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**CARBURIZATION OF AUSTENITIC ALLOYS BY
GASEOUS IMPURITIES IN HELIUM**

by
G. Y. LAI and W. R. JOHNSON

DATE PUBLISHED: MARCH 1980

GENERAL ATOMIC COMPANY

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ABSTRACT

The carburization behavior of Alloy 800H, Inconel Alloy 617 and Hastelloy Alloy X in helium containing various amounts of H_2 , CO, CH_4 , H_2O and CO_2 was studied. Corrosion tests were conducted in a temperature range from 649° to 1000°C (1200° to 1832°F) for exposure time up to 10,000 h. Four different helium environments, identified as A, B, C and D, were investigated. Concentrations of gaseous impurities were 1500 $\mu\text{atm } H_2$, 450 $\mu\text{atm CO}$, 50 $\mu\text{atm } CH_4$ and 50 $\mu\text{atm } H_2O$ for Environment A; 200 $\mu\text{atm } H_2$, 100 $\mu\text{atm CO}$, 20 $\mu\text{atm } CH_4$, 50 $\mu\text{atm } H_2O$ and 5 $\mu\text{atm } CO_2$ for Environment B; 500 $\mu\text{atm } H_2$, 50 $\mu\text{atm CO}$, 50 $\mu\text{atm } CH_4$ and $<0.5 \mu\text{atm } H_2O$ for Environment C; and 500 $\mu\text{atm } H_2$, 50 $\mu\text{atm CO}$, 50 $\mu\text{atm } CH_4$ and 1.5 $\mu\text{atm } H_2O$ for Environment D. Environments A and B were characteristic of high-oxygen potential, while C and D were characteristic of low-oxygen potential. The results showed that the carburization kinetics in low-oxygen potential environments (C and D) were significantly higher, approximately an order of magnitude higher at high temperatures, than those in high-oxygen potential environments (A and B) for all three alloys. Thermodynamic analyses indicated no significant differences in the thermodynamic carburization potential between low- and high-oxygen potential environments. It is thus believed that the enhanced carburization kinetics observed in the low-oxygen potential environments were related to kinetic effects. A qualitatively mechanistic model was proposed to explain the enhanced kinetics. The present results further suggest that controlling the oxygen potential of the service environment can be an effective means of reducing carburization of alloys.

CONTENTS

ABSTRACT	iii
1. INTRODUCTION	1
2. MATERIALS AND EXPERIMENTS	3
3. EXPERIMENTAL RESULTS	9
4. DISCUSSION	20
5. SUMMARY AND CONCLUSIONS	38
6. ACKNOWLEDGMENTS	40
7. REFERENCES	41

FIGURES

1. Mass of carbon increase as a result of exposure in Environments C/D at various temperatures and times for Hastelloy Alloy X: (a) Heat C-15567, (b) Heat 5-2831	14
2. Rates of carbon increase in Alloy 800H as a result of exposure to Environments A, B and D as a function of inverse of exposure temperatures (absolute temperature)	16
3. Rates of carbon increase in Inconel Alloy 617 as a result of exposure to Environments B, C and D as a function of inverse of exposure temperatures (absolute temperature) . . .	17
4. Rates of carbon increase in Hastelloy Alloy X as a result of exposure to Environments A, B, C and D as a function of inverse of exposure temperatures (absolute temperatures) .	18
5. Calculated carbon activities in the test environment for various temperatures based on the reaction: $\text{CO} + \text{H}_2 \rightleftharpoons \text{C} + \text{H}_2\text{O}$. Also plotted are calculated carbon activities in equilibrium with Cr_{23}C_6 in indicated alloys	22
6. Calculated carbon activities in the test environment for various temperatures based on the reaction: $2\text{CO} \rightleftharpoons \text{C} + \text{CO}_2$. Also plotted are calculated carbon activities in equilibrium with Cr_{23}C_6 in indicated alloys	23
7. Calculated carbon activities in the test environment for various temperatures based on the reaction: $\text{CH}_4 \rightleftharpoons \text{C} + 2\text{H}_2$. Carbon activities in austenitic alloys not plotted in the figure are well below the range in the figure	24

FIGURES (continued)

8.	Calculated carbon activities in the test environment for various temperatures based on the reaction: $\text{CH}_4 + \text{CO} \rightleftharpoons 2\text{C} + \text{H}_2\text{O} + \text{H}_2$. Carbon activities in austenitic alloys not plotted in the figure are well below the range in the figure	25
9.	Calculated partial pressure of CO in equilibrium with Cr, Cr_2C_6 and Cr_2O_3 at indicated Cr activities in accordance with the reaction $27\text{Cr} + 6\text{CO} \rightleftharpoons \text{Cr}_{23}\text{C}_6 + 2\text{Cr}_2\text{O}_3$ as a function of $1/T$. Also plotted are partial pressures for Environments A, B, C and D	27
10.	Equilibrium oxygen partial pressures for various metallic oxides as a function of $1/T$ (K^{-1}). Also plotted in the figure are calculated oxygen partial pressures for Environments A, B, C and D based on the reaction: $\text{H}_2\text{O} \rightleftharpoons 1/2 \text{O}_2 + \text{H}_2$	30
11.	The stability diagram for the Cr-O-C system at 900°C (1650°F). Also shown in the figure are calculated oxygen partial pressures in Environments A, B, C and D	32
12.	Optical photomicrographs showing the typical oxide scale formed on the metal surface of Alloy 800H, Inconel Alloy 617 and Hastelloy Alloy X after exposure to environment A at 871°C (1600°F) for 5000 h (a,b,c) and to environment B at 900°C (1650°F) for 5000 h (d,e,f). The phases indicated by the arrow were found to be chromium-depleted metal matrix phases. Unetched	33
13.	Optical photomicrographs showing the typical oxide-carbide duplex scale formed on the metal surface of Alloy 800H (a), Inconel Alloy 617 (b), and Hastelloy Alloy X (c) after exposure to environment C at 900°C (1650°F) for 3000 h. Unetched	34
14.	Schematics of the qualitatively mechanistic model for carburization of metals in high-oxygen potential environments (A and B) and low-oxygen potential environments (C and D)	37

