JUN 8 1965

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Report COO-1319-24

A STUDY OF THE MECHANISM OF STRESS CORROSION
CRACKING IN THE IRON-NICKEL-CHROMIUM ALLOY SYSTEM

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U. S. ATOMIC ENERGY COMMISSION
Chicago Operations Office
Argonne, Illinois

Contract No. AT(11-1)-1319

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RF Project 1673

Report No. COO-1319-24
(Q-7)

QUARTERLY

REPORT

By

THE OHIO STATE UNIVERSITY
RESEARCH FOUNDATION

1314 KINNEAR RD.
COLUMBUS, OHIO 43212

To U. S. ATOMIC ENERGY COMMISSION
Chicago Operations Office
Argonne, Illinois

Contract No. AT(11-1)-1319

On A STUDY OF THE MECHANISM OF STRESS CORROSION
CRACKING IN THE IRON-NICKEL-CHROMIUM ALLOY
SYSTEM

For the period 17 December 1964 - 16 March 1965

Submitted by R. W. Staehle, F. H. Beck, M. G. Fontana,
and J. P. Hirth
Department of Metallurgical Engineering

Date 5 April 1965

I. INTRODUCTION

The aims of this program are (1) to evaluate systematically the susceptibility of iron-nickel-chromium alloys to stress-corrosion cracking in chloride- and hydroxide-containing environments, and (2) to establish a reliable model for stress-corrosion cracking in iron-chromium-nickel alloys. The general experimental program is evaluating commercial and specially prepared alloys over the range of iron and nickel ratios and up to 40% chromium. Studies are being conducted in the areas of stress-corrosion experiments, electrochemistry, and physical metallurgy. Important objectives of this program are (1) to integrate the studies in the stress corrosion, physical metallurgy, and electrochemical areas; and (2) to conduct the studies with a high degree of self-consistency. Through this approach it is expected that the model for cracking which is eventually developed will have a high degree of credibility.

II. STRESS-CORROSION CRACKING EXPERIMENTS

A. RECIRCULATING AUTOCLAVE SYSTEM

During the past quarter additional work has been required to "de-bug" the circulating autoclave system. This has involved mainly work on time-to-cracking circuitry, safety interlocks activated by temperature and pressure alarms, pump control, pressure seals, and stressing jigs. It appears that the system is now ready for running preliminary experiments and these should begin shortly.

B. SCOPING STUDIES OF ALLOY COMPOSITION

The autoclave portion of a preliminary scoping study of commercial and special alloys is about complete in a 600°F, 0.1 w/o NaCl, 100 psi oxygen over pressure environment. The details of this experiment were described in the previous progress report. Available additional results are summarized in Table I. The wires used in these tests are 0.015-inch diameter and have received identical fabrication treatments. The final treatment is to strand anneal at 2200°F. The stay time in the furnace is approximately 10 seconds following which they are quenched. Specimens are exposed in these scoping studies using their as-received surface. Studies of effects of special surface preparations will be undertaken at a later date.

Figure 1 shows alloy 2 (95 Fe-5 Ni) in the stressed and non-stressed conditions. Both alloys are about equally oxidized and stress appears to have little effect.

Figure 2 shows alloy 33 (55 Fe-35 Ni-10 Cr). The non-stressed specimen has no obvious surface oxidation or localized chemical attack. The stressed specimen exhibits both uniform oxidation and intergranular cracking.

Table I. Results from Experiments at 600°F in 0.1 w/o NaCl Solution and 100 psi Oxygen Overpressure (Specimens stressed initially to 40,000 psi except where noted)¹ — 24-Hour Test Period

