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Contract AT(04-3)-368
Project Agreement No. 6

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EVALUATION STUDY OF HASTELLOY X AS A
NUCLEAR CLADDING

Quarterly Progress Report
1 July through 30 September 1967

November 1967

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APPROVED BY:

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Manufacturing Operations

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EVALUATION STUDY OF HASTELLOY X AS A
NUCLEAR CLADDING

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1.0 INTRODUCTION AND SUMMARY

This report has been prepared to summarize technical progress during the period from 1 July through 30 September 1967, under the program to evaluate Hastelloy X as a cladding material for nuclear fuels. The reported work is authorized by Project Agreement No. 6 of Contract AT(04-3)-368 between the USAEC and the Aerojet-General Corporation. The reported work was performed, primarily, at San Ramon, California, by personnel of the Nuclear Engineering and Manufacturing Operations of the Nuclear Products and Services Group.

Microconstituents were extracted from specimens of as-received low-cobalt Hastelloy X tubing and commercial-cobalt Hastelloy X sheet material. The X-ray diffraction analyses of the residues identified a high and a low parameter M_6C -type carbide in both materials. A similar evaluation of specimens of commercial-cobalt sheet material which had been aged for 2680 hr in inert gas at 1300°F revealed that both of the M_6C -type carbides exist after aging at relatively low temperature, along with sigma and mu phase.

Electron microprobe analyses were performed on samples of low- and commercial-cobalt Hastelloy X tubing which had been exposed in air at 1900 and 2000°F to identify localized changes in the chemical composition of the carbide precipitates, oxide layers and matrix of the alloy. The most significant result of this work was the determination that the Mn-rich outer oxide layer exists on samples aged at temperatures up to 2000°F.

The thermogravimetric tests of specimens of four heats of Hastelloy X were terminated by equipment failure after 3400 hr of exposure. An evaluation of the data accumulated to date revealed that no justification existed

for continuing the test.

The assembly of the apparatus for the performance of tensile creep tests in argon was completed and the system was checked out. Tests of two specimens were initiated but the work was interrupted almost immediately by failure of four cooling water lines.

The centrifugal creep testing of specimens from three heats of Hastelloy X at 1300°F in argon was terminated after completion of the scheduled 3000 hr of uninterrupted testing. The data were reduced and analyzed. Comparison of the data showed one heat (low cobalt E-9505) consistently displayed superior strength properties than the other two heats (commercial cobalt X5-4793, X5-4904 vacuum melted). Based upon these data vacuum melting does not appear to significantly affect the creep properties of Hastelloy X.

The design of centrifugal creep specimens for testing at 1400°F in argon was completed and fabrication of these test specimens was in progress at the end of September.

2.0 TECHNICAL PROGRESS

2.1 TASK A - OXIDATION STUDIES (G. W. Titus and W. L. Clarke, Jr.)

2.1.1 Task A-2, Commercial-Cobalt Specimen Evaluations

2.1.1.1 Microconstituent Extraction and Analysis

Significant effort was devoted during the period to the definition of exact X-ray diffraction patterns of specimen residues by the use of refined extraction techniques. Electrolytic extractions were performed in 10% HCl/methanol as had been the case in earlier work* but the cells were operated at 5 volts and 0.3 amperes to yield a current density of about 0.2 amps/in² as contrasted to 2 amps/in² used previously. A Pt cathode was used in place of the Hastelloy X cathode and extraction times of 6-8 hr were employed rather than 24 hr extraction time used in the earlier work. A fine and a coarse particle precipitate were obtained by: agitating the extraction solution and permitting it to settle for five minutes; carefully decanting and filtering the liquid to separate the fine precipitate held in suspension; and, finally, filtering the material which settled to the bottom of the beaker to separate the coarse precipitates. Filtering was accomplished using a one-inch vacuum filter funnel and Versapor Type 6429 filters (0.9 μ pore size). The one-inch filter discs, with the filtered precipitates in place, were used as the samples for X-ray diffraction analyses; no additional sample preparation was required. X-ray diffraction analyses of randomly-selected clean filter discs reveals that this type of filter did not result in any background peaks in the analysis trace.

* See Quarterly Progress Report for period 5 July through 30 September 1966; AGN-8205.

Specimens of as-received (unexposed) low-cobalt tubing and commercial-cobalt sheet materials were extracted and analyzed by X-ray diffraction using the techniques described above. The results of these analyses are tabulated for the tubing and sheet materials in Tables 1 and 2, respectively. High and low parameter M_6C -type carbides were coexistent in both materials and, in both cases, the low parameter version appeared in the fine precipitate. The lattice parameters for the low-cobalt tubing were 10.901 and 10.955Å, while those for the commercial-cobalt sheet materials were 10.910 and 10.923Å.

A ZrN phase and a $M_{23}C_6$ -type carbide were identified in both materials. The majority of the ZrN was in the fine precipitates, while the majority of the $M_{23}C_6$ was in the coarse precipitates. The lattice parameter for the $M_{23}C_6$ was 10.961Å in the low-cobalt tubing and 10.734Å in the commercial-cobalt sheet material.

Specimens of the commercial-cobalt sheet material aged for 2680 hr in inert gas at 1300°F were reanalyzed using the techniques described above. The results of this work, presented in Table 3, indicated that the low parameter M_6C' and high parameter M_6C type-carbide phases coexist after aging. It had previously been expected that the M_6C' (low parameter carbide) occurred after aging at lower temperatures (1300 to 1600°F) and the M_6C (high parameter carbide) was more predominant after aging at higher temperatures (1750°F and above). Based on the findings shown in Table 3 and a re-evaluation of previously obtained X-ray diffraction patterns, it has now been established that the two distinctly separate carbide phases (M_6C and M_6C') are always present after aging at any temperature. However, the lattice parameters of the two carbide phases are consistently lower at the lower temperatures and become consistently higher as time and temperature increase, indicating a change in precipitate chemistry with changing aging conditions.

