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

R. W. Staehle
Department of Metallurgical Engineering

October 15, 1969 - January 14, 1970

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QUARTERLY

REPORT

By

THE OHIO STATE UNIVERSITY
RESEARCH FOUNDATION

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On STRESS CORROSION CRACKING OF Fe-Cr-Ni ALLOYS
IN CAUSTIC ENVIRONMENTS

For the period October 15, 1969 - January 14, 1970

Submitted by R. W. Staehle
Department of Metallurgical Engineering

Date June 9, 1970

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1.0 ABSTRACT

Results from stress corrosion and electrochemical studies are reported for Fe-Cr-Ni alloy exposed to NaOH-H₂O solutions at temperatures below 200°C. Stress corrosion studies show intergranular-transgranular transitions as the alloys and applied potentials are varied. The electrochemical studies show that increasing temperature causes the protective films to dissolve more rapidly. Also the addition of chromium to Fe-Ni appears to be deleterious to the general corrosion resistance.

2.0 CONCLUSIONS

1. For iron-base alloys containing nickel and chromium additions, a variety of SCC behavior is observed in boiling 50% NaOH (135°C) solutions. It is possible to obtain intergranular-transgranular transitions by changes in the alloy composition (change Fe-11Ni-14Cr to Fe-31Ni-13Cr). For the Fe-11Ni-14Cr alloy, ductile behavior can be obtained by lowering the potential to -1054 mV_h.

2. Polarization studies over the 25-100°C temperature range and from 1.0-10.0N NaOH show that increasing the temperature causes a drastic reduction in the passivity. Further, additions of 20% chromium to Fe-Ni alloys substantially increases the corrosion rate in the same solutions.

3.0 INTRODUCTION

This work is being conducted in support of the LMFBR 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 form 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. Work performed on a given task is described. Note that work is not performed each quarter on all tasks owing to schedule for starting the respective task and also to vagaries in academic schedules for graduate students.

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 for the purpose of finding 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

None this Quarter.

Task 2 - General Effects of Alloy and Environmental Composition in Aqueous Solvents on Stress Corrosion Cracking (D.V. Subrahmanyam)

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

1. Effects of Temperature on Electrode Kinetics for Type 304 Stainless Steel

Some initial work on electrochemical kinetics was required to serve as a basis for these studies on Type 304 stainless steel. Such information is useful for determining the nature of the electrochemical regions in which cracking occurs.

Figure 1 shows the potential-time curves for unloaded and loaded (100% of y.s.) Type 304 stainless steel in boiling (135°) 50% NaOH. At this temperature the corrosion potentials shifted toward active values with time. This trend can be produced either by an increase in the anodic kinetics or a decrease in the cathodic kinetics. Which of these operates here is not clear. Although there were differences in the initial potentials, the alloy in the loaded and unloaded conditions reach about the same potential, i.e., -389 mV for unloaded and -369 mV for loaded specimens. A steady state potential for loaded Type 304 is obtained in 50 hours. While for the unloaded alloy the potentials drift slightly. At room temperature, the corrosion potentials shifted toward

Fig. 1 - Effect of time on open circuit corrosion potentials of 304 alloy in 50 per cent NaOH solution.

noble values as given below:

Time, (hr)	E_{corr}, mV_H
~ 0	-594
2	-434
20	- 94

From these trends it is reasonable to conclude that increasing the temperature accelerates the breakdown of the protective film.

The effect of temperature was investigated also by measuring the corrosion potential as temperature was progressively increased. The results of these measurements are plotted in Fig. 2. Time effects are negligible because the potential is unlikely to change by more than 20 mV in 20 hours. The difference of potential between room temperature and boiling point is 170 mV. The trend from Fig. 2 confirms those of the above paragraph.

Figure 3 summarizes the potentiostatic polarization curves for Type 304 alloy in boiling 50% sodium hydroxide. These curves were determined at 20 mV increments and waiting for three minutes at each potential. Tafel slopes of 60 mV/decade are observed. These curves show a very broad range of active dissolution extending from 1 to 1000 μA . Film formation is complete in a very narrow range of potentials. However these currents are high and do not constitute significant operational passivity.

2. Stress Corrosion of Type 304 Stainless Steel

Failure times were determined at -254 mV_H and at the corrosion potential, i.e., -369 mV_H. There was no failure in 15 days at all potential except at -254 mV_H. The currents at these potentials were between 10^{-6} to 10^{-4} A.

Figures 4-8 show the scanning electron micrographs for alloy 304 in boiling 50% sodium hydroxide after different test conditions.

The unloaded specimen, after 15 days exposure, reveals minor pitting attack (Fig. 4). A generalized attack is noticeable (Fig. 5a) with fine transgranular cracks in the oxide-coated areas (Fig. 5b) for the specimen loaded to 100% of y.s. under open circuit conditions ($E_{corr} = -369$ mV_H after 15 days). Note the two slip systems operating (lower right-hand corner). Figures 6a and 6b show the crack tip and stressed surface at a potential of -254 mV_H. Necking at the crack tip (Fig. 6a) indicates plastic deformation. The stressed surface (Fig. 6b) suggest the intergranular type of cracking. At a potential

Fig. 2 - Effect of temperature on corrosion potentials.