Specimen No.	Alloy Designation	Alloy Composition, Atomic % ²				Fail ^{3,4}	Comments ⁶
		Fe	Ni	Cr	Other		
18-1	50-Ti	7.9	75.8	14.8	1.5 Ti	end	Stressed specimen pitted
18-2	54-Zn	64	14.8	19.7	1.5 Zn	yes	Not yet metallographically examined (nme)
18-3	54-W	64	14.8	19.7	1.5 W	yes	nme
18-4	54-V	64	14.8	19.7	1.5 V	yes	nme
18-6	54-Re	64	14.8	19.7	1.5 Re	yes	nme
18-8	21-S	45	49.9	5	.1 S	yes	nme
18-9	78-N	25	45	30	.1 N	no	nme
18-10	78-Mo	24.6	44.3	29.6	1.5 Mo	no	nme
18-11	78-Be	24.6	44.3	29.6	1.5 Be	yes	nme
18-12	76	45	25	30	--	yes	nme
18-13	67	45	30	25	--	yes	nme
18-14	66	55	20	25	--	yes	nme
18-15	65	60	15	25	--	yes	nme
17-5	53	70	10	20	--	yes	nme
17-7	52	75	5	20	--	no	nme
17-10	50-Al	7.9	75.8	14.8	1.5 Al	yes	nme
17-11	62	--	80	20	--	end ⁵	Stressed specimen pitted
17-12	60	15	65	20	--	no	Stressed specimen heavily oxidized
17-13	58	35	45	20	--	no	nme
17-15	56	55	25	20	--	yes	nme
16-1	33	55	35	10	--	yes	Intergranular cracking and uniform oxide
16-2	32	65	25	10	--	yes	nme
16-3	21-N	45	49.9	5	.1 N	yes	nme
16-4	45	55	30	15	--	yes	Oxidized entire thickness
16-5	2	95	5	--	--	yes	Oxidized heavily on stressed and unstressed

Table I. (Continued)

Specimen No.	Alloy Designation	Alloy Composition, Atomic % ²				Fail ^{3,4}	Comments ⁵
		Fe	Ni	Cr	Other		
16-6	16 Cr-20 Ni					yes	No oxidation, intergranular and transgranular cracks, pitting
16-7	Incoloy-800 ⁸					yes	Stressed specimen oxidized clear through but only in local region
16-8	78-S	25	45	30	.1 S	no	nme
16-9	54-S	64.9	15	20	.1 S	yes	Pitting, transgranular cracks, buckled oxide, non-stressed specimen was un-attacked
16-10	54-P	64.9	15	20	.1 P	yes	Extensive transgranular cracking, no attack on non-stressed specimen
16-11	50-S	8	76.9	15	.1 S	no	No general surface attack, but 0.002" deep pits in stressed specimen
16-12	50-P	8	76.9	15	.1 P	no	No obvious oxidation but intergranular pitting on stressed specimen
16-13	54-O	64.9	15	20	.1 O	yes	Transgranular cracks, inside of cracks heavily oxidized
16-14	54-N	64.9	15	20	.1 N	yes	nme
16-15	54-Mo	64	14.8	19.7	1.5 Mo	yes	nme
12-1	4-(A)	75	25	--	--	no	nme
12-3	54-C	64.9	15	20	.1 C	yes	nme
12-5	16	80	15	5	--	yes	nme
12-6	6	55	45	--	--	yes	nme
12-7	4-(B)	70	30	--	--	yes	nme
12-9	21-Be	44.3	49.3	4.9	1.5 Be	yes	nme
12-10	19	65	30	5	--	yes	nme
12-12	18	70	25	5	--	yes	nme
12-14	21-Mo	44.3	49.3	4.9	1.5 Mo	yes	nme
12-15	34	45	45	10	--	yes	nme

FOOTNOTES FOR TABLE I

- 1 This is the uncorrected applied stress and is based on the initial load attached to the specimen divided by specimen area. Not included are corrections due to change in shear modulus of spring with temperature, bouyancy of water, torsional stress on wire due to thermal expansion of spring, and creep of specimen.
- 2 These are nominal alloy compositions.
- 3 Some specimens are not reported because failure occurred at the end rather than the middle area. Failures at the end are believed to result from crevice effects. Specimens metallographically examined in center area but which broke at the end are designated "end."
- 4 Failure may result from cracking, pitting, or excessive oxidation.
- 5 Specimen failed at end, but was examined. Metallography was performed on region from center section.
- 6 All specimens are 0.015" diameter wires. All metallographic examinations are taken from center portion of wire. Specimen is about two inches long.
- 7 Furnished by Dr. Allio, Westinghouse Research Laboratories.
- 8 Furnished by Dr. Spalaris, General Electric, Vallecitos. This is a special higher purity Incoloy-800.