The M_6C' phase was predominant in the samples of the fine precipitate and the M_6C phase was predominant in the coarse sample, which also contained the sigma and mu phase constituents discussed in detail in earlier reports.*

* AGN-8224, Quarterly Progress Report, 1 January through 31 March 1967.

AGN-8232, Quarterly Progress Report, 1 April through 30 June 1967.

TABLE 1 - X-RAY DIFFRACTION ANALYSIS AND PHASE IDENTIFICATION
FOR AS-RECEIVED LOW-COBALT HASTELLOY X TUBING

Fine Particles

<u>2θ</u>	<u>I/I₀</u>	<u>d(Å)</u>	<u>Phase</u>	<u>hkl</u>	<u>a₀</u>
32.90	W	2.722	M ₆ C	400	
34.30	W	2.614	ZrN		
36.00	MW	2.495	M ₆ C	331	
37.70	VW	2.386	M ₂₃ C ₆		
39.72	W	2.269	ZrN		
40.70	S	2.217	M ₆ C	422	10.885
41.60	VW	2.171	M ₂₃ C ₆		
42.42	VW	2.131	M ₆ C	200	
43.22	VS	2.093	M ₆ C	333	10.905
44.18	MW	2.050	M ₂₃ C ₆		
47.20	MS	1.926	M ₆ C	440	10.895
48.20	VW	1.888	M ₂₃ C ₆		
50.20	MW	1.817	M ₆ C; M ₂₃ C ₆	422	
55.90	VW	1.645	M ₂₃ C ₆		
57.20	W	1.610	M ₂₃ C ₆ ; ZrN		
60.50	MW	1.530	M ₆ C	551	
65.60	MW	1.423	M ₆ C	553	
67.04	VW	1.396	M ₆ C	553	
68.30	VW	1.373	ZrN		
70.42	MW	1.337	M ₆ C	733	
73.60	MS	1.287	M ₆ C; M ₂₃ C ₆	644	10.920
75.40	M	1.261	M ₆ C; M ₂₃ C ₆		
77.20	VW	1.236	M ₆ C; M ₂₃ C ₆	602	
79.90	VVW	1.201	M ₆ C	720	
88.90	MW	1.101	M ₆ C	755	
91.80	W	1.074	M ₆ C	820	
93.30	W	1.060	M ₆ C	862	
94.00	W	1.054	M ₆ C	773	

a₀ Average: 10.901Å

Table 1 - ContinuedCoarse Particles

<u>2θ</u>	<u>I/I₀</u>	<u>d(Å)</u>	<u>Phase</u>	<u>hkl</u>	<u>a₀</u>
32.80	W	2.730	M ₆ C		
34.10	VW	2.629	ZrN		
35.80	MW	2.508	M ₆ C		
37.60	VW	2.392	M ₂₃ C ₆		
39.50	W	2.281	ZrN		
40.50	MS	2.227	M ₆ C	422	10.935
41.40	W	2.181	M ₂₃ C ₆		
43.00	VS	2.103	M ₆ C	333	10.957
43.70	S	2.071	M ₂₃ C ₆	511	10.790
46.90	M	1.937	M ₆ C	440	10.958
50.00	W	1.824	M ₆ C		
50.90	MW	1.794	M ₂₃ C ₆	531	10.638
57.10	VW	1.613	ZrN		
60.50	W	1.530	M ₆ C		
65.40	W	1.427	M ₆ C		
68.10	W	1.379	ZrN		
73.20	M	1.293	M ₆ C; M ₂₃ C ₆	644	10.971
75.00	MW	1.266	M ₂₃ C ₆	822	10.774
77.00	VW	1.238	M ₂₃ C ₆		
80.30	VW	1.196	M ₂₃ C ₆		
88.60	W	1.104	M ₆ C		
90.50	VW	1.086	M ₆ C		

a₀ Average: 10.955Å(M₆C)
 10.734Å(M₂₃C₆)

TABLE 2 - X-RAY DIFFRACTION ANALYSES AND PHASE IDENTIFICATION
FOR AS-RECEIVED COMMERCIAL-COBALT HASTELLOY X
SHEET

Fine Particles

<u>2θ</u>	<u>I/I₀</u>	<u>d(Å)</u>	<u>Phases</u>	<u>hkl</u>	<u>a₀</u>
33.1	W	2.706	M ₆ C		
34.3	VW	2.614	ZrN		
36.1	MW	2.488	M ₆ C		
39.6	VW	2.276	ZrN		
40.7	S	2.217	M ₆ C	422	10.908
43.2	VS	2.094	M ₆ C	333	10.910
44.0	MW	2.058	M ₂₃ C ₆		
47.2	MS	1.926	M ₆ C	440	10.895
50.2	MW	1.817	M ₆ C		
51.2	W	1.784	M ₂₃ C ₆		
56.0	VW	1.642	ZrN		
57.1	VW	1.613	M ₂₃ C ₆		
60.5	MW	1.530	M ₆ C		
65.6	MW	1.423	M ₆ C		
70.4	MW	1.337	M ₆ C		
73.5	MS	1.288	M ₆ C	644	10.929
75.2	MW	1.264	M ₂₃ C ₆		
79.6	VW	1.204	M ₆ C		
80.6	VW	1.192	M ₂₃ C ₆		
88.8	MW	1.102	M ₆ C		
91.5	W	1.076	M ₂₃ C ₆		
93.5	W	1.058	M ₆ C		