TABLES

1.	Exposure conditions for the alloys under investigation	5
2.	Heat number, form, and grain size of alloys tested in each environment	6
3.	Chemical composition of alloys under investigation	7
4.	Results of bulk carbon analysis on specimens exposed in Environment A	10

TABLES (continued)

5. Results of bulk carbon analysis on specimens exposed in Environment B	11
6. Results of bulk carbon analysis on specimens exposed in Environment C	12
7. Results of bulk carbon analysis on specimens exposed in Environment D	13

1. INTRODUCTION

Helium is used as a primary coolant in high-temperature gas-cooled reactors (HTGRs) designed in the U.S., Germany, Japan, etc. Because it is chemically inert, helium cannot react with structural materials under normal or accident conditions. However, the primary coolant helium in an operating gas-cooled reactor can be contaminated by small amounts (parts per million levels) of gaseous impurities. The principal impurities are H_2 , CO , CH_4 and H_2O . These impurities may arise from a variety of sources, such as water and steam ingress, reactions of ingressed steam with the core graphite, outgassing of reactor materials, proton diffusion through steam tubes, oil ingress, etc. Corrosion of metals by these gaseous impurities at elevated temperatures can be quite significant. Recent studies (Refs. 1-5) have indicated that the corrosion of austenitic alloys by these gaseous impurities includes both oxidation and carburization. Carburization is believed to be a more important mode of corrosion in affecting the material performance. Current understanding of these gas-metal interactions, particularly their mechanistic and kinetic aspects, however, is still limited. Extensive work is required to fully understand the corrosion behavior and how these corrosive environments affect the long-term mechanical properties before reliable criteria for the material performance under these special conditions can be established.

The HTGR can be utilized for electricity generation through steam raising or direct-cycle gas-turbine systems and for process heat applications. The levels of helium coolant impurities are expected to be different for the different systems adopted for the HTGR. One of the major differences lies in the oxygen potential (or oxygen partial pressure) of the environment. In the case of a steam-raising system where continuous ingress of low levels of water and steam into the primary coolant is possible, the environment is expected to have a relatively high oxygen potential (i.e., low ratios of H_2 to H_2O). On the other hand,

it is anticipated that the helium coolant in the direct-cycle gas-turbine or process heat systems will be much "drier" (i.e., high ratios of H_2 to H_2O) due to the lack of potential sources of continuous water and steam ingress. This paper reports the results obtained from corrosion tests in the helium environments which are characteristic of both steam-raising and gas-turbine/process heat systems. It is the intent of this paper to integrate these results and offer some insights on the mechanistic and kinetic aspects of the corrosion with particular emphasis on carburization in these high-temperature gaseous environments.

The alloys investigated were Alloy 800H, Inconel^{*} Alloy 617 and Hastelloy^{**} Alloy X. These alloys are being considered as candidate materials for the construction of many high-temperature structural components of HTGRs.

^{*}Inconel is a registered trademark of the International Nickel Company, Inc.

^{**}Hastelloy is a registered trademark of Cabot Corporation.

2. MATERIALS AND EXPERIMENTS

Corrosion tests were conducted in four different helium environments: A, B, C and D. Concentrations of the gaseous impurities in each environment are shown in Table 1. Each test environment was generated by first purifying high-purity nuclear-grade helium and then adding controlled quantities of H_2 , CH_4 , CO, CO_2 and H_2O to the purified helium.

Purification of the helium for Environments A, B and C (once-through systems) was accomplished by passing commercial-purity helium through activated charcoal and/or molecular sieve (5 Å diam) at liquid nitrogen temperature, while the initial helium supply for Environment D (recirculating loop) was provided by means of a special bypass purification section as described in Ref. 6. The H_2 , CH_4 , CO and CO_2 concentrations for Environments A, B and C were established by adding controlled quantities of research-purity gases to the purified helium by means of low-flow vacuum leak valves. H_2O was added to Environments A and B by a bypass flow through hydrated oxalic acid crystals at room temperature and at a controlled flow rate, which was calibrated to yield the desired moisture level. No H_2O was deliberately added to Environment C and no CO_2 was added to Environments A and C.

The H_2 , CO and H_2O contents for the recirculating loop (Environment D) were regulated by varying the amount of H_2O injected into the loop as well as the temperature and gas flow through a graphite furnace, where H_2O was converted to CO and H_2 . CH_4 was injected into the loop independently. No CO_2 was deliberately added to this environment.

After obtaining the desired concentrations of gaseous impurities, the gas mixtures were introduced into high-temperature retorts where the corrosion tests were conducted. For tests conducted in A, B and C environments, the test gas after passing through the retort was exhausted to the atmosphere. The once-through test systems used for conducting tests in the A and B environments and in the C environment are described in detail in Ref. 1 and Ref. 2, respectively. The test facility (CIIR^{*}, a recirculating loop) for Environment D tests is described fully in Ref. 6. All test systems were leak tight with no detectable oxygen (by means of gas chromatography) in the system. The test gas before and after passing through each retort was analyzed by a gas chromatograph for H₂ (except for Environment D), CO, CH₄ and CO₂. The H₂ level for the circulating loop was monitored by converting it to H₂O in a CuO furnace and employing a P₂O₅ Meeco Model W electrolytic H₂O analyzer. H₂O for Environments A, B and C was measured using an EG&G dew point hygrometer, while H₂O for Environment D was monitored with a Goldsmith P₂O₅ cell with extra-fine platinum electrodes. Flow rates for the once-through and recirculating loop systems were maintained, depending on the specific system (Table 1), at 180 to 1000 cm³/min such that no significant changes or depletion in the concentration of each gas compound were observed between inlet and exit streams of the test retort. Variations of the above impurities during test were generally +10% of the nominal.

Alloys studied in the present investigation included Alloy 800H, Inconel Alloy 617 and Hastelloy Alloy X. The alloy heat numbers, material forms, grain sizes and specimen configurations for each test environment are shown in Table 2. The chemical compositions of the alloys are tabulated in Table 3.