Cowan-Dean etch

125x

Neg. #185

Cowan-Dean etch

125x

Neg. #186

Fig. 1 - Alloy 2 (95 Fe - 5 Ni) after 24 hours exposure at 600°F to water containing 0.1 w/o NaCl and 100 psi oxygen over-pressure at ambient. Non-stressed specimen above and stressed specimen below. Initial uncorrected stress applied is 40,000 psi. Cowan-Dean etch (10 ml H₂O, 5 ml HNO₃, 15 ml HCl, 5 g CuSO₄, 30-40 ml H₂O).

Cowan-Dean etch

125x

Neg. #197

Cowan-Dean etch

125x

Neg. #196

Fig. 2 - Alloy 33 (55 Fe - 35 Ni - 10 Cr) exposed to identical conditions as exposed alloy in Fig. 1. Non-stressed specimen above and stressed below

Figure 3 shows a special 16 Cr-20 Ni alloy furnished by Westinghouse Research. This alloy exhibits pitting in both stressed and non-stressed conditions. The stressed specimen exhibits both intergranular and transgranular cracking.

Figure 4 shows alloy 45 (55 Fe-30 Ni-15 Cr). The non-stressed specimen exhibits pits about 0.003" deep. The stressed specimen was oxidized completely through in some cases and in other locations there was intense local oxidation.

Figure 5 shows Incoloy-800 in the stressed and non-stressed conditions. Except for the intense localized oxidation there was no other attack shown in the stressed specimen. There was no observable surface reaction on the non-stressed specimen.

Figure 6 shows alloy 50-P (8 Fe-76.9 Ni-15 Cr-0.1 P). The non-stressed specimen shows no obvious surface oxidation or localized attack; the stressed specimen shows pitting plus intergranular attack.

Figure 7 shows alloy 54-P (64.9 Fe-15 Ni-20 Cr-0.1 P). The stressed alloy exhibits heavy transgranular cracking. Two entirely different cracking patterns are observed. One is heavily oxidized inside the crack and the other is not. There is some tightly adherent oxide on the surface of the stressed specimen but none on the non-stressed.

Figure 8 shows alloy 54-S (same as 54-P but 0.1 a/o S). The stressed specimen exhibits transgranular cracking, heavy local pitting-oxidation, and a thin buckled oxide. The non-stressed specimen is not obviously affected.

It is not appropriate at this time to offer much speculation on the significance of the results described above and in the figures. However, several general preliminary observations can be made as follows:

- (1) In the absence of stress there is very little oxidation, pitting, or other attack except in the very-low-alloy (alloy 2, for example) materials.
- (2) The presence of stress promotes oxidation, cracking, and pitting, the extent of each depending greatly on the alloy. Reasons for such phenomena are generally apparent (i.e., breakdown of local passive conditions) but elucidation of the details of the process require further work.
- (3) Cracking can be both intergranular and transgranular in this alloy system.
- (4) The environmental conditions are generally severe and the uncorrected 40,000 psi applied stress is generally above the yield. See Table II in Section III for room temperature mechanical properties.

Cowan-Dean etch 125x Neg. #205

Cowan-Dean etch 125x Neg. #203

Cowan-Dean etch 350x Neg. #204

Fig. 3 - Special 16 Cr - 20 Ni - Bal Fe alloy exposed to conditions identical to those in Fig. 1. Top photo is non-stressed, middle is stressed, and bottom is detail of another area in stressed specimen.

Cowan-Dean etch 125x Neg. #191

Cowan-Dean etch 125x Neg. #192

Cowan-Dean etch 125x Neg. #193

Fig. 4 - Alloy 45 (55 Fe - 30 Ni - 15 Cr) exposed to conditions identical to those in Fig. 1. Top is non-stressed, middle is one area of stressed, and bottom is another area of stressed. Section in bottom photo is completely oxidized.

