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Report No. COO-2018-8 (Q-3)

STRESS CORROSION CRACKING OF FE-CR-NI ALLOYS
IN CAUSTIC ENVIRONMENTS

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Department of Metallurgical Engineering

July 16, 1969 - October 14, 1969

U. S. Atomic Energy Commission
Chicago Operations Office
Argonne, Illinois

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REPORT

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1314 KINNEAR RD.
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On STRESS CORROSION CRACKING OF Fe-Cr-Ni ALLOYS
IN CAUSTIC ENVIRONMENTS

For the period July 16, 1969 - October 14, 1969

Submitted by Roger W. Staehle
Department of Metallurgical Engineering

Date May 18, 1970

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1.0 ABSTRACT

Stress corrosion tests have been run on commercial stainless steels in boiling 50% NaOH solutions. Resistance to cracking of wire specimens is shown to be proportional to the content of nickel plus chromium. Anodic polarization curves have been determined over a range of NaOH concentrations and for specially prepared alloys in the composition range of stainless steels. All alloys appear to persist in the passive range at room temperature although the anodic current increases as the NaOH concentration increases.

2.0 SUMMARY AND CONCLUSIONS

1. For commercial stainless steels increasing the Ni + Cr content increases the resistance to stress corrosion cracking in boiling 50% NaOH.

2. Studies of the anodic polarization of iron-rich Fe-Cr-Ni alloys shows that these alloys are passive at room temperatures in the range of 0.3 to 10.0 normal NaOH solutions.

3.0 INTRODUCTION

This work is being conducted in support of the IMFBR activities to identify the possible incidences and extent of caustic stress corrosion cracking which could cause premature equipment failure. The most likely sites where such SCC is possible are (1) in the steam generator at the Na-H₂O interface and (2) on exterior surfaces where Na spills or leaks can hydrolyze to NaOH solutions in the presence of moisture.

Caustic SCC is a serious consideration in iron base alloy systems and becomes generally more serious with increasing iron concentrations in the alloy as shown in COO-2018-4.

This program is approaching the problem by conducting both direct SCC studies and fundamental studies aimed at understanding the phenomenon. The work is outlined in a series of tasks and work performed on a given task.

4.0 PROGRESS IN INDIVIDUAL TASKS

Task 1 - Tests of Engineering Geometries

A. Aims

The purpose of this work is to conduct experiments which simulate possible failure modes in engineering geometries to find what kind of corrosion phenomena might occur in the event of a sodium leak. From this information more specific definitions could be given to fundamental studies.

B. Results to Date

None

Task 2 - General Effects of Alloy and Environmental Composition in Aqueous Solvents on Stress Corrosion Cracking

A. Aims

The purpose of this task is to assess the general effects of environment and alloy chemistry on the incidence of caustic stress corrosion cracking. Variables will include alloy chemistry, temperature, concentration of OH^- , identity of cation, dissolved gases including oxygen, and electrochemical potential.

B. Results (D. V. Subrahmanyam)

1. Introduction to Present Work

The various alloying elements in an Fe-Ni-Cr alloy can alter the failure behavior in a given caustic medium significantly. The effect of alloying elements in sodium hydroxide solutions has been studied by Snowden,¹ Kagan and Mikharlova,² Truman and Perry,³ Akolzin,⁴ and Coriou et al.^{5,6} These investigations have usually been in 1 to 50% sodium hydroxide solutions at temperatures above 200°C. The present work will describe the results of studies with commercial austenitic stainless steels in boiling sodium hydroxide solutions. These alloys are being used until the equipment is deemed sufficiently reliable to begin work on the higher purity alloys.

2. Procedure

The alloys chosen for the present study are commercial stainless steels of Types 202, 304, 304L, 308, 309, 310, 316, 316L and

347. The nominal chemical compositions and yield stresses of these alloys are given in Table I. Wires of 0.38 mm were degreased in acetone and exposed to 50% boiling sodium hydroxide solutions (125-130°C) at a constant load equivalent to 150% of the yield stress. The experimental set-up is given in Fig. 1. Detailed procedure of loading the specimens are described in a previous report.⁷ The failure times were determined. The photomicrographs of failed specimens were taken in which the failure zone is included.