All specimen surfaces were in the as-machined condition (16 rms or better). The specimens were exposed isothermally at the temperatures given in Table 1. Exposure times were up to 10,000 h.

*Central Institute for Industrial Research, Oslo, Norway.

TABLE 1
EXPOSURE CONDITIONS FOR THE ALLOYS UNDER INVESTIGATION

Test Environment	Gaseous Impurities in Helium (μatm)					Exposure Temperature [$^{\circ}\text{C}$ ($^{\circ}\text{F}$)]
	H_2	CO	CH_4	H_2O	CO_2	
A ^(a)	1500	450	50	50	<0.5 ^(e)	649, 760, 871 (1200, 1400, 1600)
B ^(b)	200	100	20	50	5	649, 760, 871, 900 (1200, 1400, 1600, 1650)
C ^(c)	500	50	50	<0.5 ^(e)	<0.5 ^(e)	800, 900 (1472, 1650)
D ^(d)	500	50	50	1.5	<0.5 ^(e)	650, 800, 900, 1000 (1202, 1472, 1650, 1832)

(a) 1.5-atm helium pressure at a flow rate of $\sim 180 \text{ cm}^3/\text{min}$ flowing through the test retort (5.08-cm-diam alumina retort).

(b) 1.5-atm helium pressure at a flow rate of $\sim 300 \text{ cm}^3/\text{min}$ flowing through the test retort (5.08-cm-diam alumina retort).

(c) 1.05-atm helium pressure at a flow rate of $\sim 500 \text{ cm}^3/\text{min}$ flowing through the test retort (17.1-cm-diam Inconel Alloy 600 retort).

(d) 1.7-atm helium pressure at a flow rate of $\sim 1000 \text{ cm}^3/\text{min}$ flowing through the test retort (7.9-cm-diam Inconel Alloy 600 retort).

(e) Concentration was below the detection limit of the instrument (0.5 μatm).

TABLE 2
HEAT NUMBER, FORM AND GRAIN SIZE OF ALLOYS TESTED IN EACH ENVIRONMENT

Test Environment	Material	Heat Number	Material Form (Specimen Size)	ASTM Grain Size
A	Alloy 800H	HH6979A	1.9-cm-diam rod (0.15 x 1.9 cm diam)	4
	Inconel Alloy 617	XX00A7US	0.12-cm-thick sheet (0.12 x 1.5 x 2 cm)	5
	Hastelloy Alloy X	2600-1-4547	0.14-cm-thick sheet (0.14 x 1.5 x 2 cm)	4
B	Alloy 800H	HH6993A	1.9-cm-diam rod (0.13 x 1.25 x 2.54 cm)	4
	Inconel Alloy 617	XX00A5US	0.165-cm-thick sheet (0.13 x 1.25 x 2.54 cm)	7
	Hastelloy Alloy X	2600-1-4547	0.14-cm-thick sheet (0.13 x 1.25 x 2.54 cm)	5
C	Inconel Alloy 617	XX00A4US	1.27-cm-diam rod (0.63-cm-diam x 2.54-cm length)	3
	Hastelloy Alloy X	C15567	1.27-cm-diam rod (0.63-cm-diam x 2.54-cm length)	4
		5-2831	1.27-cm-diam rod (0.63-cm-diam x 2.54-cm length)	3
D	Alloy 800H	HH6979A	1.9-cm-diam rod (0.63-cm-diam x 2.54-cm length)	4
	Inconel Alloy 617	XX00A4US	1.27-cm-diam rod (0.63-cm-diam x 2.54-cm length)	3
	Hastelloy Alloy X	C15567	1.27-cm-diam rod (0.63-cm-diam x 2.54-cm length)	4
		5-2831	1.27-cm-diam rod (0.63-cm-diam x 2.54-cm length)	3

TABLE 3
CHEMICAL COMPOSITION OF ALLOYS UNDER INVESTIGATION

Material	Heat Number	Chemical Composition (wt %) ^(a)												
		C	Fe	Ni	Cr	Co	Mo	Al	Ti	Mn	Si	S	P	Other
Alloy 800H	HH6979A	0.07	43.6	33.4	20.4	--	--	0.43	0.46	0.77	0.39	0.004	--	Cu = 0.49
	HH6993A	0.07	45.9	31.7	20.1	--	--	0.25	0.35	0.80	0.32	0.006	--	Cu = 0.53
Inconel Alloy 617	XX00A7US	0.08	0.5	54.5	22.3	12.45	9.13	0.89	--	0.03	0.16	0.007	--	
	XX00A5US	0.07	0.19	55.9	21.8	12.24	8.71	0.99	--	0.03	0.06	0.007	--	
	XX00A4US	0.07	0.15	54.7	22.3	12.46	9.09	1.06	--	0.02	0.08	0.007	--	
Hastelloy Alloy X	2600-1-4547	0.08	18.7	Ba1	21.3	2.03	9.44	--	--	0.61	0.32	0.004	0.003	W - 0.62 B - <0.002
	C15567	0.08	19.1	Ba1	21.5	2.11	8.95	--	--	0.71	0.53	0.005	0.018	W - 0.45 B - 0.0029
	5-2831	0.09	18.8	Ba1	21.5	2.08	8.76	0.22	0.01	0.61	0.50	0.005	0.023	W - 0.60 Cu - 0.16 B - <0.002

(a) Vendor's analysis.

Following exposure, the specimens were subjected to a detailed metallurgical examination that included optical metallography, electron microprobe analysis, scanning electron microscopy with energy dispersive analyzer, X-ray diffraction, and bulk carbon analysis.

3. EXPERIMENTAL RESULTS

The results of bulk carbon analysis of specimens exposed to the various environments are presented in Tables 4 through 7. The results are expressed as mass of bulk carbon increase per unit surface area (g/cm^2), which was determined by the relationship

$$\Delta M \text{ (g/cm}^2\text{)} = \Delta C \left(\frac{W}{A} \right) ,$$

where ΔM = mass of carbon increase (g/cm^2),

ΔC = difference in carbon (weight fraction) before and after exposure,

W = weight of unexposed specimen (g),

A = surface area of the specimen exposed to the test environment (cm^2).