Cowan-Dean etch 125x Neg. #207

Cowan-Dean etch 125x Neg. #206

Fig. 5 - Incoloy-800 specially prepared to have low impurities. Exposed to conditions identical to those for Fig. 1. Top specimen is non-stressed, bottom is stressed.

Cowan-Dean etch 125x Neg. #198

Cowan-Dean etch 250x Neg. #199

Fig. 6 - Alloy 50-P (8 Fe - 76.9 Ni - 15 Cr - 0.1 P) exposed to conditions identical to those in Fig. 1. Top specimen is non-stressed, bottom is stressed.

Cowan-Dean etch 125x Neg. #190

Cowan-Dean etch 125x Neg. #189

Cowan-Dean etch 125x Neg. #188

Fig. 7 - Alloy 54-P (64.9 Fe - 15 Ni - 20 Cr - 0.1 P) exposed to same conditions as Fig. 1. Top specimen is non-stressed and bottom two are stressed but from different locations.

Cowan-Dean etch 125x Neg. #209

Cowan-Dean etch 125x Neg. #184

Cowan-Dean etch 350x Neg. #183

Fig. 8 - Alloy 54-S (64.9 Fe - 15 Ni - 20 Cr - 0.1 S) exposed to conditions identical to those in Fig. 1. Top photo is non-stressed, middle is stressed, bottom is stressed.

C. SCOPING STUDIES OF ENVIRONMENTAL EFFECTS

The results described in the previous section raise numerous questions - one of the most important relating to the role of the environment. To this end scoping studies have been initiated in which a series of alloys is being systematically exposed to various components of the water-oxygen-chloride environment (i.e., water, water-oxygen, water-chloride, oxygen). As of the date of this report, the water environment study has been completed. Following this, the water-oxygen, water-chloride, water-hydrogen, water-chloride-oxygen, and other environments will be used, as appropriate.

In the water test, double-distilled water was used; the autoclave was filled to the top and the test assembly inserted; the autoclave was then degassed by heating to 220°F leaving the top valve open; the top valve was then closed and temperature increased to 600°F; the test duration was 24 hours.

In general there was very little attack on the stressed specimens in the water environment. A comparison between stressed and non-stressed alloys is shown in Figs. 9 and 11. Figure 10 shows alloy 60 (15 Fe-65 Ni-20 Cr) in water and water-oxygen-chloride environments, both specimens at 40,000 psi. Figure 10 shows the same comparison for alloy 62 (80 Ni-20 Cr). From a comparison of Figs. 10 and 11 with Figs. 2-9 it appears that more than the stress is required for the accelerated pitting and oxidation phenomena. The environment appears to play an important part. Additional experiments and additional metallography are underway and should illuminate further the nature of environmental differences.

III. MECHANICAL PROPERTIES

In order for the stress-corrosion cracking experiments to be conducted at some stress relative to the mechanical capability of a given material, it is necessary to determine the tensile properties of all compositions as a function of temperature.

Tensile properties of the various alloys at room temperature are summarized in Table II. There is little comment appropriate at this time. Work is continuing to extend these data to higher temperatures. Some work will also be done to determine the effects of annealing time and grain size.

It is planned generally to obtain the tensile properties on all alloys from room temperature to 1200°F, at 200°F increments. The stress-corrosion experiments will be stressed at some fractional value of the yield strength and comparison from alloy to alloy will be made on the basis of yield point fraction.