3. Results and Discussion

The effects of alloy composition on failure times of various austenitic stainless steels are given in Table II. It can be seen that the 202 alloy showed the lowest failure times (least resistance) and alloys of Type 308 and 310 containing higher combined nickel plus chromium did not fail in 15 days (maximum resistance). The individual effects are discussed below.

a. The effect of total concentration of Ni and Cr as plotted against failure time, is given in Fig. 2. This plot shows that the resistance of the alloy to stress corrosion increases with increasing Ni and Cr contents in the alloy. It can be seen that the alloys with Ni + Cr -- 29-33% (Type 308) and Ni + Cr -- 43-48% (Type 310) did not break after 15 days while an alloy with Ni + Cr -- 34-39% (Type 309 containing 22-24 Cr) broke in 176.6 hours (approx. 8 days). These patterns are not entirely self consistent but general trends are apparent with total Ni + Cr. The beneficial effects of increasing Ni and Cr are in agreement with observations by Snowden,¹ Kagan and Mikharlova,² Truman and Perry,³ Akolzin et al.⁴ and Coriou et al.⁵ It is generally accepted that the beneficial effect of Ni is determined by the chromium concentration in the alloy. Truman and Perry³ also observed the reversal in the trend of increasing resistance with increasing Ni and Cr at a chromium level of 20 and 25. However, to explain the observations of Truman and Perry³ and our observations, more detailed study in a narrow range of chromium and nickel contents is required.

b. The addition of molybdenum to Type 304 increased the life of the specimen slightly (Type 316) while the addition of columbium and tantalum reduced the life of the specimen (Type 347). The effect of adding carbon to Type 304 and Type 316 stainless steels did not produce significant effects on failure times. (Compare Type 304 with 304L and Type 316 with 316L).

In an experiment with Type 304 alloy it has been found that increasing the stress level reduced the life of the specimen.

Figure 3 shows the photomicrographs of the failed specimens. Physical examination of all the specimens, in general, showed a black coating over the surface which is presumably oxide. The photomicrographs revealed the following features:

TABLE I - COMPOSITIONS AND YIELD STRESSES OF AUSTENITIC
STAINLESS STEELS UNDER PRESENT STUDY

Alloy No.	COMPOSITION, %							Yield Stress, psi
	C	Mn	Si	P	S	Cr	Ni	
202*	0.15 (max.)	7.5-10.0	1.00	0.06	0.03	17.00-19.00	4.00-6.00	39,500
304	0.08 (max.)	2.00	1.00	0.065	0.03	18.00-20.00	8.00-12.00	20,600
304L	0.03 (max.)	2.00	1.00	0.065	0.03	18.00-20.00	8.00-12.00	21,200
308	0.08 (max.)	2.00	1.00	0.045	0.03	19.00-21.00	10.00-12.00	24,000
309	0.20 (max.)	2.00	1.00	0.045	0.03	22.00-24.00	12.00-15.00	26,300
310	0.25 (max.)	2.00	1.50	0.045	0.02	24.00-26.00	19.00-22.00	25,600
316**	0.08 (max.)	2.00	1.00	0.045	0.03	16.00-18.00	10.00-14.00	20,100
316L**	0.03 (max.)	2.00	1.00	0.045	0.03	16.00-18.00	10.00-14.00	25,700
347***	0.08 (max.)	2.00	1.00	0.045	0.03	17.00-19.00	9.00-13.00	28,000

*Nitrogen 0.25%

**Molybdenum 2.00-3.00%

***Cb-Ta: 10 x c, Min.

Fig. 1 - Experimental Arrangement (not drawn to scale)

TABLE II - EFFECT OF ALLOY COMPOSITION ON
FAILURE TIMES

Boiling 50 per cent sodium hydroxide (temp. 125-130°C)

Alloy No.	% of Yield Stress	Failure Time, hr.*
202	150	45.3
304	90	122.6
304	150	96.8
304L	150	94.2
308	150	360 -- not failed
309	150	176.6
310	150	360.0 - not failed
316	150	113.0
316L	150	105.0
347	150	81.3

*The failure times are the average of two experiments

Fig. 2 - Effect of Ni + Cr Concentration in the Time to Breaking of Wire Specimens Exposed to Boiling NaOH at 125°C and Stressed at 150% of the Annealed Yield Strength

(a) Type 202

(b) Type 304L

(c) Type 347

Fig. 3 - Photomicrographs of Specimens Exposed to Boiling NaOH
at 125 C and Stressed at 150% of Their Yield Stress 200X

(d) Type 316

(e) Type 316L

(f) Type 309

(g) Type 310

1. The alloys of Type 202, 309, 316, 316L and 347 underwent ductile fracture with some general or pitting corrosion.
2. The alloys of Type 304 and 304L showed some intergranular cracks besides ductile fracture.
3. The alloys Type 308 and 310, which did not fail in 15 days, showed very little general corrosion. The photomicrograph for Type 308 is not shown since it exhibited identical features.