a₀ Average: 10.910Å

Table 2 - ContinuedCoarse Particles

<u>2θ</u>	<u>I/I₀</u>	<u>d(Å)</u>	<u>Phases</u>	<u>hkl</u>	<u>a₀</u>
36.03	W	2.493	M ₆ C		
39.60	VW	2.276	ZrN		
40.60	M	2.222	M ₆ C	422	10.932
43.15	VS	2.096	M ₆ C	333	10.920
43.92	VS	2.061	M ₂₃ C ₆	511	10.738
47.20	M	1.926	M ₆ C	440	10.895
50.18	W	1.818	M ₆ C		
51.10	MS	1.787	M ₂₃ C ₆	531	10.597
60.50	MW	1.530	M ₆ C		
65.50	MW	1.425	M ₆ C		
70.40	MW	1.337	M ₆ C		
73.40	MS	1.290	M ₆ C	644	10.946
75.10	M	1.265	M ₂₃ C ₆	822	10.753
					a ₀ Average: 10.923Å(M ₆ C) 10.691Å(M ₂₃ C ₆)

TABLE 3 - X-RAY DIFFRACTION PATTERNS FOR HASTELLOY
ALLOY X COMMERCIAL-COBALT SHEET (HEAT X4762)
SPECIMENS AGED 2680 HR IN INERT GAS AT 1300°F

<u>Coarse Particles</u>			<u>Fine Particles</u>			<u>Probable Phases</u>
<u>2θ</u>	<u>I/I₀</u>	<u>d(Å)</u>	<u>2θ</u>	<u>I/I₀</u>	<u>d(Å)</u>	
32.9	MW	2.72				M ₆ C
33.2	M	2.70	33.3	MW	2.69	M ₆ C'
35.9	M	2.50				M ₆ C
36.3	M	2.47	36.3	MW	2.47	M ₆ C'; σ
38.2	W	2.36	38.3	MW	2.35	μ
39.1	VW	2.30	39.7	W	2.27	σ
40.6	S	2.22	40.7	MS	2.22	M ₆ C
40.9	MS	2.21	41.0	MS	2.20	M ₆ C'

Table 3 - Continued

<u>2θ</u>	Coarse Particle <u>I/I₀</u>	<u>d(Å)</u>	<u>2θ</u>	Fine Particles <u>I/I₀</u>	<u>d(Å)</u>	<u>Probable Phases</u>
42.0	W	2.15				$\sigma; \mu$
43.3	VS	2.09	43.5	VS	2.08	$M_6C; M_6C'; \sigma$
44.2	W	2.05				$\sigma; \mu$
45.6	VW	1.99				σ
46.5	VW	1.95				$\sigma; \mu$
47.1	MS	1.93	47.2	M	1.93	M_6C
47.6	M	1.91	47.6	MS	1.91	$M_6C'; \sigma$
50.1	MW	1.82				$M_6C; \mu$
50.5	MW	1.81	50.4	MW	1.81	$M_6C'; \sigma$
			50.8	MW	1.80	M_6C'
60.5	MW	1.53	60.8	W	1.52	M_6C
61.0	MW	1.52	61.2	MW	1.51	M_6C'
65.6	MW	1.42				M_6C
66.1	MW	1.41	66.4	W	1.41	M_6C'
70.4	MW	1.34				M_6C
71.2	MW	1.32	71.4	MW	1.32	M_6C'
73.4	S	1.29	73.6	MW	1.29	M_6C
74.2	MS	1.28	74.3	M	1.28	M_6C'
75.1	W	1.26				M_6C
76.0	W	1.25				M_6C'
88.9	MW	1.10				M_6C
89.9	MW	1.09				M_6C'
91.8	W	1.07				M_6C
94.4	W	1.05				M_6C'

Table 3 - ContinuedPORTIONS OF ABOVE PATTERN ATTRIBUTED TO EITHER σ OR μ PHASES

<u>d(Å)</u>	<u>I/I₀</u>	<u>Sigma*</u>		<u>Mu **</u>	
		<u>d(Å)</u>	<u>I/I₀</u>	<u>d(Å)</u>	<u>I/I₀</u>
				2.608	M
2.479	M	2.47	VVW		
2.358	W	2.39	W	2.380	VS
2.300	VW	2.29	MW		
2.154	W	2.15	MS	2.174	M
2.090	VS	2.09	M	2.073	VS
2.050	W	2.04-2.06	M	2.047	M
1.989	VW	1.995	MS	2.030	MW
1.953	VW	1.955	MS	1.951	MW
1.914	M	1.91	MS	1.908	MW
1.820	MW	1.815	VVVW		
1.810	MW	1.79	MW		

* (NASA-TN-D-1325)

** J. Radavich, Micro-Met Laboratories, West Lafayette,
Indiana.

The portion of the total X-ray pattern attributed to either sigma or mu phase is shown separately in Table 3. The overlap in d-spacings for the two constituents is evident from these data although, in this particular sample, the pattern agrees more favorably with patterns reported for sigma than with those reported for mu phase.

2.1.1.2 Microprobe and Microscopy Analyses

Electron microprobe analyses of low- and commercial-cobalt tubing specimens exposed in air at 1900 and 2000°F were completed. Identification of the four samples analyzed is shown in Table 4. The purpose of the analyses was to determine localized changes in chemical composition of carbide precipitates, oxide layers and the matrix of the alloy. The analyses performed included semi-quantitative chart-recorded specimen scans to determine concentration changes of Si, Mo, Cr, Mn, Fe and Ni occurring across the metal-oxide (M-O) reaction zone. Quantitative microprobe analyses for the above elements were also conducted on the outer oxide layer of sample #2, and on at least five carbide precipitates in each of the four samples. In addition, X-ray scanning images for the above-mentioned elements were made in the area of the specimen traverse on each sample.