Table II. Room Temperature Tensile Results^{2,3,4}

Alloy Designation	Alloy Composition, Atomic %				Tensile Stress,	Yield Stress, ¹	% Elong. ⁵
	Fe	Ni	Cr	Other	psi	psi	
1	100	--	--	--	115,750	115,750 ⁶	1.125
2	95	5	--	--	69,750	58,700 ⁶	3.65
					72,000	61,000 ⁶	5.05
3	90	10	--	--	107,000	100,500 ⁶	3.1
					107,000	100,500 ⁶	3.1
4A	75	25	--	--	116,800	104,500 ⁶	3.38
					116,800	104,500 ⁶	3.38
4B	70	30	--	--	63,150	35,400	25.0
					63,150	34,250	27.5
5	65	35	--	--	59,600	35,900	4.0
					60,200	35,900	4.2
6	55	45	--	--	68,500	33,300	25.1
					69,600	33,300	27.1
8	35	65	--	--	80,000	27,650	30.3
					80,000	27,650	30.3
12	--	100	--	--	35,300	27,650	8.71
					35,300	27,650	8.71
13	95	--	5	--	73,300	68,400	2.8
					72,850	62,750	2.8
14	90	5	5	--	102,000	95,200	2.81
					102,000	95,200	2.52
15	85	10	5	--	59,300	24,100	27.6
					59,300	24,100	27.6
16	80	15	5	--	59,100	22,100	28.1
					59,100	26,150	28.1
17	75	20	5	--	97,500	64,800	10.4
					97,700	64,800	10.7
18	70	25	5	--	117,500	117,500	1.29
					113,700	113,700	1.13
19	65	30	5	--	119,000	102,500	1.69
20	55	40	5	--	63,850	24,850	28.1
					63,850	24,850	28.1
21	45	50	5	--	68,900	23,500	27.5
					67,800	23,400	25.3

Table II. (Continued)

Alloy Designation	Alloy Composition, Atomic %				Tensile Stress, psi	Yield Stress, ¹ psi	% Elong. ⁵
	Fe	Ni	Cr	Other			
21 Be	44.3	49.3	4.9	1.5 Be	77,000	29,100	33.2
					76,500	29,100	29.2
21 Mo	44.3	49.3	4.9	1.5 Mo	76,300	29,650	29.8
					76,300	29,650	31.7
21 N	45	49.9	5	.1 N	70,250	25,450	29.8
					69,600	25,450	25.9
21 S	45	49.9	5	.1 S	80,750	39,500	31.7
					80,750	39,500	27.6
24	15	80	5	--	72,350	20,900	23.0
					73,500	20,900	24.7
29	80	10	5	--	39,900	12,750	10.7
					41,775	12,750	12.1
32	65	25	10	--	68,400	20,900	29.0
					68,400	20,900	27.6
33	55	35	10	--	66,250	23,300	24.2
					66,250	23,300	24.2
34	45	45	10	--	59,650	20,900	22.5
					61,000	20,900	26.4
41	75	10	15	--	115,000	80,500	11.5
					112,500	71,500	11.5
42	70	15	15	--	72,700	21,600	29.2
					73,500	21,600	33.7
43	65	20	15	--	69,500	20,900	30.6
					69,500	20,900	28.7
45	55	30	15	--	61,450	19,600	22.75
					61,450	19,600	24.2
46	45	40	15	--	70,000	24,000	28.6
					70,000	24,000	28.9
48	25	16	15	--	78,700	26,000	29.2
					78,700	26,000	27.0
50	8	77	15	--	76,900	24,300	25.3
					73,100	24,300	19.95

Table II. (Continued)