As shown in Tables 4 through 7, carburization was observed in all four environments. The data presented are somewhat limited for establishing the rate of carburization for each alloy and environment. Nevertheless, an attempt was made to examine the carburization rate in terms of carbon increase as a function of time for Hastelloy Alloy X tested in C and D environments. The gas chemistry of Environment C was essentially the same as Environment D except for a lower moisture level. No significant differences in carburization kinetics were observed between these two environments. The same heat of material was used for both environments. Accordingly, the data from both environments were plotted together and are shown in Fig. 1.

TABLE 4
RESULTS OF BULK CARBON ANALYSIS^(a) ON SPECIMENS EXPOSED IN ENVIRONMENT A

Material	Heat Number	Specimen Number	Test Temp [°C (°F)]	Test Time (h)	Percent of Carbon Increase (wt %) ^(b)	Mass of Carbon Increase (g/cm ²) ^(c)
Alloy 800H	HH6979A	4-7	649 (1200)	5,000	0	0
		5-7	760 (1400)	5,000	0.001	5.8×10^{-6}
		8-6-1	871 (1600)	2,000	0.009	5.23×10^{-5}
		8-6	871 (1600)	5,000	0.010	5.8×10^{-5}
Inconel Alloy 617	XX00A7US	5-5	760 (1400)	5,000	0	0
Hastelloy Alloy X	2600-1-4547	4-2	649 (1200)	5,000	0.002	1.15×10^{-5}
		5-2	760 (1400)	5,000	0	0
		6-2	871 (1600)	5,000	0.007	4.0×10^{-5}

(a) One measurement per specimen.

(b) The difference in bulk carbon content (wt %) before and after exposure.

(c) Mass of carbon increase per unit surface area of the specimen after exposure to the environment.

TABLE 5
RESULTS OF BULK CARBON ANALYSIS^(a) ON SPECIMENS EXPOSED IN ENVIRONMENT B

Material	Heat Number	Specimen Number	Test Temp [°C (°F)]	Test Time (hr)	Percent of Carbon Increase (wt %) ^(b)	Mass of Carbon Increase (g/cm ²) ^(c)
Alloy 800H	HH6993A	HD2	649 (1200)	5000	0.002	1.0×10^{-5}
		HD4	649 (1200)	7000	0.01	5.06×10^{-5}
		HE2	760 (1400)	5000	0.008	3.98×10^{-5}
		HE4	760 (1400)	7000	0.014	7.0×10^{-5}
		HF2	871 (1600)	5000	0.05	2.5×10^{-4}
		HF4	871 (1600)	7000	0.048	2.38×10^{-4}
		HG2	900 (1650)	5000	0.025	1.28×10^{-4}
		HG4	900 (1650)	7000	0.028	1.4×10^{-4}
Inconel Alloy 617	XX00A5US	7D2	649 (1200)	5000	0.003	1.69×10^{-5}
		7D4	649 (1200)	7000	0.0085	4.7×10^{-5}
		7E2	760 (1400)	5000	0.007	3.85×10^{-5}
		7E4	760 (1400)	7000	0.009	4.85×10^{-5}
		7F2	871 (1600)	5000	0.025	1.4×10^{-4}
		7F4	871 (1600)	7000	0.0265	1.43×10^{-4}
		7G2	900 (1650)	5000	0.011	5.96×10^{-5}
		7G4	900 (1650)	7000	0.024	1.32×10^{-4}
Hastelloy Alloy X	2600-1-4547	XD2	649 (1200)	5000	0.004	2.17×10^{-5}
		XD4	649 (1200)	7000	0.012	6.53×10^{-5}
		XE2	760 (1400)	5000	0.006	3.36×10^{-5}
		XF4	760 (1400)	7000	0.015	8.24×10^{-5}
		XF2	871 (1600)	5000	0.025	1.38×10^{-4}
		XE4	871 (1600)	7000	0.013	6.98×10^{-5}
		XG2	900 (1650)	5000	0.023	1.27×10^{-4}
		XG4	900 (1650)	7000	0.042	2.28×10^{-4}

(a) Two measurements per specimen.

(b) The difference in bulk carbon content (wt %) before and after exposure.

(c) Mass of carbon increase per unit surface area of the specimen after exposure to the environment.

TABLE 6
RESULTS OF BULK CARBON ANALYSIS ON SPECIMENS EXPOSED IN ENVIRONMENT C

Material	Heat Number	Specimen Number	Test Temp [°C (°F)]	Test Time (hr)	Percent of Carbon Increase (wt %) ^(a)	Mass of Carbon Increase (g/cm ²) ^(b)
Inconel Alloy 617	XX00A4US	09156	800 (1472)	3000	0.011	1.37×10^{-4}
		09157	800 (1472)	7000	0.014	1.74×10^{-4}
		09158	800 (1472)	10000	0.0175 ^(c)	2.19×10^{-4}
		09142A	900 (1650)	3000	0.051 ^(c)	6.30×10^{-4}
		09178	900 (1650)	7000	0.076 ^(c)	4.05×10^{-4}
Hastelloy Alloy X	C15567	08161	800 (1472)	3000	0.018	2.22×10^{-4}
		08115	800 (1472)	3000	0.018	2.24×10^{-4}
		08116	800 (1472)	3000	0.018	2.24×10^{-4}
		08182	800 (1472)	3000	0.028	4.92×10^{-4}
		08162	800 (1472)	7000	0.018	2.22×10^{-4}
		08163	800 (1472)	10000	0.028	3.41×10^{-4}
		08125	900 (1650)	3000	0.078	9.60×10^{-4}
		08126	900 (1650)	3000	0.068	8.37×10^{-4}
		08184	900 (1650)	3000	0.068	1.19×10^{-3}
		08142A	900 (1650)	3000	0.038 ^(c)	4.66×10^{-4}
		08143A	900 (1650)	7000	0.118	1.45×10^{-3}
	5-2831	08607	800 (1472)	3000	0.017	2.09×10^{-4}
		08683	800 (1472)	3000	0.017	2.87×10^{-4}
		08684	800 (1472)	3000	0.015	2.52×10^{-4}
		08653	800 (1472)	3000	0.010	1.23×10^{-4}
		08602	900 (1650)	3000	0.05	6.15×10^{-4}
		08604	900 (1650)	3000	0.10	1.24×10^{-3}
		08643	900 (1650)	3000	0.06	7.41×10^{-4}

(a) The difference in bulk carbon content (wt %) before and after exposure.