This type of behavior differs from the observations reported in literature, where transgranular or intergranular, or, at least, mixed type of failure is observed. It is reasonable to expect this difference in mode of cracking of these alloys because of the high stress level; namely, 150% of yield stress in these constant load experiments.

Task 3 - General Effect of Alloy and Environmental
Composition in Sodium Base Environments
Containing NaOH on Stress Corrosion Cracking

A. Aims

The purpose of this task is to perform a parallel program to that in Task 2 except for the environment being a sodium base.

B. Results

None to date.

Task 4 - Dissolution and Passivation Behavior

A. Aims

This task is aimed at obtaining information on the kinetics of oxidation and reduction processes in electrochemically accessible environments. This includes aqueous and fused salt environments as opposed to liquid metals. This work will define those regions where activated dissolution and passivation occur as a function of alloy, temperature, and environmental composition. The definition of these regions provide guides to where stress corrosion might occur and also provide direct information on corrosion rates.

B. Results
(K. G. Sheth)

1. Introduction to Present Work

This study considers the phenomenology of electrochemical behavior of four iron-chromium-nickel alloys in sodium hydroxide solutions. The potentiodynamic technique has been used to obtain anodic polarization behavior.

The data were obtained using a potential controller to make the anodic polarization scans. An x-y recorder was used along with a potentiometer and electrometer to record the current and potential. The potential was continuously changed with the help of a motor potentiometer.

The description of the experimental apparatus, procedure, and the results of the investigations follows.

2. Experimental Apparatus and Procedure

Four high purity and homogeneous iron-chromium-nickel alloys were used. The composition of the alloys is given in Table III. (Other alloys will be studied later).

TABLE III - COMPOSITIONS OF THE ALLOYS USED
IN THE POLARIZATION

Alloy No.	Alloy Composition, wt.%		
	Fe	Cr	Ni
29	80.2	9.3	10.5
41	75.4	14.0	10.6
45	54.7	13.9	31.4
56	55.1	18.6	26.3

The specimens were cylindrical with 0.32 cm diameter. The length of specimens were taken in such a way to get the total surface area exposed to the solution equal to 1.0 cm². One end of the specimen was threaded as shown in Fig. 4. A stainless steel rod which was slipped into teflon tubing was threaded at one end and was then screwed into the specimen. Before screwing in, the specimen were abraded with emery paper ranging from 200-grit to 600-grit finish, washed with distilled water, and dried with acetone. This treatment of the specimens gave fairly reproducible results. The sodium

Fig. 4 - Schematic Diagram of Specimen Arrangement Used for Polarization Studies

hydroxide solution was prepared by using double distilled water and carbonate free saturated sodium hydroxide solution. A simplified diagram of the potentiostatic system is illustrated in Fig. 5. The working electrode is polarized continuously at a constant rate with the help of a motor potentiometer. Potentials and the current are recorded on a x-y recorder.

3. Results

Figure 6 shows the effect of scanning rate on the shape of anode polarization curve of an Fe-14Cr-10Ni alloy in 5.0 N NaOH solution. Figures 7-10 give the effect of alkali concentration on the corrosion potential, E_{corr} , and anode polarization on alloys No. 4A, 29, 41 and 45, respectively. The NaOH concentration was varied from 0.342 to 9.542N. It appears that the passive current increases with the increase in alkali concentration and the oxygen evolution potential (transpassive region) is shifted toward a more anodic side in alkali concentration. This is in accord with thermodynamic prediction. Figure 11 gives the anodic polarization curve for four binary Fe-Ni alloys in 1.0N sodium hydroxide. Although it is too early to conclude anything significant concerning the effect of alloying elements, it appears that increasing the concentration of chromium shifts the oxygen evolution potential toward more active potentials. The corresponding cathodic curves are given in Fig. 12. Anodic polarization curves for ternary alloys are shown in Fig. 13.