Alloy Designation	Alloy Composition, Atomic %				Tensile Stress,	Yield Stress, ¹	% Elong. ⁵
	Fe	Ni	Cr	Other	psi	psi	
50 Al	7.9	75.8	14.8	1.5 Al	125,800	102,000	3.54
					125,800	102,000	3.54
50 Be	7.9	75.8	14.8	1.5 Be	85,500	31,600	26.9
					85,350	31,600	27.5
50 Mo	7.9	75.8	14.8	1.5 Mo	90,500	31,600	27.0
					90,500	31,600	27.0
50 P	8	76.9	15	.1 P	94,600	39,500	32.6
					94,600	40,700	30.3
50 S	8	76.9	15	.1 S	83,600	48,000	12.38
					82,500	48,000	11.5
50 Ti	7.9	75.8	14.8	1.5 Ti	81,400	31,650	18.5
					78,750	31,100	18.5
52	75	5	20	--	81,900	25,850	26.5
					81,900	25,850	26.5
52 Be	64	14.8	19.7	1.5 Be	119,500	65,800	25.3
					121,200	65,800	28.0
53	70	10	20	--	72,750	25,700	41.4
54	65	15	20	--	76,800	24,300	41.0
					76,800	24,300	43.5
54 Al	64	14.8	19.7	1.5 Al	79,500	28,250	33.2
54 Au	64	14.8	19.7	1.5 Au	86,600	40,400	38.8
					85,900	40,400	37.8
54 Be	64	14.8	19.7	1.5 Be	77,700	28,250	33.2
					77,700	28,250	33.2
54 C	64.9	15	20	.1 C	76,000	27,650	36.0
					76,300	27,650	41.8
54 Co	64	14.8	19.7	1.5 Co	78,000	26,000	40.4
					79,400	26,000	40.4
54 Cu	64	14.8	19.7	1.5 Cu	78,000	37,600	28.1
					75,200	37,600	24.1
54 Ir	64	14.8	19.7	1.5 Ir	83,800	33,300	43.0
					83,800	33,300	43.0

Table II. (Continued)

Alloy Designation	Alloy Composition, Atomic %				Tensile Stress, psi	Yield Stress, ¹ psi	% Elong. ⁵
	Fe	Ni	Cr	Other			
54 Mn	64	14.8	19.7	1.5 Mn	74,750	25,950	28.2
54 Mo	64	14.8	19.7	1.5 Mo	80,700	29,700	46.0
					81,400	29,700	45.2
54 N	64.9	15	20	.1 N	95,200	42,100	33.2
54 O	64.9	15	20	.1 O	73,750	27,300	35.7
54 P	64.9	15	20	.1 P	84,750	38,400	41.0
					84,750	38,400	41.0
54 Pd	14.8	19.7	19.7	1.5 Pd	83,750	33,500	39.0
54 Pt	64	14.8	19.7	1.5 Pt	81,000	26,700	46.3
54 Re	64	14.8	19.7	1.5 Re	84,500	31,600	50.3
					84,500	31,600	50.3
54 Rh	64	14.8	19.7	1.5 Rh	85,100	31,600	44.4
					85,100	31,600	46.0
54 Ru	64	15.8	19.7	1.5 Ru	86,100	31,100	54.0
54 S	64.9	15	20	.1 S	84,500	40,400	41.0
					84,300	40,400	40.5
54 Si	64	14.8	19.7	1.5 Si	82,200	31,600	33.8
					83,200	28,750	38.8
54 Ti	64	14.8	19.7	1.5 Ti	84,750	32,200	35.4
					84,750	32,200	34.4
54 V	64	14.8	19.7	1.5 V	78,200	29,300	37.9
					78,200	29,300	38.9
54 W	64	14.8	19.7	1.5 W	83,200	32,200	45.0
					83,200	32,200	45.0
54 Zn	64	14.8	19.7	1.5 Zn	77,000	27,700	37.5
					77,000	27,700	37.5
55	60	20	20	--	77,700	26,550	33.7
					77,700	26,550	33.2
56	55	25	20	--	77,000	26,550	33.7
					77,000	26,550	33.7
56	55	25	20	--	73,000	26,700	27.3
					73,600	27,250	30.4
57	45	35	20	--	75,750	28,250	29.8
					75,750	28,250	29.8

Table II. (Continued)