(b) Mass of carbon increase per unit surface area of the specimen after exposure to the environment.

(c) Average of two measurements per specimens, others one measurement per specimen.

TABLE 7
RESULTS OF BULK CARBON ANALYSIS ON SPECIMENS EXPOSED IN ENVIRONMENT D

Material	Heat Number	Specimen Number	Test Temp [°C (°F)]	Test Time (hr)	Percent of Carbon Increase (wt %) ^(a)	Mass of Carbon Increase (g/cm ²) ^(b)
Alloy 800H	HH6979A	40141	899(1650)	8,700	0.08	9.48×10^{-4}
		40142	900(1650)	3,000	0.06	7.11×10^{-4}
		40143	1000(1832)	3,000	0.07	8.31×10^{-4}
Inconel Alloy 617	XX00A4US	09145	650(1202)	3,000	0.007	8.73×10^{-5}
		09146	650(1202)	10,000	0.045 ^(c)	5.60×10^{-5}
		09141	800(1472)	3,000	0.011	1.36×10^{-4}
		09142	800(1472)	10,000	0.008	9.99×10^{-5}
		09143	900(1650)	3,000	0.096	1.19×10^{-3}
		09144	900(1650)	10,000	0.206	2.53×10^{-3}
Hastelloy Alloy X	C15567	08145	650(1202)	3,000	0.008	9.96×10^{-5}
		08146	650(1202)	10,000	0.008	9.96×10^{-5}
		08141	800(1472)	3,000	0.028	3.45×10^{-4}
		08142	800(1472)	10,000	0.068	8.40×10^{-4}
		08143	900(1650)	3,000	0.058	7.17×10^{-4}
		08144	900(1650)	10,000	0.168 ^(c)	2.08×10^{-3}
	5-2831	08645	800(1472)	3,000	0.015	1.84×10^{-4}
		08646	800(1472)	10,000	0.03	3.70×10^{-4}
		08647	900(1650)	3,000	0.08	9.66×10^{-4}
		08648	900(1650)	10,000	0.15	1.84×10^{-3}
		08649	1000(1832)	3,000	0.11	1.35×10^{-3}
		08650	1000(1832)	10,000	0.221	2.73×10^{-3}

(a) The difference in bulk carbon content (wt %) before and after exposure.

(b) Mass of carbon increase per unit surface area of the specimen after exposure to the environment.

(c) Average of two measurements per specimen, others one measurement per specimen.

Fig. 1. Mass of carbon increase as a result of exposure in Environments C/D at various temperatures and times for Hastelloy Alloy X: (a) Heat C15567, (b) Heat 5-2831.

It is assumed, as a first approximation, that carburization of this alloy follows a law of the type $\Delta M = kt^n$, where ΔM is the mass of carbon increase per unit surface area, t is the exposure time, and k and n are constants. Both n and k were determined by a least-squares fit. Values of the rate exponent (n) are slightly less than or about 0.5 (the theoretical value for a parabolic rate) at 800°C (1472°F) and higher than 0.5 (between 0.5 and 1.0) at higher temperatures [e.g., 900° and 1000°C (1650° and 1832°F)]. Obviously, more data are needed before the carburization rate constants in these environments can be quantitatively established.

Nevertheless, a significant observation was made in the present investigation. In examining the kinetic data generated for the four environments, it was found that the rates of carburization in high-oxygen potential environments (A and B) were significantly lower than those of low-oxygen potential environments (C and D). This difference became more pronounced at higher temperatures [e.g., 900° and 1000°C (1650° and 1832°F)], regardless of whether rates were calculated by a parabolic or a linear rate law. For the purpose of illustrating this observation, carburization rates for all four environments were calculated using the parabolic law and are shown in Figs. 2 through 4 for the three alloys. The data for the C and D environments and those for the A and B environments can be grouped into two separate sets. Clearly, the rates obtained from low-oxygen potential environments (C and D) were significantly higher than those of high-oxygen potential environments (A and B) for all three alloys.

It might be argued that the heats of material used in C and D environments were different from those used in A and B environments, suggesting that the differences in carburization rates could be attributed to heat-to-heat variations. However, specimens of Alloy 800H tested in both A and D environments were from the same heat of material. Carburization rates observed in Environment D (low-oxygen potential) were an

Fig. 2. Rates of carbon increase in Alloy 800H as a result of exposure to Environments A, B and D as a function of inverse of exposure temperatures (absolute temperatures).

Fig. 3. Rates of carbon increase in Inconel Alloy 617 as a result of exposure to Environments B, C and D as a function of inverse of exposure temperatures (absolute temperatures).

Fig. 4. Rates of carbon increase in Hastelloy Alloy X as a result of exposure to Environments A, B, C and D as a function of inverse of exposure temperatures (absolute temperatures).

order of magnitude higher than those in Environment A (high-oxygen potential), as illustrated in Fig. 2. Also, two heats of Hastelloy Alloy X were tested in C and D environments, and carburization rates were found to be quite similar. The kinetic data obtained by Inouye (Ref. 7) using different heats of Alloy 800H, Inconel Alloy 617 and Hastelloy Alloy X, tested in an environment similar to C and D environments, were comparable to those obtained in C and D environments in the present study. Tests conducted on the same heat of Hastelloy Alloy X in both A and B environments revealed no significant differences in carburization kinetics (Fig. 4). It is therefore believed that the differences in carburization kinetics observed between the "wet" (A and B) and "dry" (C and D) environments are related to the relative oxygen potentials for the two types of environments.