Fig. 3 - Potentiostatic polarization curve for alloy 304 in boiling 50 per cent NaOH solution by stepping method.

Fig. 4 - Type 304 stainless steel in
boiling 50% NaOH exposed for
15 days. 500X

(a) Clean surface

(b) Oxide-covered surface

Fig. 5 - Type 304 stainless steel in boiling 50% NaOH at load = 100% of Y.S.
(Final corrosion potential = -369 mV_H) 1000X

(a) Crack tip

(b) Cracked surface

Fig. 6 - Crack tip and stressed surface of type 304 stainless steel at $E_h = -254$ mV in boiling 50% NaOH. Load = 100% of Y.S. 200X

of -204 mV_H the intergranular cleavage still prevails with shallow pits inside the grains (Fig. 7a). These features are magnified in Fig. 7b. At a potential of -154 mV_H very generalized attack can be seen in Fig. 8.

3. Preliminary Stress Corrosion Studies on Other Fe-Cr-Ni Alloys

Preliminary electrochemical measurements and stress corrosion studies of three specially melted alloys were initiated. The compositions of these alloys along with corrosion potentials, tensile properties, and SCC times are included in Table I. These alloys have approximately the same chromium concentration but have a wide range of nickel concentrations. Galvanostatic experiments in boiling 50% NaOH were conducted for the three alloys and the results are given in Fig. 9. The current-time curves for the Fe-11Ni-14Cr alloy at two potentials are shown in Fig. 10. The two potentials in this curve were selected to be in the active and passive ranges.

TABLE I - Alloy Properties and Test Results

Alloy No.	%*		Yield Strength (psi)	Failure Time at 100% of y.s. (hr)**	Corrosion *** Potential (mV _H)
	Cr	Ni			
32	13.5	31	16,000	142,261	788
33	9.1	46	22,000	360,NF	780
41	14.2	10.96	93,200	24	920

* Balance iron

** Boiling 50% Na/OH at 135°C

*** Unloaded but in boiling 50% NaOH at 135°C

Scanning election micrographs of failed specimens are shown in Figs. 11 and 12. Figure 11 shows the Fe-31Ni-13Cr alloy which broke in 142 hours at 100% of the yield stress in boiling 50% NaOH. The cracking is clearly transgranular and exhibits the quasi-cleavage fractographic pattern. Figure 12 shows a Fe-11Ni-14Cr alloy exposed to boiling 50% NaOH at 100% of its yield strength at 17 μA anodic current. The failure time was 69 hours. Here the cracking is intergranular and a considerable amount of necking is observed. At -1054 mV_H the Fe-11Ni-14Cr alloy exhibits a totally ductile behavior as shown in Fig. 13. This represents a substantial shift in behavior from that of Fig. 12 which operated in a range of -200 to 400 mV_H .

(a) 500X

(b) 5000X

Fig. 7 - Clean surface of Type 304 stainless steel at $E_h = -204$ mV in boiling 50% NaOH.
Load = 100% of Y.S.

Fig. 8 - Clean surface of Type 304 stainless steel at $E_h = -154$ mV in boiling 50% NaOH. Load = 100% of Y.S. 200X

Fig. 9 - Galvanostatic anodic polarization in boiling NaOH solution.

Fig. 10 - Current-time curves for Fe-11Ni-14Cr in boiling 50% NaOH at 100% of Y.S.

(a) Fractional surface at 200X

(b) Stressed surface at 5000X

Fig. 11 - Scanning electron micrographs of Fe-31Ni-13Cr alloy exposed to boiling 50% NaOH at 100% of Y.S.

(a) 200X

(b) 2000X

Fig. 12 - Scanning electron micrographs of Fe-11Ni-14Cr alloy in boiling 50% NaOH. Exposed at 100% of Y.S. at 17 μ A applied current. Failure time = 69 hours.

(a) 200X

(b) 2000X

Fig. 13 - Scanning electron micrographs of Fe-11Ni-14Cr alloy in boiling 50% NaOH. Exposed at 100% of Y.S. at $E_h = -1054$ mV.

4. Discussion

Conclusions are not easily drawn from the foregoing data because sufficient experiments have not yet been conducted to ascertain trends. It is interesting to note that a wide range of failure modes is exhibited as shown by fractography. It is further surprising that these alloys lose their passivity so easily under the boiling condition.

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

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 this quarter.

Task 4 - Dissolution and Passivation Behavior (K. G. Sheth)

A. Aims

This task is aimed at obtaining information on the kinetics of oxidation and reduction processes in electrochemically accessible (i.e., ionically conducting) 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 will provide a guide to where stress corrosion might occur and also will provide direct information on corrosion rates.

B. Results

The present work is investigating the effects of temperature, pH, and alloy concentration on the polarization behavior of Fe-Ni and (Fe + 20Cr) - Ni Alloys. Composition of various alloys used for this investigation is given in Table II. These alloys were selected with a view in mind to study the effect of nickel in Fe-Ni.