TABLE 4 - IDENTIFICATION OF SPECIMENS ANALYZED BY THE
ELECTRON MICROPROBE

<u>Sample No.</u>	<u>Heat No.</u>	<u>Material Type</u>	<u>Aging Environ.</u>	<u>History Temp., °F</u>	<u>Duration Time, hr</u>
2	E9517	Low-Cobalt Tubing	Air	1900	7500
3	E9517	Low-Cobalt Tubing	Air	2000	7500
4	X14593	Commercial-Cobalt Tubing	Air	1900	7500
6	X14593	Commercial-Cobalt Tubing	Air	2000	7500

Each sample was electro-plated with a 1 to 5 mil nickel layer prior to metallographic preparation to preserve the edge and protect the brittle oxide layer. The plated samples were then mounted in a conducting, copper-filled diayll pthalate resin and polished through one micron diamond paste.

The area to be analyzed in each sample was selected so that the path of the semi-quantitative specimen traverses passed through a manganese rich outer oxide layer.

Photomicrographs and electron beam scanning images for Si, Mo, Cr, Mn, Fe, and Ni were made in each of the selected areas. These data are shown in Figures 1 and 2, respectively, for commercial-cobalt specimens exposed at 1900 and 2000°F. The area scanned was approximately 300 microns square. The fact that these data indicate that the outer oxide is Mn-rich is significant; this finding resolves earlier* uncertainties relating to the maximum temperature at which the Mn-rich structure persists. In addition, as was the case with specimens exposed at temperatures up to 1800°F, no substantial amounts of Fe, Mo or Ni were identified in the oxide layers. Based on microprobe analyses of specimens aged at lower temperatures*, the SiO₂ particles (Figures 1-b and 2-b) appear to be migrating towards the M-O interface where, after exposure to 1900 and 2000°F, a relatively dense layer of SiO₂ particles forms directly under the M-O interface.

Semi-quantitative, chart-recorded specimen traverses for the six elements were made on each sample from a point in the outside surface oxides to a point in the metal matrix 200 microns from the metal-oxide interface. The X-ray intensity of each element, measured in terms of counts accumulated in a given time during the traverse was continually recorded as a function of position on the sample. These values were later reduced to the chemical compositions presented in Tables 5 through 8. The composition profiles in the metal matrix prepared from these data are shown in Figures 3 through 6.

The build-up of Cr and Mn in the oxide layers results in a depletion of these elements in the metal matrix near the M-O interface as can be seen in Figure 3 through 6. Similarly, the formation of Si-rich precipitates at the interface causes a Si depletion in this region. The concentration of Mo, on the other hand, is slightly higher near the metal-oxide interface. In the case of Sample No. 2, the Mo concentration decreases abruptly at a point in the metal matrix where carbides are first observed, i.e., about 75 microns from the M-O interface. This decrease is to be expected since the carbides are

* AGN-8224, Quarterly Progress Report, 1 January through 31 March 1967.

171-67-927

(a)

(b)

SiK α

(c)

MoL α

(d)

CrK α

(e)

MnK α

(f)

FeK α

(g)

NiK α

FIGURE 1. X-RAY SCANNING IMAGES (25kv) FOR A SPECIMEN (No. 4) OF HASTELLOY X COMMERCIAL-COBALT TUBING AGED 7500hr IN AIR AT 1900°F (230X)

AGN-8246

(a)

(b)
SiK α

(c)
MoL α

(d)
CrK α

(e)
MnK α

(f)
FeK α

(g)
NiK α

FIGURE 2. X-RAY SCANNING IMAGES (25kv) FOR A SPECIMEN (No. 6) OF HASTELLOY X COMMERCIAL-COBALT TUBING AGED 7500hr IN AIR AT 2000°F (230X)

TABLE 5 - COMPOSITION PROFILE IN METAL MATRIX, SAMPLE NO. 2

Approximate distance from metal- oxide inter- face Microns	Concentration Weight % Unnormalized						Total Weight %
	Si	Cr	Mn	Fe	Ni	Mo	
10	.36	15.9	.12	21.0	54.5	8.2	100.1
30	.36	16.5	.11	21.0	54.0	8.2	100.2
50	.42	16.7	.16	21.1	54.6	8.1	101.9
70	.40	17.1	.17	21.0	54.1	8.2	101.0
90	.44	17.6	.18	21.0	54.0	7.5	100.8
110	.40	17.8	.22	20.7	54.0	7.4	100.6
130	.41	18.2	.21	20.7	54.1	7.5	101.1
150	.38	18.6	.25	20.6	54.0	7.4	101.3
170	.38	18.9	.33	20.4	53.3	7.1	100.4
190	.41	19.2	.33	20.2	53.3	7.1	100.5

TABLE 6 - COMPOSITION PROFILE IN METAL MATRIX, SAMPLE No. 3

Approximate distance from metal- oxide inter- face Microns	Concentration Weight %						Total Weight %
	Si	Cr	Mn	Fe	Ni	Mo	
10	.42	16.3	.15	21.5	53.8	8.1	100.3
30	.38	16.3	.13	21.1	54.1	8.5	100.5
50	.40	16.4	.15	20.9	55.1	8.2	101.2
70	.40	16.9	.16	21.1	54.3	8.1	101.0
90	.41	17.0	.17	20.9	54.3	8.1	100.9
110	.44	17.3	.17	20.9	54.5	8.0	101.3
130	.43	17.8	.20	20.8	54.0	8.0	101.2
150	.47	17.9	.23	20.7	54.0	7.6	101.0
170	.46	18.1	.27	20.6	54.0	7.6	101.0
190	.46	18.4	.27	20.4	54.1	7.3	101.0