4. DISCUSSION

The helium coolant in an operating reactor generally makes a complete circuit (viz., from the core to the heat exchangers or gas turbines and back to the core) in several seconds. The gas components in the coolant do not react with each other to reach an equilibrium state under this dynamic condition. Thus, the state of the gas mixtures in the helium coolant is a nonequilibrium condition. In order to simulate this nonequilibrium state of the gas mixtures in the helium coolant, the flow rate of the test gas employed in the tests, although not reaching the reactor flow rate, is normally fast enough to maintain a nonequilibrium state, i.e., no depletion of each gas component during passage through the test retort. This was checked by gas analysis at the inlet and exit streams of the test retort.

Since the gas mixtures in the environment are in a nonequilibrium state, the gas-metal reaction can be approximated by considering each individual chemical reaction interacting with the metal. Carburization of metals by the gas mixtures is possible by one or more of the following reactions:

The carbon activity in the gas phase for each of the four reactions can be calculated by its equilibrium constant (k):

$$a_c = k_1 \frac{P_{\text{CO}} \cdot P_{\text{H}_2}}{P_{\text{H}_2\text{O}}}, \text{ for Reaction (1)}$$

$$a_c = k_2 \frac{(P_{CO})^2}{P_{CO_2}}, \text{ for Reaction (2)}$$

$$a_c = k_3 \frac{P_{CH_4}}{(P_{H_2})^2}, \text{ for Reaction (3)}$$

$$a_c = (k_4)^{\frac{1}{2}} \left(\frac{P_{CH_4} \cdot P_{CO}}{P_{H_2O} \cdot P_{H_2}} \right)^{\frac{1}{2}}, \text{ for Reaction (4)}$$

The carbon activity in the gas phase for each reaction has been calculated and plotted in terms of temperatures and gas chemistries, as illustrated in Figs. 5 through 8. Also plotted in the figures are equilibrium carbon activities in austenitic alloys, which are assumed to be in equilibrium with $Cr_{23}C_6$. This is a reasonable assumption, since $Cr_{23}C_6$ carbides are the most commonly observed carbides in austenitic alloys. The carbon activity maps thus constructed allow prediction of whether or not carburization of metals is thermodynamically feasible. When the carbon activity in the gas phase (i.e., helium environment) is greater than the carbon activity in the metal, carburization of the metal is thermodynamically feasible; conversely, decarburization of the metal is expected. The experimental results were then compared with the thermodynamic predictions made by these carbon activity maps. This comparison can reveal information regarding the possible dominating chemical reaction (or reactions) that controls the gas-metal reaction with regard to carburization or decarburization. This method of analysis was discussed in detail in a previous paper (Ref. 1).

Carburization was observed in all four environments at the temperatures tested [i.e., 649° to 1000°C (1200° to 1832°F)]. The rates of carburization were observed to increase with increasing temperatures. These experimental observations were then compared with the thermodynamic

Fig. 5. Calculated carbon activities in the test environment for various temperatures based on the reaction: $\text{CO} + \text{H}_2 \rightleftharpoons \text{C} + \text{H}_2\text{O}$. Also plotted are calculated carbon activities in equilibrium with Cr_{23}C_6 in indicated alloys.

Fig. 6. Calculated carbon activities in the test environment for various temperatures based on the reaction: $2CO \rightleftharpoons C + CO_2$. Also plotted are calculated carbon activities in equilibrium with $Cr_{23}C_6$ in indicated alloys.

Fig. 7. Calculated carbon activities in the test environment for various temperatures based on the reaction: $CH_4 \rightleftharpoons C + 2H_2$. Carbon activities in austenitic alloys not plotted in the figure are well below the range in the figure.

Fig. 8. Calculated carbon activities in the test environment for various temperatures based on the reaction: $CH_4 + CO \rightleftharpoons 2C + H_2O + H_2$. Carbon activities in austenitic alloys not plotted in the figure are well below the range in the figure.

predictions using the carbon activity maps. Reaction (1) ($\text{CO} + \text{H}_2 \rightleftharpoons \text{C} + \text{H}_2\text{O}$), as illustrated in Fig. 5, predicts decarburization at all test temperatures in Environment B and at temperatures above approximately 750° to 800°C (1382° to 1472°F) for Environments A, C and D. This is not consistent with the experimental observations, and reaction (1) is thus believed to play no part in the carburization of metals in the temperature range of interest. Reaction (2) ($2\text{CO} \rightleftharpoons \text{CO}_2 + \text{C}$) predicts carburization at temperatures up to approximately 850°C (1562°F) and decarburization at 900°C (1650°F) and above for Environment A. Decarburization is predicted for Environment B at temperatures above approximately 706°C (1300°F), and for Environments C and D at temperatures above approximately 760°C (1400°F). The predictions are, again, not in total agreement with the experimental observations. Thus, reaction (2) is not considered to be a dominant reaction controlling the carburization-decarburization reactions for the alloys tested. Carbon activities (a_c) for reactions (3) and (4) in all four test environments at all test temperatures were orders of magnitude higher than those of the alloys, as illustrated in Figs. 7 and 8. Carburization is predicted at all test temperatures by reactions (3) and (4). This is consistent with the experimental observations. Therefore, reactions (3) and/or (4) are believed to control the carburization reactions.

Carburization of austenitic alloys is also thermodynamically possible through reaction (5) below:

This reaction results in simultaneous carburization and oxidation of chromium in the alloy by CO. Equilibrium CO partial pressures (P_{CO}) for the reaction for chromium activities (a_{Cr}) of 0.5 and 1.0 are shown in Fig. 9. [Chromium activities of 0.5 to 1.0 represent an approximate range for Alloy 800H and Hastelloy Alloy X in a temperature range of interest (Ref. 8); somewhat lower values of a_{Cr} for Inconel Alloy 617 are expected.] Figure 9 predicts carburization in Environments A and B at all test temperatures [900°C (1650°F) and lower] and in Environments C and D

Fig. 9. Calculated partial pressure of CO in equilibrium with Cr, Cr_{23}C_6 and Cr_2O_3 at indicated Cr activities in accordance with the reaction $27\text{Cr} + 6\text{CO} \rightleftharpoons \text{Cr}_{23}\text{C}_6 + 2\text{Cr}_2\text{O}_3$ as a function of $1/T$. Also plotted are partial pressures of CO in Environments A, B, C and D.

at test temperatures below 900°C (1650°F). At test temperatures of 900° and 1000°C (1650° and 1832°F), Environments C and D could either carburize or decarburize the alloy depending on its chromium activity (a_{Cr}). It is thus tentatively concluded that Reaction (5) may also be important in the carburization process.