TABLE II - Chemical Composition of Experimental Alloys

Alloy No.	Components, wt. %			
	Fe	Ni	Cr	Others
2	94.8	5.2	-	-
3	89.5	10.5	-	-
4	79.2	20.8	-	-
4a	74.1	25.9	-	-
12	-	100	-	-
51	81.1	-	18.9	-
55	60.2	21.1	18.7	-
58	34.7	46.9	18.4	--
62	-	81.9	18.1	-

The corrodent or the electrolyte used for these investigations was 1.0, 5.0 and 10.0N sodium hydroxide. Temperatures studied were in the range 25-90°C.

The results of anodic and cathodic polarization curves of these alloys are shown in Figs 14-27. These figures are arranged together in groups to facilitate the understanding of the effects of composition, temperature, and solution concentration. Figures 14-16 give the effect of nickel addition on the anodic behavior of Fe-Ni binary alloys in various alkali concentration. Figure 17 gives the effect of nickel on anodic behavior of Fe-20Cr-Ni alloys. It can be seen from these figures that the addition of nickel shifts the corrosion potential slightly toward more positive values. In the case of dilute alkali and lower temperature range the corrosion potential is the passive range. With increases in alkali concentration or temperature (Fig. 16) the potentials shift to a more active region and an active peak is also observed. It can also be observed that replacing nickel with chromium in the Fe-Ni binary considerably increases its corrosion rate, especially in high alkali concentration (Fig. 17). This is in fair agreement with Pourbaix diagrams showing the comparatively high solubility of Cr oxide compared to that of Ni oxides in high potential-pH regions. Addition of nickel reduces the passive currents and, in general, shifts the oxygen evolution

Fig. 14 - Anodic polarization behavior in 1.0N NaOH at 25°C

Fig. 15 - Anodic polarization behavior in 5.0N NaOH at 25°C

Fig. 16 - Anodic polarization behavior in 10.0N NaOH at 50°C

Fig. 17 - Anodic polarization behavior in 10.0N NaOH at 25°C

Fig. 18 - Cathodic polarization behavior in 1.0N NaOH at 25°C

Fig. 19 - Cathodic polarization behavior in 5.0 NaOH at 25°C

Fig. 20 - Cathodic polarization behavior in 10.0N NaOH at 25°C

Fig. 21 - Effect of temperature on anodic polarization behavior of alloy-2 in 10.0N NaOH

Fig. 22 - Effect of temperature on anodic polarization behavior of alloy-3 in 10.0N NaOH

Fig. 23 - Effect of temperature on anodic polarization behavior of alloy-4 in 10.0N NaOH

Fig. 24 - Effect of temperature on anodic polarization behavior of alloy-58 in 10.0N NaOH.

Fig. 25 - Effect of temperature on cathodic polarization behavior of alloy-3 in 10.0N NaOH

Fig. 26 - Effect of temperature on cathodic polarization behavior of alloy-58 in 10.0N NaOH

Fig. 27 - Effect of alkali concentration on anodic polarization behavior of alloy-3 at 25°C

potential to more active direction though the change is minimal. Figures 18-20 show the cathodic polarization behavior of Fe-Ni and Fe-Ni-Cr alloys. Here also, as in the case of anodic polarization, addition of nickel reduces the passive current, and shifts the hydrogen evolution potential toward slightly positive value. The cathodic curves were analyzed to determine the Tafel slopes for the hydrogen evolution reaction. The values for the slope(b) for cathodic evolution of hydrogen and anodic evolution of oxygen are given in Table III. Tsirman* has, in the case of iron, reported the value of slope(b) for anodic evolution of oxygen to be 46-49 mV/decade. These values are in fair agreement with observed values.

TABLE III - Tafel Parameters

Alloy No.	Alkali Conc., M	b_a , mV	b_c , mV
2	1.0	45	90
2	10.0	45	-
3	1.0	45	90
3	10.0	45	-
4	1.0	45	90
4	10.0	50	-
51	10.0	50	0.110
55	10.0	50	0.110
58	10.0	50	0.110

* A. I. Tsirman, Zh. Fiz. Khim., 37, 1598 (1963).

Figures 21-26 show the effect of temperature on anodic and cathodic polarization behavior of all the above alloys. In case of Fe-Ni binary alloys at room temperature, the corrosion potential is in the passive region and a critical current peak required for passivation is not observed. However, as the temperature is raised to 50°C and above, the corrosion potential appears to shift toward a more active direction, and an anodic current peak is observed (Figs. 21-23). Increases in temperature also shift the oxygen evolution potential toward the more active direction.

The effect of alkali concentration is shown in Fig. 27. Increases in alkali concentration increases the passive current and shifts the anodic oxygen evolution potential toward more active direction. This is also in good agreement with theoretical predictions.

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 this quarter.

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.

B. Results

None this quarter.

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 this quarter.

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 this quarter.

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 this quarter.

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 this quarter.

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 this quarter.

Task 12 - Detailed Nature of the Passive Film

A. Aims

The aim of this task is to define the detailed aspects of chemistry 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 this quarter.

Investigator.....

Date.....

Supervisor.....

Date.....

For The Ohio State University Research Foundation

Executive Director.....

Date.....