TABLE 7 - COMPOSITION PROFILE IN METAL MATRIX SAMPLE NO. 4

Approximate distance from metal- oxide inter- face Microns	Concentration Weight %						Total Weight %
	Si	Cr	Mn	Fe	Ni	Mo	
10	.45	18.4	0.14	20.2	51.5	8.4	99.0
30	.45	18.2	0.15	20.3	51.5	8.0	98.6
50	.43	18.4	0.14	20.1	51.7	8.0	98.8
70	.45	18.4	0.12	20.1	51.1	7.9	98.1
90	.42	19.1	0.15	20.2	51.3	7.8	98.9
110	.47	19.4	0.19	20.0	50.9	7.6	98.6
130	.48	19.7	0.24	20.0	51.1	7.5	99.0
150	.51	20.2	0.27	19.8	50.7	7.3	98.7
170	.45	20.2	0.29	19.4	51.1	7.1	98.5
190	.50	20.3	0.35	19.5	51.0	6.9	98.6

TABLE 8 - COMPOSITION PROFILE IN METAL MATRIX, SAMPLE NO. 6

Approximate distance from metal- oxide inter- face Microns	Concentration Weight %						Total Weight %
	Si	Cr	Mn	Fe	Ni	Mo	
10	.40	15.2	0.09	20.9	52.7	8.8	98.1
30	.37	15.2	0.11	20.8	52.9	8.9	98.3
50	.36	15.4	0.11	21.0	52.4	8.8	98.1
70	.41	15.6	0.13	20.8	52.9	8.9	98.8
90	.40	16.0	0.14	20.5	52.8	8.8	98.7
110	.44	16.4	0.15	20.3	52.3	8.7	98.3
130	.42	16.6	0.19	20.4	52.3	8.2	98.0
150	.46	17.1	0.19	20.3	52.5	8.3	98.9
170	.47	17.3	0.21	20.0	52.1	8.1	98.2
190	.50	18.0	0.23	20.0	52.1	8.0	98.9

FIGURE 3. ELEMENT DISTRIBUTION IN THE METAL MATRIX OF SAMPLE NO. 2

FIGURE 4. ELEMENT DISTRIBUTION IN THE METAL MATRIX OF SAMPLE NO. 3

17.1-67-930

FIGURE 5. ELEMENT DISTRIBUTION IN THE METAL MATRIX OF SAMPLE NO. 4

FIGURE 6. ELEMENT DISTRIBUTION IN THE METAL MATRIX OF SAMPLE NO. 6

Mo rich. The concentration of Ni and Fe in all of the samples changed only slightly with distance from the M-O interface.

Two discrete oxide layers are formed during the high temperature oxidation of Hastelloy X. It has been shown earlier* that the inner oxide is Cr_2O_3 while the structure of the outer oxide compares favorably to a MnCr_2O_4 structure. Some uncertainty existed, however, as to whether or not the Mn structure persisted after exposures at temperatures as high as 1900 or 2000°F. To resolve this uncertainty, quantitative microprobe point-counting analyses were performed at three different points in the outer oxide of Sample No. 2, to determine the percentages of Si, Mo, Cr, Mn, Fe and Ni. The results of these analyses are given in Table 9. It can be seen that the inner oxide is close in composition to Cr_2O_3 , while the outer oxide is rich in Mn as well as Cr. The composition of the outer oxide is somewhat similar to that of a MnCr_2O_4 spinel structure as is indicated by the data in Table 10.

TABLE 10 - COMPARISON OF COMPOSITION OF OUTER OXIDE
LAYER WITH SPINEL

<u>Composition, weight percent</u>		
<u>Element</u>	<u>Outer Oxide</u>	<u>MnCr_2O_4 Spinel</u>
Mn	23.3	24.6
Fe	1.0	
Cr	38.4	46.7
Ni	4.5	
O	32.7 (balance)	28.7

Compositional changes in the carbide precipitates with distance from the M-O interface were also observed (see Tables 11 and 12). In each sample, the Ni, Cr, and Si concentration increased while the Fe, Mo, and W concentration decreased with increasing distance from the M-O interface. With the exception of Sample No. 4, the carbon concentration tended to be lower in the carbides nearest the interface.

* AGN-8224, Quarterly Progress Report, 1 January through 31 March 1967.

TABLE 9 - QUANTITATIVE ANALYSES OF OXIDE LAYERS ON SAMPLE NO. 2

Location	Point No.	Concentration Weight %						O(bal)
		Si	Cr	Mn	Fe	Ni	Mo	
Outer Oxide		<.1	38.4*	23.3*	1.0	4.5	<.1	32.7
		<.1	38.4*	23.3*	1.1	4.6	<.1	32.5
		<.1	38.9*	23.0*	1.0	4.3	<.1	32.7
Inner** Oxide		<.1	66.0*	<.1	<.1	.3	<.1	33.5
		<.1	66.0*	<.1	<.1	.3	<.1	33.5

* Computer data corrected for atomic number effects.