In addition to carburization-decarburization reactions, oxidation of metals by gaseous impurities in helium may also occur. Either one or both of the following two chemical reactions can be responsible for the oxidation:

The oxygen potential (P_{O_2}) of the test environment can be determined from the above two reactions through their equilibrium constants (k) as shown below:

$$k_6 = \frac{(P_{O_2})^{1/2} \cdot P_{H_2}}{P_{H_2O}}, \text{ for reaction (6)}$$

$$k_7 = \frac{(P_{O_2})^{1/2} \cdot P_{CO}}{P_{CO_2}}, \text{ for reaction (7)}$$

No CO_2 was deliberately added to Environment A and only a little (5 μ atm) was added to Environment B; significant amounts of water were present in both A and B environments. The calculated oxygen potential based on Reaction (6) for both A and B environments was significantly higher than that based on Reaction (7). Thus, the oxygen potential in both A and B environments is believed to be dominated by Reaction (6) or the P_{H_2O}/P_{H_2} ratio. For Environments C and D, H_2O and CO_2 were not

deliberately added to the test gas. The concentration of CO_2 in both C and D environments was not measurable by a gas chromatograph. For calculation of the oxygen potential in Reaction (7), $0.5 \mu\text{atm}$ of CO_2 (detection limit of the gas chromatograph) was assumed. H_2O was not measurable in Environment C, and $0.5 \mu\text{atm}$ was assumed in the calculation of oxygen potential in Reaction (6). Environment D contained a very low concentration ($1.5 \mu\text{atm}$) of H_2O . It is shown from the calculation of the oxygen potential from Reactions (6) and (7) that either reaction could be dominating the oxygen potential for C and D environments. It is assumed in this paper that Reaction (6) or $P_{\text{H}_2\text{O}}/P_{\text{H}_2}$ ratios controlled the oxygen potential of C and D environments.

The oxides that are thermodynamically capable of forming on these alloys are shown in Fig. 10, where the oxygen partial pressure (P_{O_2}) in equilibrium with the oxide is plotted as a function of temperature.² Also included in the figure are calculated oxygen potentials of test environments based on Reaction (6) or $P_{\text{H}_2\text{O}}/P_{\text{H}_2}$ ratios. Oxidation of those elements in the alloy is thermodynamically feasible when P_{O_2} in the test environment is greater than those in equilibrium with the oxides. All four environments are reducing to iron oxides (and less stable oxides of nickel, molybdenum, cobalt, etc.) and oxidizing to chromium and elements more reactive than chromium (silicon, titanium, aluminum, etc.). Cr_2O_3 oxides are at the borderline at high temperatures in C and D environments, which exhibit a much lower oxygen potential.

It is clear from the foregoing discussion that the gas-metal reactions are controlled by both oxygen and carbon potentials in the test environment. Depending on their combined potentials, either oxidation or carburization, or both, can occur at the gas-metal interface to form a scale. The type of reactions and corrosion products can be approximately predicted by thermodynamic stability diagrams. The most important system in oxidation/carburization of austenitic alloys in these high-temperature gaseous

Fig. 10. Equilibrium oxygen partial pressures for various metallic oxides as a function of $1/T$ (K^{-1}). Also plotted in the figure are calculated oxygen partial pressures for Environments A, B, C and D based on the reaction: $H_2O \rightleftharpoons 1/2 O_2 + H_2$.

environments is the Cr-O-C system. The stability diagram of the Cr-O-C system at 900°C (1650°F) is illustrated in Fig. 11. The stability diagram defines the region in which the phases are stable and are expected to form at the gas-metal interface.

The present investigation showed carburization of alloys in all four environments. Carbon activities in these environments are thus believed to be equal to or greater than those in equilibrium with Cr_{23}C_6 carbides, otherwise carburization should not have occurred (Fig. 11). Therefore, in the present discussion of oxidation and carburization of metals, it is sufficient to consider only the oxide-carbide boundary in the thermodynamic stability diagrams such as the one shown in Fig. 11. At 900°C (1650°F), for example, the oxygen potentials in Environments A and B are orders of magnitude higher than oxygen partial pressures in equilibrium with both Cr_2O_3 oxides and chromium carbides (i.e., Cr_{23}C_6 , Cr_7C_3 and Cr_3C_2). Cr_2O_3 oxides are thus predicted to form on the alloy surface at the gas-metal interface. This is confirmed in the present investigation.

Figure 12 shows typical oxide scales formed on the alloy surface of Alloy 800H, Inconel Alloy 617 and Hastelloy Alloy X after exposure to Environments A and B. The scale analyzed by X-ray diffraction was found to consist primarily of Cr_2O_3 and MnCr_2O_4 spinels for all three alloys. Carburization of the metal underneath the scale can only occur when carbon penetrates through the oxide scale.

For Environments C and D, the oxygen potentials either are slightly above or intersect the oxide-carbide boundary. Either oxides or carbides are thus expected to form on the alloy surface. It was observed experimentally that after exposure to these environments (C and D), the scale formed on the alloy surface of these three alloys consisted of oxides after short-time exposure (≥ 1000 h) and of oxides and carbides after long-term exposure (≥ 3000 h) at 800°, 900° and 1000°C (1472°, 1650°, and 1832°F). Figure 13 shows typical scales consisting of oxides and

Fig. 11. The stability diagram for the Cr-O-C system at 900°C (1650°F). Also shown in the figure are calculated oxygen partial pressures in Environments A, B, C and D.

Fig. 12. Optical photomicrographs showing the typical oxide scale formed on the metal surface of Alloy 800H, Inconel Alloy 617 and Hastelloy Alloy X after exposure to Environment A at 871°C (1600°F) for 5000 h (a,b,c) and to Environment B at 900°C (1650°F) for 5000 h (d,e,f). The phases indicated by the arrow were found to be chromium-depleted metal matrix phases. Unetched.