Alloy Designation	Alloy Composition, Atomic %				Tensile Stress,	Yield Stress, ¹	% Elong. ⁵
	Fe	Ni	Cr	Other	psi	psi	
58	35	45	20	--	79,750	29,700	30.1
					79,750	29,700	30.1
60	15	65	20	--	89,500	32,200	28.1
					89,500	32,200	28.1
62	--	80	20	--	64,500	31,000	9.83
					69,500	31,100	12.1
64	65	10	25	--	107,000	71,200	34.9
					107,000	71,200	39.2
65	60	15	25	--	80,000	31,100	36.8
					80,000	31,100	39.4
66	55	20	25	--	79,500	29,100	35.7
					79,500	29,100	35.7
67	45	30	25	--	79,500	29,650	35.5
					79,500	29,650	35.5
74	60	10	30	--	112,700	94,250	29.8
					112,700	94,250	33.2
76	45	25	30	--	83,900	33,300	34.0
					83,900	33,300	34.8
77	35	35	30	--	91,700	36,700	32.6
					91,700	36,700	32.6
78	25	45	30	--	94,400	36,200	32.6
					94,400	36,200	32.6
78 Be	24.6	44.3	29.6	1.5 Be	96,600	40,800	34.2
					96,250	40,800	31.4
78 Mo	24.6	44.3	29.6	1.5 Mo	98,100	44,000	33.9
					98,100	44,000	33.9
78 N	25	45	30	.1 N	123,500	66,750	37.6
					123,500	66,750	36.0
78 S	25	45	30	.1 S	99,600	60,400	25.85
					100,900	60,400	36.2
83	35	30	35	--	100,600	48,300	30.9
					100,100	48,300	35.1

Table II. (Continued)

Alloy Designation	Alloy Composition, Atomic %				Tensile Stress, psi	Yield Stress, ¹ psi	% Elong. ⁵
	Fe	Ni	Cr	Other			
85	15	50	35	--	102,000	43,500	32.2
					102,000	43,500	32.0
88 B	15	45	40	--	107,000	49,700	33.1
90 B	--	60	40	--	114,000	27,800	34.8
					113,500	27,800	36.0
Incoloy 800					75,250	26,700	26.7
					75,250	26,700	27.0
Alloy 1620					75,200	31,100	22.5
					76,600	31,100	25.0

¹0.2% offset²Strain rate = 5 cm/min.

Gage length = 17.78 cm (7 inches)

³All wires subject to identical heat treatment. Final treatment was 2200°F for 10 seconds followed by cold hydrogen quench. This is the well-known strand annealing procedure used for stainless steels.

⁴Alloy wires were 0.015" diameter.

⁵Obtaining reduction in area values is difficult on the 0.015" diameter because of the delicate nature of the measurements which must be made. All specimens and tensile curves are being kept on file in the event reduction in area data are needed.

⁶The tensile values for these alloys are questionable and may be affected by heat treatment and contamination. Detailed rechecks of alloy composition will be made.

Cowan-Dean etch 350x Neg. #222

Cowan-Dean etch 350x Neg. #218

Fig. 9 - Alloy 60 (15 Fe - 65 Ni - 20 Cr) stressed at a 40,000 psi uncorrected stress exposed at 600°F to (above) double-distilled deaerated water and (below) to water + 0.1 w/o NaCl + 100 psi oxygen over pressure.

Cowan-Dean etch 350x Neg. #219

Cowan-Dean etch 350x Neg. #221

Fig. 10 - Alloy 62 (80 Ni - 20 Cr) stressed at a 40,000 psi uncorrected stress exposed at 600°F to (above) double-distilled deaerated water and (below) to water + 0.1 w/o NaCl + 100 psi oxygen over pressure.

5000x Neg. #2-22

Fig. 11 - Type 304 stainless steel after 30 min. at 40,000 psi in boiling MgCl_2 . Specimen in as-received condition. Electron photomicrograph of oxide film removed by bromine dissolution.

4000x Neg. #5-14

Fig. 12 - Type 304 stainless steel after 60 min. at 25,000 psi in boiling MgCl_2 . Specimen is fully annealed.

IV. ELECTRON METALLOGRAPHY

Work continues aimed at characterizing the details of surface structure of specimens exposed to environments which promote stress-corrosion cracking. The purposes of this work are to (a) elucidate some of the micro-details of initial stages of surface attack and (b) determine whether certain surface structures can be correlated with susceptibility to cracking.