** Composition data obtained from semi-quantitative chart scans.

TABLE 11 - QUANTITATIVE ANALYSES OF CARBIDE PRECIPITATES

Sample No.	PPt. No.	Distance from M-O interface Microns	Concentration Weight %							
			Si	Cr	Mn	Fe	Ni	W	Mo	C(bal)
2	1	70	4.4	11.7	<.1	5.9	26.3	1.6	50.0	0
	2	127	4.5	12.0	<.1	4.8	25.7	1.6	48.6	2.8
	3	183	4.7	14.2	<.1	4.3	26.1	1.3	46.9	2.3
	4	235	5.0	14.2	<.1	3.9	26.6	1.5	44.2	4.6
	5	310	5.5	15.9	<.1	3.9	26.8	1.4	44.2	2.2
	6	520	5.4	16.5	<.1	3.8	27.3	1.3	41.8	3.8
3	1	99	3.5	12.1	<.1	5.6	24.4	1.7	50.2	2.3
	2	134	3.6	12.2	<.1	5.4	24.7	1.7	46.6	5.8
	3	183	4.0	12.8	<.1	5.0	25.0	1.6	48.5	3.1
	4	252	4.3	13.9	<.1	4.6	25.6	1.5	47.6	2.7
	5	490	5.1	15.6	<.1	4.0	27.0	1.4	44.1	2.8

TABLE 12 - QUANTITATIVE ANALYSES OF CARBIDE PRECIPITATES

Sample No.	Ppt. No.	Distance from M-O Interface Microns	Concentration, Weight %							
			Si	Cr	Mn	Fe	Ni	W	Mo	C(bal)
4	1	150	5.7	12.6	.04	4.0	26.5	1.6	45.0	4.4
	2	169	6.0	14.6	.05	3.4	28.8	1.5	42.2	3.4
	3	338	6.4	16.4	.08	2.5	28.4	1.6	39.7	4.8
	4	475	6.1	17.9	.08	2.5	28.8	1.3	38.9	4.3
	5	555	6.5	17.6	.08	3.1	29.5	1.4	39.6	2.3
6	1	221	5.2	12.4	.06	4.4	25.1	2.2	47.7	2.8
	2	278	5.4	13.1	.06	4.2	25.6	2.2	45.8	3.6
	3	320	5.6	13.6	.05	4.0	25.8	2.0	44.6	4.3
	4	395	5.7	13.4	.09	3.7	26.1	1.8	43.7	5.4
	5	484	6.1	14.8	.05	3.6	26.4	1.8	43.7	3.6

The measured intensity data from the microprobe was reduced to chemical compositions using an automatic digital computer. A description of the correction equations used in the reduction of the data will be presented in the topical report to be issued shortly. In addition to these corrections, an atomic number correction was applied to the Mn and Cr data for the oxides; the details of this correction have been described by Archand and Mulvey.*

2.1.2 Task A-3, High Temperature Oxidation Tests

2.1.2.1 Thermogravimetric Tests

Failure of a furnace element caused interruption of the thermogravimetric test of specimens of four heats of Hastelloy X (two tubing and two sheet heats) which were being conducted in air at 1900°F. These tests had accumulated a total of 3400 hr of uninterrupted exposure at the time of furnace failure. In consideration of the volume of satisfactory data generated up to the time of interruption, and the fact that experience had shown that a restart of such a test results in erratic data, a decision was made to terminate this test.

A linear plot of the raw data obtained through 3000 hr of testing is shown in Figure 7. As in the case of earlier tests, the commercial-cobalt sheet material (Heat X-4762) demonstrates the best oxidation behavior (lower uniform weight gain), although the weight gain rates for all four heats are nearly equal. Very good consistency among the data for the remaining three heats is apparent; a total terminal difference in weight gain of only 0.5 mg/cm² was measured between heats.

* Archand & Mulvey, X-Ray Optics and X-Ray Microanalysis, Academic Press, pg. 411.

FIGURE 7. WEIGHT GAIN VERSUS TIME FOR SPECIMENS OF VARIOUS HASTELLOY X HEATS EXPOSED IN AIR AT 1900°F

2.2 TASK B - MECHANICAL PROPERTIES STUDIES (R. M. Baciarelli)

2.2.1 Task B-2, Phase II 1800°F Tests

2.2.1.1 Tensile Creep Tests

The data developed during comparison tensile creep tests of Hastelloy X were reported earlier.* As discussed in that report, the small difference between tensile and centrifugal creep data could be attributed to a time factor and a structure factor which are not taken into account by the relations which transform centrifugal creep data into tensile creep data. Apparently, this factor becomes important when considering complex alloys such as Hastelloy X. Dr. J. Dorn (University of California and consultant to Aerojet) has attempted, without success thus far, to develop a mathematical expression in which these factors are considered. However, even though a difference exists which is thought to be caused by the effects of aging on microstructure, this difference is smaller than the differences which were observed between the data developed during several tensile creep tests of Hastelloy X conducted by different laboratories, as discussed earlier.*

2.2.2 Task B-3, Adaptation of Centrifugal and Tensile Creep Apparatus for Inert Atmosphere Testing

2.2.2.1 Adaptation of Tensile Creep Apparatus

Two SATEC Model RB-1800 tensile creep vacuum retorts were installed and the modifications and piping necessary for the use of these retorts in high purity argon atmosphere testing were completed.

Following modification and assembly, shakedown tests of both machines were performed during which the machines operated for 24 hr in vacuum (20 microns) at 1400°F for additional 30 hours in an argon atmosphere. During this time, the oxygen and moisture content of each system decreased from 5 ppm and 25 ppm, respectively, at the end of the 30 hour run. The test set up is shown in Figure 8 and the control panel is shown in Figure 9.

* AGN-8232, Quarterly Progress Report 1 April through 30 June 1967.

FIGURE 8. VACUUM - INERT ATMOSPHERE TENSILE CREEP APPARATUS

171-67-934

FIGURE 9. CONTROL INSTRUMENTATION PANEL

171-67-935

2.2.3 Task B-4, 1300°F Creep Tests in Argon Atmosphere

2.2.3.1 Centrifugal Creep Test

A centrifugal creep test of specimens of three heats of Hastelloy X (Heat Nos. X5-4793, E-9505 and a vacuum-melted heat) at five stress levels in high purity argon at 1300°F, was terminated after 3000 hours of uninterrupted testing. During the test, the temperature varied between 1299 and 1302°F, the oxygen content was maintained at a level less than 0.5 ppm and the moisture content at a level less than 3 ppm.