Fig. 13. Optical photomicrographs showing the typical oxide-carbide duplex scale formed on the metal surface of Alloy 800H (a). Inconel Alloy 617 (b) and Hastelloy Alloy X (c) after exposure to Environment C at 900°C (1650°F) for 3000 h. Unetched.

carbides formed on specimen surfaces. The oxides in the scale were found by electron microprobe analysis to be enriched in Cr and Mn. X-ray diffraction analysis indicated that the oxides in the scale consisted primarily of Cr_2O_3 and MnCr_2O_4 . The following hypothesis can be offered to explain the presence of oxides and carbides in the scale when the oxygen potential in the environment is at or near the borderline. Assuming P_{O_2} in the environment is slightly above the borderline, oxides are expected to form initially on the metal surface. After nucleation and some growth of the oxides, P_{O_2} at the nearby areas may decrease to favor the formation of carbides until the formation of oxides is again favored. The experimental results of the present investigation appear to suggest this type of repetitive growth process for the scale formation in Environments C and D (Fig. 13).

Previously, it was concluded that Reaction (3) and/or (4) may be controlling the carburization reactions, and that Reaction (5) may also be important in these reactions. Considering Reaction (3), the carbon activities of the four environments at the test temperatures are many orders of magnitude higher than those in the alloys. The carbon activities in Environment B (one of the high-oxygen potential environments) are higher than those of C and D environments (low-oxygen potential environments) at all test temperatures. For Reaction (4) the carbon activities, although slightly higher in Environment C, are about the same in A and D environments. As for Reaction (5), carburization potential in A or B environment is higher than that of C and D environments. Therefore, it is believed that the differences in carburization rates between A/B (high-oxygen potential) and C/D (low-oxygen potential) environments are not due to differences in the thermodynamic carburization potential but are related to kinetic effects.

As discussed previously, the scale formed in A and B environments (high-oxygen potential) consisted mainly of Cr_2O_3 and MnCr_2O_4 oxides. The oxide scale was found by optical microscopy and scanning electron

microscopy to be dense and adherent. It is believed that carburization of the metal underneath the oxide scale can occur only when carbon penetrates through the scale.

A scale consisting of oxides and carbides was observed to form on the alloy surfaces in C and D environments (low-oxygen potential). The oxides in the scale were found to be Cr_2O_3 and MnCr_2O_4 . Similar to the specimens exposed in A and B environments, carbon coming from the test environment would have to penetrate through the scale to carburize the metal underneath. Nevertheless, carbides in the scale may become additional sources of carbon to carburize the metal underneath the scale. The oxygen potential of C and D environments was near the borderline of the oxide-carbide boundary. Thus, carbides formed in the scale are in a metastable condition and can convert to oxides whenever the local oxygen potential increases above the P_{O_2} in equilibrium with both Cr_2O_3 oxides and carbides. It was hypothesized previously that the local oxygen potential at the gas-metal interface may fluctuate during scale formation. Whenever dissociation of carbides in the scale occurs, additional carbon atoms become available to diffuse into the metal underneath the scale resulting in more carburization. This qualitatively mechanistic model is offered to explain the enhanced carburization kinetics when the scale formed on the metal surface consists of oxides and carbides. The model is illustrated graphically in Fig. 14. The present model suggests that increasing the oxygen potential of C and D environments to the levels where only oxides ($\text{Cr}_2\text{O}_3/\text{MnCr}_2\text{O}_4$) form the scale (without carbides) can significantly reduce carburization kinetics. Based on the results of the present investigation, it is suggested that controlling the oxygen potential of the service environment can be an effective means of reducing carburization of metals.

WHEN THE LOCAL OXYGEN POTENTIAL IS HIGHER THAN THE P_{O_2} IN EQUILIBRIUM WITH Cr_2O_3 AND CARBIDES, THE CARBIDE DISSOCIATES RESULTING IN ADDITIONAL CARBON ATOMS PENETRATING THROUGH THE OXIDE SCALE TO CARBURIZE THE SUBSTRATE ALLOY; FOR EXAMPLE,

$$2Cr_{23}C_6 + 69O \rightarrow 23Cr_2O_3 + 12C$$

Fig. 14. Schematics of the qualitatively mechanistic model for carburization of metals in high-oxygen potential environments (A and B) and low-oxygen potential environments (C and D).

5. SUMMARY AND CONCLUSIONS

The carburization behavior of Alloy 800H, Inconel Alloy 617 and Hastelloy Alloy X in four different helium environments (A, B, C and D) at temperatures from 649° to 1000°C (1200° to 1832°F) for exposure times up to 10,000 h was studied. Environments A and B are characteristic of high-oxygen potential, while C and D are characteristic of low-oxygen potential. The major results and conclusions are as follows:

1. Carburization was observed in all four environments. Carburization kinetics in the low-oxygen potential environments (C and D) were found to be significantly higher, approximately an order of magnitude higher at high temperatures, than those in the high-oxygen potential environments (A and B) for all three alloys. Thermodynamic analyses indicated no significant differences in the thermodynamic carburization potential between low- and high-oxygen potential environments. It is thus believed that the enhanced carburization kinetics observed in the low-oxygen potential environments are related to kinetic effects.
2. The scale formed on the metal surface in the high-oxygen potential environments consisted of oxides (Cr_2O_3 and MnCr_2O_4), while the scale formed in the low-oxygen potential environments consisted of oxides and carbides. The carbides formed in the scale in the low-oxygen potential environments are believed to be in a metastable condition in those environments. Whenever dissociation of carbides in the scale occurs due to increases in local oxygen potential, additional carbon atoms become available to carburize the metal underneath the scale. This qualitatively mechanistic model, illustrated graphically in Fig. 14, is proposed to explain the enhanced carburization kinetics observed in the low-oxygen potential environments.

The present model suggests that increasing the oxygen potential of C and D environments to levels where only oxides form in the scale (without carbides) can significantly reduce carburization kinetics.

3. The present results suggest that controlling the oxygen potential of the service environment can be an effective means of reducing carburization of austenitic alloys. The effect of increasing the oxygen potential on the corrosion rates of HTGR core graphite must be considered in evaluating the practical application of this approach.

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