Cr-2Si after 1000 hours at 1050 C exposure. Near edge - (a) Back scattered image, (b) C. Near middle of specimen - (c) Back scattered image, (d) C. 400x

Figure 37 Microstructure of Ni-2Cr-5Si after exposure for 1000 hours at 1050 C. Etched 250x

Figure 38 Microstructure of Ni-20Cr-1Y after exposure for 1000 hours at 1050 C. Etched 250x

a

b

c

d

e

f

Figure 39

Microprobe analysis of Ni-20Cr-1Y after 1000 hours at 1050 C exposure
Near edge - (a) Back scattered image, (b) Cr (c) Y (d) C. Near middle
of specimen - (e) Back scattered image (f) C. 400x

Figure 40 Microstructure of Ni-20Cr after exposure for 3000 hours at 1050
C. Etched 200x

a

b

c

d

e

f

Figure 41 Microprobe analysis of Ni-20Cr after 3000 hours at 1050 C exposure. Near edge - (a) Back scattered image, (b) Cr (c) C. Near middle of specimen - (d) Back scattered image, (e) Cr (f) C. 500x

a

b

c

Figure 42 Microprobe analysis of Ni-50Cr after 3000 hours at 1050 C exposure.
(a) Back scattered image (b) Cr (c) C. 500x

Figure 43 Microstructure of Ni-50Cr after 3000 hours exposure at 1050 C.
Etched 200x

Figure 44 Microstructure of Ni-20Cr-8Al after 3000 hours exposure at 1050
C. Etched 200x

a

b

c

d

Figure 45 Microprobe analysis of Ni-20Cr-8Al after 3000 hours at 1050 C Exposure. Near edge - (a) Back scattered image, (b) Cr (c) Al (d) C. 500x

e

f

g

h

Figure 45 (contd.) Microprobe analysis of Ni-20Cr-8Al after 3000 hours at 1050 °C exposure. Near middle of specimen - (e) Back scattered image, (f) Cr (g) Al (h) C. 500x

Figure 46 Microstructure of Ni-20Cr-2Al-8Ti after exposure for 3000 hours
at 1050 C. Etched 200x

a

b

c

d

e

Figure 47 Microprobe analysis of Ni-20Cr-2Al-8Ti after 3000 hours at 1050 C exposure (a) Back scattered image (b) Cr (c) Ti (d) Al (e) C. 500x

a

b

c

Figure 48

Microprobe analysis of Ni-20Cr-5Ti after 3000 hours at 1050 C exposure.
Near middle of specimen (a) Back scattered image, (b) Ti (c) C.
500x

Figure 49 Microstructure of Ni-20Cr-2Si after exposure for 3000 hours at 1050 C. Etched 200x

a

b

c

Figure 50 Microprobe analysis of Ni-20Cr-2Si after 3000 hours at 1050 C exposure. Near middle of specimen - (a) Back scattered image, (b) Cr (c) C. 500x

Figure 51 Microstructure of Ni-20Cr-5Si after exposure for 3000 hours at 1050 C. Etched 100x

a

b

c

d

Figure 52 Microprobe analysis of Ni-20Cr-5Si after 3000 hours at 1050 C exposure. Near middle of specimen - (a) Back scattered image (b) Cr (c) Si (d) C. 500x

Figure 53 Microstructure of Ni-20Cr-1Y after exposure for 3000 hours at 1050 C. Etched 200x

a

b

Figure 54 Microprobe analysis of Ni-20Cr-1Y after 3000 hours at 1050 C exposure. Near middle of specimen - (a) Cr (b) C. 400x

Figure 55 Microstructure of Ni-20Cr-2Nb after exposure for 3000 hours at 1050 C. Etched 100x

a

b

c

d

Figure 56

Microprobe analysis of Ni-20Cr-2Nb after 3000 hours at 1050 C exposure. Near middle of specimen (a) Back scattered image, (b) Cr (c) Nb (d) C. 500x

Figure 57 Microstructure of Ni-20Cr after exposure for 6000 hours at 1050 C. Etched 200x

a

b

c

Figure 58

Microprobe analysis of Ni-20Cr after 6000 hours at 1050 C exposure.
Near middle of specimen - (a) Back scattered image, (b) Cr (c) C.
500x

Figure 59 Microstructure of Ni-50Cr after exposure for 6000 hours at 1050 C. Etched 100x

Figure 60 Microstructure of Ni-20Cr-8Al after exposure for 6000 hours at 1050 C. Etched 100x

Figure 61 Microstructure of Ni-20Cr-2Al-8Ti after exposure for 6000 hours at 1050 C. Etched 100x

Figure 62 Microstructure of Ni-20Cr-8Al-2Ti after exposure for 6000 hours at 1050 C. Etched 100x

Figure 63 Microstructure of Ni-20Cr-5Ti after exposure for 6000 hours at 1050 C. Etched 200x

Figure 64 Microstructure of Ni-20Cr-2Si after exposure for 6000 hours at 1050 C. Etched 100x

Figure 65 Microstructure of Ni-20Cr-5Si after exposure for 6000 hours at 1050 C. Etched 100x

Figure 66 Microstructure of Ni-20Cr-1Y after exposure for 6000 hours at 1050 C. Etched 200x

a

b

Figure 67

Microprobe analysis of Ni-20Cr-1Y after 6000 hours at 1050C exposure. Near middle of specimen - (a) Back scattered image (b) C. 500x

Figure 68

Microstructure of Ni-20Cr-2Nb after exposure for 6000 hours at 1050 C. Etched 100x

Figure 69 Increase in bulk carbon content as a function of temperature (3000 hour exposure).