The data obtained was reduced by the Fortran IV computer program described earlier.* Plots of $\ln$ of pseudo-elastic stress (σ_{pe}) versus $\ln$ of strain (ϵ) were constructed (Figures 10, 11 and 12). Using the slope of the curves and the values of σ_{pe} , the values of the true creep stress (σ_c) were calculated for all specimens from the relation:

$$\sigma_c = \sigma_{pe} \left[\frac{2}{3} + \frac{1}{3} \left(\frac{d \ln \sigma_{pe}}{d \ln \epsilon} \right) \epsilon / \dot{\epsilon} \right] \quad (1)$$

where:

$$\frac{d \ln \sigma_{pe}}{d \ln \epsilon} = \frac{1}{n}$$

and n = slope of plots of σ_{pe} vs ϵ .

Table 13 is a compilation of the calculated data and Figures 13, 14 and 15 are plots of strain (ϵ) versus time for the three heats.

Available time-stress-strain literature data and prior Aerojet creep data on Hastelloy X in air at 1800°F were reviewed and analyzed to derive design criteria for the specimens to be tested at 1300°F in argon. This analysis indicated that, to obtain total strains at 1300°F comparable to those obtained, for example, in 1000 hrs of testing at 1800°F (3.2% total strain at 1409 psi applied creep stress), the applied stress would have to increased by a factor of about 4.5 (i.e., ~ 6,000 psi applied stress to obtain a total strain of 3% at 1300°F in 1000 hours of testing). Since the total strains required in 3000 hours for this test were 3%, 1%, .5%, .2% and .1%, respectively, for the

* AGN-8224, Quarterly Progress Report, 1 January - 31 March 1967.

FIGURE 10. STRAIN VERSUS STRESS FOR CONSTANT $\dot{\epsilon}$ -
HASTELLOY X HEAT E-9505

17.1-67-936

FIGURE 11. STRAIN VERSUS STRESS FOR CONSTANT $\epsilon/\dot{\epsilon}$ -
HASTELLOY X HEAT X5-4793

FIGURE 12. STRAIN VERSUS STRESS FOR CONSTANT $\epsilon / \dot{\epsilon}$ - VACUUM-MELTED - HASTELLOY X

12.1-67-938

TABLE 13 - STRESS-STRAIN DATA FOR HASTELLOY X TESTED IN
CENTRIFUGAL CREEP AT 1300°F IN HIGH PURITY
ARGON

<u>Heat No.</u>	<u>Specimen No.</u>	<u>ϵ_t (%)</u>	<u>σ_{pe} (psi)</u>	<u>$\bar{\sigma}_{pe}$ (psi)</u>	<u>n</u>	<u>1/n</u>	<u>n'</u>	<u>σ_c (psi)</u>
E-9505	36-6	.217	1394.0	1413.8	.60	1.834	1.222	1724.8
	37-6	.171	1044.8	1056.1	"	"	"	1288.4
	6-6	.152	881.7	891.1	"	"	"	1087.1
	7-6	.142	742.7	750.1	"	"	"	915.1
	8-6	.137	603.8	609.1	"	"	"	743.1
X5-4793	31-6	.260	1710.8	1721.9	.70	1.429	1.143	1968.1
	32-6	.200	1088.0	1094.1	"	"	"	1250.5
	33-6	.190	808.2	812.5	"	"	"	928.7
	34-6	.158	668.4	671.4	"	"	"	767.4
	35-6	.102	544.0	545.7	"	"	"	623.7
vacuum- melted (X5-4904)	1-6	.297	1711.2	1726.3	.70	1.429	1.143	1973.2
	2-6	.232	1088.7	1097.0	"	"	"	1253.9
	3-6	.156	808.2	812.5	"	"	"	928.7
	4-6	.163	668.2	671.7	"	"	"	767.7
	5-6	.130	544.0	546.3	"	"	"	624.4

$\dot{\epsilon}$ = strain rate

$\epsilon_t\%$ = total strain (%)

σ_{pe} = initial pseudo elastic stress (psi)

$\bar{\sigma}_{pe}$ = average pseudo elastic stress (psi)

$\sigma_c = \sigma_{pe} \left[\frac{2}{3} + \frac{1}{3} \left(\frac{d \ln \sigma_{pe}}{d \ln \epsilon} \right)_{\epsilon/\dot{\epsilon}} \right] = \bar{\sigma}_{pe} \cdot n' = \text{calculated tensile creep stress}$

$\frac{1}{n} = \left(\frac{d \ln \sigma_{pe}}{d \ln \epsilon} \right)_{\epsilon/\dot{\epsilon}}$

$n' = \frac{2}{3} + \left(\frac{1}{3} \frac{1}{n} \right)$

HASTELLOY X-9505 SAMPLE 36-6 $\sigma_c = 1724.8$ HASTELLOY X-9505 SAMPLE 37-6 $\sigma_c = 1288.4$ HASTELLOY X-9505 SAMPLE 6-6 $\sigma_c = 1087.1$ HASTELLOY X-9505 SAMPLE 7-6 $\sigma_c = 915.1$ HASTELLOY X-9505 SAMPLE 8-6 $\sigma_c = 743.1$