Surface studies using bromine stripping of oxide films are under way and surfaces of several commercial stainless steels and high-nickel alloys have been examined. The specimen in a stressed condition is first exposed to boiling MgCl_2 for a given length of time. The specimen is then oxidized for 45 minutes at 800°F in $\text{NaNO}_3\text{-KNO}_3$ salt. The film is stripped with a bromine (2-8%) methanol solution and then is viewed in the electron microscope.

The alloys examined to date are Type 304 stainless, Type 309 stainless, Type 310 stainless, Incoloy-800, Incoloy-825, and Inconel-600. The above materials have been studied in the as-received condition and Type 304 and 309 in the annealed condition. Typical observed structures are shown in Figs. 11-15. There appear to be obvious similarities and differences in various structures. For example, the as-received stainless steels show similarities. There are relatively long parallel grouping which may be due to martensite plates or defect structure at slip lines. There is a background structure, more apparent in annealed than in as-received, which has the appearance of dissolution ledges. Finally (most pronounced in Type 304) there are what appear to be spikes (see Fig. 11) extending from the parallel structure. These spikes may be the result of preferential tunneling attack, as has been suggested by some investigators, or may be related primarily to a surface attack. Future studies are planned to clarify these questions.

The annealed Type 304 and 309 appear generally similar, as shown in Figs. 12 and 14. There is a background of ledge-like structure common to both materials. In addition, there are dark circular "blobs" which appear to be pits. They may be related to some of the preferential oxidation seen in Figs. 1-8.

The higher Ni alloys shown in Figs. 16-18 exhibit fewer structural details having the regularity observed in the stainless steels above. There is evidence of irregular boundaries and some extracted precipitates. In the Inconel-600 there is evidence of square structure, possibly etch pits. There is little regularity in the structural detail of Incoloy-825, although some evidence of dissolution, as indicated by a wavelike structure, exists.

V. ELECTROCHEMICAL STUDIES

Anodic polarization equipment has been assembled and checked out. An important feature of this equipment is a continuous recording system capable of covering seven orders of magnitude in current density. The capability to obtain reproducible data and to obtain important correspondences in results from

4000x Neg. #5-19

Fig. 13 - Type 309 stainless steel after 60 min. exposure at 25,000 psi in boiling MgCl_2 . Specimen in as-received condition.

8000x Neg. #5-27

Fig. 14 - Type 309 stainless steel after 60 min. exposure at 25,000 psi in boiling MgCl_2 . Specimen in fully annealed condition.

8000x Neg. #7-22

Fig. 15 - Type 310 stainless steel exposed for 100 min. at 40,000 psi to boiling MgCl_2 . Specimen in as-received condition.

8000x Neg. #7-18

Fig. 16 - Incoloy-800 exposed 100 minutes at 40,000 psi to boiling MgCl_2 . Specimen in as-received condition.

4000x Neg. #7-12

Fig. 17 - Incoloy-825 exposed for 100 minutes at 40,000 psi to boiling MgCl_2 . As-received.

8000x Neg. #6-23

Fig. 18 - Inconel-600 exposed for 100 minutes at 40,000 psi to boiling MgCl_2 . As-received.

different voltage scanning rates has been established. The next quarterly progress report will include a detailed discussion of the system being used and results from scoping studies.

ACKNOWLEDGEMENTS

The assistance being arranged by the following organizations is gratefully acknowledged:

Battelle Memorial Institute -- melting of ingots and preparation of specimens

The Duriron Company, Inc., -- chemical analyses

International Nickel Company, Inc., -- donations of Ni-270 melting stock and commercial alloys

United States Steel Corp., (Monroeville) -- donations of commercial alloys

United States Steel Corp., (American Steel and Wire Division) -- donations of wires of commercial stainless steels

Stellite Division of Union Carbide -- donations of wires of Hastelloy-type alloys

The assistance of Messrs. Farrar, DeLuca, Cowan, Postle, and Simmons in the metallography, x-ray studies, performance of experiments, and construction of equipment is acknowledged.

Technical contributions to this report were made by Messrs. R. L. Beauchamp, R. Cochran, M. Dean, T. Smith, J. R. Upp, and R. W. Staehle.

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