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ELECTROCHEMICAL CORROSION STUDIES OF SNAP CONTAINER MATERIALS

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by

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**U.S. NAVAL RADIOLOGICAL
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ABSTRACT

Corrosion rates of Haynes 25, Hastelloy C and Hastelloy N in natural seawater were determined by galvanostatic polarization techniques. Values of approximately 10^{-2} mils per year were obtained for each of the alloys. No significant effect of aeration and solution stirring on the corrosion rates was observed.

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SUMMARY

A program to study the release of radionuclides from nuclear power sources to an ocean environment requires knowledge of the corrosion rates in seawater of the material containing the radionuclide. To obtain an initial indication of the corrosion rates of certain container materials, Hastelloy C, Hastelloy N and Haynes-25 alloys, electrochemical polarization studies in seawater were conducted on them.

The results indicate that the corrosion rate is of the order of 10^{-2} mils per year for all three alloys. No significant effect of aeration, deaeration, and solution stirring on the corrosion rates was observed.

INTRODUCTION

For several years this laboratory has been investigating the release of radionuclide fuels employed in operational and proposed Systems for Nuclear Auxiliary Power (SNAP) to the seawater environment.

The work was initially concerned with the seawater reactions and solubility rates of fuel forms used in SNAP radionuclide power devices.^{1,*} The program was subsequently broadened to include studies of the release of fission products from irradiated nuclear power reactor fuels to seawater,^{2,3,4,**} and measurement of the corrosion rates in seawater of the material containing the SNAP radionuclides. The container materials considered for this initial corrosion study were Haynes 25, Hastelloy C and Hastelloy N.

Because of the expected low corrosion rates of these alloys, especially sensitive techniques of corrosion rate measurement were required. The use of three such techniques was instituted: 1) measurement of the concentration of radioactivity leached into the seawater from nuclear reactor-activated alloy specimens, 2) activation analysis of corrosion products in seawater in which inactive alloy specimens have been placed (to determine whether the gamma recoil from the thermal neutron activation of the alloys in the first technique has affected their corrosion properties), and 3) established electro-chemical techniques.

* M.G. Lai and H.A. Goya, "Radionuclide Release From Radionuclide Power Sources. II. Release From Plutonium Metal to Seawater", U.S. Naval Radiological Defense Laboratory, USNRDL-TR- (in preparation).

**L. W. Weisbecker, "Radionuclide Release From Aero-Space Nuclear Reactor Fuels. IV. Phase Two: Safeguards Analysis of Pulsed Neutron Irradiation of Fuel in Water", U.S. Naval Radiological Defense Laboratory, USNRDL-TR- (in preparation).

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The data obtained by these techniques will be compared in a later report. Differences in the experimental conditions will be accounted for in the comparison.

The present report describes a study of the corrosion rates of Haynes 25, Hastelloy C and Hastelloy N in seawater using electrochemical techniques only.

BACKGROUND

Anodic and cathodic polarization of the metal by an externally applied current are the basic electrochemical measurements which provide data for the study of the corrosion behavior of a metal in a corrosive solution. The magnitude of the polarization is expressed in terms of a displacement of the metal electrode potential from its equilibrium (or corrosion) potential by the applied current. This displacement of potential is termed overvoltage. Electrochemical polarization data, in the form of plots of overvoltage versus current, can be obtained in two ways depending on whether the potential or the current is chosen as the independent variable. When potential is the independent variable the term "potentiostatic polarization" applies; when current is the independent variable the term "galvanostatic polarization" applies. The type of polarization used to obtain the present results was galvanostatic.

For low values of applied current, expressed in units of amp/cm^2 of metal electrode surface and referred to as current density, the dominant process controlling polarization is the reaction occurring at the electrode. For values of overvoltage greater than about 50 millivolts the relation between overvoltage and current density in this region is

$$E = \beta \log \frac{i}{i_0} \quad (1)$$

where E represents the magnitude of polarization in volts (i.e., the overvoltage), β and i_0 are constants for a given system and i is the applied current density. Equation 1 is referred to as the Tafel equation*, β is the Tafel slope when E is plotted against $\log i$, and i_0 is the exchange current density. At higher values of i the electrode

*Named after J. Tafel who first proposed a similar equation in 1904 to express hydrogen overvoltage as a function of current density. (Z. physik. Chem. 50:641 (1904)).

reaction becomes diffusion-controlled, and a plot of E against log i becomes non-linear.

Stern and Geary^{5,6} have shown that when a system can be described by Eq. 1 the following relation applies when the overvoltage is less than about 50 millivolts.

$$\Delta E/\Delta i = \frac{\beta_a \beta_c}{2.3 i_{\text{corr}} (\beta_a + \beta_c)} \quad (2)$$

where the quantity $\Delta E/\Delta i$ is the slope near the region of the corrosion potential for which the change of E with i is linear, β_a and β_c refer to the Tafel slopes for anodic and cathodic polarization, respectively and i_{corr} is the corrosion current in units of amp/cm². This relation is valid for both anodic and cathodic polarization. However, in practice the cathodic polarization curve is used since it is experimentally easier to obtain in the region below 50 millivolts. i_{corr} is calculated from Eq. 1 and 2 using the experimentally determined values of β_a , β_c and $\Delta E/\Delta i$.