FIGURE 13. STRESS-STRAIN DATA FOR HASTELLOY X TESTED IN CENTRIFUGAL CREEP AT 1300°F IN HIGH PURITY ARGON

HASTELLOY X-4793 SAMPLE 31-6 $\sigma_c = 1968.1$ HASTELLOY X-4793 SAMPLE 32-6 $\sigma_c = 1250.5$ HASTELLOY X-4793 SAMPLE 33-6 $\sigma_c = 928.7$ HASTELLOY X-4793 SAMPLE 34-6 $\sigma_c = 767.4$ HASTELLOY X-4793 SAMPLE 35-6 $\sigma_c = 623.7$

FIGURE 14. COMPARATIVE DATA FOR HASTELLOY X TESTED IN CENTRIFUGAL CREEP AT 1300°F IN HIGH PURITY ARGON

FIGURE 15. CENTRIFUGAL CREEP DATA - STRAIN VERSUS TIME FOR VACUUM-MELTED HASTELLOY X TESTED IN HIGH PURITY ARGON AT 1300°F

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five specimens of each heat, it was determined that a stress of 2000 psi should be sufficient to obtain a total strain of 3% in 3000 hours. This value was reduced slightly to prevent a possible early termination of the test in case the high stress specimens deflect more than calculated.

As indicated by the data in Table 13, the design of the specimens resulted in creep stresses of 1968.1, 1973.2 and 1724.8 psi for the highest stressed specimen in each set. These stresses should, according to calculation, have resulted in approximately the total strains required. However, as shown in Table 13 the total strains produced in this experiment closely approximated the two lowest desired stress levels (.1% and .2% ϵ_t) but the total strain was low by a factor of about 3 for the intermediate stress (.5% ϵ_t) and low by factors of 5 and 10 for the two highest stresses, respectively. It is postulated that this abnormal behavior was the result of the precipitates that occur at the test temperature (M_6C' , M_6C , $M_{23}C_6$, n-phase and σ phase). These precipitates, especially the $M_{23}C_6$ and σ phase, are known to effect the mechanical properties of Hastelloy X significantly. Changes in the mechanical properties are influenced by the amount of precipitates and by the applied stress; increasing quantities of precipitates and higher stresses both increase the tensile and creep strengths.

As pointed out earlier, the specimens most affected were those under higher stresses (in the plastic range) while those within the elastic range (within $\sim .2\%$ total strain) appeared relatively unaffected. Further investigations, including metallography, are planned to resolve these data.

Stress-strain data collected on the three heats (E-9505, X5-4793, and a vacuum melted heat) is summarized in Table 14. The comparison of strain versus time characteristics is shown in Figures 16, 17, 18, and 19.

TABLE 14 - COMPARATIVE DATA FOR HASTELLOY X TESTED
IN CENTRIFUGAL CREEP AT 1300°F IN HIGH
PURITY ARGON

<u>E-9505</u>		<u>X5-4793</u>		<u>Vacuum-melted</u>	
<u>$\epsilon_t, \%$</u>	<u>σ_c, psi</u>	<u>$\epsilon_t, \%$</u>	<u>σ_c, psi</u>	<u>$\epsilon_t, \%$</u>	<u>σ_c, psi</u>
(.217)	(1724.8)	.260	1968.1	.297	1973.2
.171	1288.4	.200	1250.5	.232	1253.9
.142	915.1	.190	928.7	.156	928.7
.137	743.1	.158	767.4	.163	767.7

$\epsilon_t, \% = \% \text{ total strain}$ $\sigma_c = \text{applied creep stress}$

FIGURE 16. COMPARISON OF STRAIN VERSUS TIME CHARACTERISTICS OF THREE HEATS OF HASTELLOY X AT HIGH APPLIED STRESSES

FIGURE 17. COMPARISON OF STRAIN VERSUS TIME CHARACTERISTICS OF THREE HEATS OF HASTELLOY X AT APPLIED STRESS OF ABOUT 1250 psi

FIGURE 18. COMPARISON OF STRAIN VERSUS TIME CHARACTERISTICS OF THREE HEATS OF HASTELLOY X AT APPLIED STRESS OF ABOUT 925 psi

FIGURE 19. COMPARISON OF STRAIN VERSUS TIME CHARACTERISTICS OF THREE HEATS OF HASTELLOY X AT APPLIED STRESS OF ABOUT 750 psi

It can be seen from the Table and the Figures that, at all stress levels, the Heat E-9505 (low-cobalt) material is consistently stronger (reached a lower total strain with the same applied stress). The data indicate that this material is about 15% stronger than Heat X5-4793 and about 20% stronger than the vacuum-melted material. The vacuum-melted heat and the X5-4793 behaved nearly identically under identical conditions; the average difference in strength between these two materials is only about 5%. This comparison indicates that, at the temperature and total strains investigated, vacuum melting does not appear to produce any significant effect on the creep properties of Hastelloy X.

2.2.3.2 Comparison of Tensile Creep Tests

Two comparison tensile creep tests were initiated in the vacuum-inert atmosphere test apparatus described in Section 2.2.2.1 at the highest and lowest stress levels for Hastelloy X Heat 5-4793. It was necessary to terminate these experiments 2 hours after the test temperature (1300°F) had been reached because four of the plastic water cooling lines ruptured. These lines were being replaced with copper tubing at the end of the report period.

2.2.4 Task B-4, 1400°F Creep Test in Argon Atmosphere

2.2.4.1 Centrifugal Creep Test

The specimens required for the 1400°F centrifugal creep test in a high purity argon atmosphere were designed and were being fabricated at the end of September. These specimens were taken from the same three Hastelloy X heats used in the 1300°F test (Heat Nos. X-5-4793, E9505 and a vacuum-melted heat). The five specimens from each heat were designed to achieve total strains of approximately 3%, 1%, 0.5%, 0.2% and 0.1% after 3,000 hours of testing with a head rotation speed of 160 rpm based on the results obtained in the 1300°F test as well as previous data.

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