Task 5 - Effect of Environment on Mechanistic Behavior

A. Aims

The aim of this work is to find the effects of environmental variables on the course of mechanistic processes associated with caustic SCC. This work emphasizes effects of environmental chemistry as opposed to Task 7 which emphasizes effects of metallurgical variables. This work also helps to interpret work in Task 6.

B. Results

None to date.

Task 6 - The Straining Electrode and Transient Dissolution

A. Aims

In other work at OSU it has been shown that the straining electrode provides a good method for identifying those regions of electrochemical potential where SCC is most likely. The purpose of this task is to extend this understanding to caustic systems.

Fig. 5 - Schematic Arrangement of Equipment for Conducting Potentiostatic Polarization Experiments

Fig. 6 - Anodic Polarization Curves Showing Effect of Scanning Rate on the Fe-10Ni-15Cr Alloy in 5.0N NaOH at 25°C

Fig. 7 - Anodic Polarization Curves Showing Effect of NaOH Concentration on Polarization Behavior of Fe-25Ni (#4A). Scanning Rate was 200 mV/hr

Fig. 8 - Anodic Polarization Curves Showing Effect of NaOH Concentration on the Fe-10Ni-10Cr(Alloy 29) alloy at 25°C. The Scanning Rate is 200 mV/hr

Fig. 9 - Anodic Polarization Curves Showing Effect of NaOH Concentration on the Fe-10Ni-15Cr (Alloy 41) alloy at 25°C. The Scanning Rate is 200 mV/hr

Fig. 10 - Anodic Polarization Curves Showing Effect of NaOH Concentration on the Fe-30Ni-15Cr (Alloy 45) Alloy at 25°C. The Scanning Rate is 200 mV/hr

Fig. 11 - Anodic Polarization Behavior of Fe-Ni Binary Alloys in 1.0N NaOH at 25°C. Scanning Rate is 200 mV/hr

Fig. 12 - Cathodic Polarization Behavior of Fe-Ni Binary Alloys in 1.0N NaOH at 25°C. Scanning Rate is 200 mV/hr

Fig. 13 - Anodic Polarization Behavior of Fe-Cr-Ni Ternary Alloy in 1.0N NaOH at 25°C. Scanning rate is 200 mV/hr

B. Results

None to date.

Task 7 - Initiation and Propagation of Cracks

A. Aims

The aim of this work is to study initiation and propagation processes in single crystals of Fe-Cr-Ni alloys. Special attention is to be paid to the crystallography of initiation and fracture processes. This work emphasizes metallurgical variables.

B. Results

None to date.

Task 8 - Corrosion Processes in Crevices

A. Aims

The aim of this work is to define electrochemical processes in crevices associated with caustic environments. Crevice effects include accelerated corrosion as well as the formation of insoluble corrosion products which expand and exert substantial forces. Both of these processes can be very destructive.

B. Results

None to date.

Task 9 - Dissolution at Grain Boundaries

A. Aims

Caustic SCC frequently occurs intergranularly and substantial intergranular attack is observed frequently when alloys are exposed to caustic environments. The purpose of this work is to define the amount and mechanism of intergranular corrosion and also that portion which is accelerated by stress.

B. Results

None to date.

Task 10 - Local Dissolution Processes on
Caustic Environments

A. Aims

The aim of this work is to investigate initiation processes using thin foils which can be exposed to the environment, stressed, and subsequently examined in the electron microscope.

B. Results

None to date.

Task 11 - Metallurgical Structure

A. Aims

The susceptibility of many alloys to SCC can be drastically altered by changing the metallurgical structure. Such changes can be effected by cold work, precipitation hardening, special heat treatments and changes in alloy composition to produce certain phases. This work will consider possibilities for applying these procedures for preventing SCC.

B. Results

None to date.

Task 12 - Detailed Nature of the Passive Film

A. Aims

The aim of this task is to define the detailed aspects of density and structure of the protective film on the surface of Fe-Cr-Ni alloys in caustic environments. This effect is important because all chemical reactions and stress corrosion cracking are related directly to the chemical or mechanical breakdown of these protective films.

B. Results

None to date.

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1. Quarterly Report No. 1, COO-1018-4, January 15, 1969 through April 15, 1969.
2. Quarterly Report No. 2, COO-2018-5, April 16, 1969 through July 15, 1969.

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June 10, 1970