EXPERIMENTAL

Polarization Equipment

The current was measured by determining the voltage drop across a precision 10 megohm or 1 megohm (for higher values of current) resistance with a Keithley 610BR electrometer. The potential of the sample electrode versus a saturated calomel electrode (S.C.E.) was measured with a high-input-impedance John Fluke differential voltmeter, model 803B. The electrometer had a stated accuracy of $\pm 1\%$ of full scale on all ranges. The voltmeter had a stated accuracy of $\pm 0.05\%$ of the input voltage from 0.1 to 500 VDC and $\pm (0.05\% + 50 \text{ microvolts})$ of the input voltage below 0.1 VDC. The power supply used was a Trygon Electronics Model S36-5. It had a stated stability of 0.05% or 18 millivolts over an 8-hour period. A diagram of the circuit used is shown in Fig. 1.

Fig. 1 Galvanostatic polarization circuit.

Polarization Cell

The polarization cell was a standard 1-liter, 4-neck, round-bottom, pyrex flask modified by a ball socket on one side to provide access for a Luggin capillary probe and saturated calomel electrode (S.C.E.) assembly. Two Beckman # 39271 platinum thimble electrodes were used as the auxiliary electrodes (Fig. 1). Test gases, either nitrogen or air, were introduced into the cell by a fritted pyrex glass bubbler.

The specimens of Haynes 25, Hastelloy C and Hastelloy N were fabricated into electrodes from rods obtained from Stellite Division, Union Carbide Co. Table 1 shows the manufacturer's analysis of the alloys. The rods were machined into right circular cylinders 0.5 inches in height and 0.25 inches in diameter. One end was drilled in each cylinder and a tapped stainless steel sleeve inserted for mounting on the electrode assembly, shown in Fig. 2. The complete polarization cell is shown in Fig. 3. In experiments where solution stirring was desired, a magnetic mixer was used.

Seawater

The seawater used in these experiments was collected near the Farallon Islands, located about 20 miles off the coast of northern California, by a previously described method. Before use the seawater was filtered through a millipore filter (0.8 micron).

PROCEDURE

Each electrode was polished with #2, #0 and #00 emery paper, in that order. After it was mounted on the electrode assembly, the electrode was cleaned in benzene, then in ethanol with the use of an ultrasonic cleaner. After a final rinse in distilled water, the electrode was placed in the polarization cell containing seawater previously saturated with either nitrogen or air. The same gas was kept bubbling through the seawater throughout the experiment.

After the sample electrode had attained a steady state corrosion potential, polarization measurements were conducted by applying current from the power supply in increasing increments at regular time intervals, usually about 5 min. For each current value the corresponding potential of the sample electrode versus S.C.E. was noted. All experiments were conducted at ambient room temperature (Ca 23°C).

TABLE 1
Manufacturer's Composition and Analysis of Test Alloys

Hastelloy C

Cr - 15.38%	Mn - .44%
W - 3.50	V - .19
Fe - 5.40	Mo - 15.94
C - .05	P - .013
Si - .67	S - .066
Co - 1.05	Ni - (57.30) balance

Hastelloy N

Cr - 6.42%	Mo - 16.22%
W - .06	P - .003
Fe - 3.93	S - .009
C - .07	Al - .01
Si - .56	Ti - .01
Co - .12	B - .006
Mn - .52	Cu - .01
V - .23	Ni - (71.82) balance

Haynes 25

Cr - 20.45%	Ni - 9.93%
W - 15.10	Al - 1.03
Fe - 1.95	P - .016
C - .07	S - .011
Si - .07	Co - (51.37) balance

Fig. 2 Specimen electrode assembly.

Fig. 3 Polarization cell

RESULTS AND DISCUSSION

Figures 4 through 11 show the plots of E against $\log i$ obtained for each of the three alloys under various conditions of aeration, deaeration, and solution stirring. In most instances the Tafel region was not too well-defined and consequently the determination of the Tafel slope by the best straight line was somewhat subjective, even when the points for the higher values of current were neglected. This subjectiveness is not reflected too severely in calculating values of i_{corr} since the term $\beta_a \beta_c / (\beta_a + \beta_c)$ in Eq. 2 is fairly insensitive to small variations in values of β_a and β_c .

On the other hand, the linear plots of E against i for cathodic polarization gave relatively good straight lines for all the alloys under all conditions. A representative plot is shown in Fig. 12 for Hastelloy C.

The most difficult measurement to obtain was the steady state corrosion potential, E_{corr} . The time required for the sample electrodes to reach a value of E_{corr} stable enough to enable polarization measurements to be made (less than a few millivolts change in an hour) was at least 15 to 20 hours after they were immersed in the seawater. In addition, after a polarization run had been completed and the power leads had been disconnected, the electrode would not return to the potential it had prior to being polarized, but would instead stabilize at a new potential which was less negative with respect to the S.C.E. The values of E_{corr} reported are those obtained before polarization. The "initial" values of E_{corr} in deaerated unstirred seawater were -0.219V, -0.155V, and -0.132V for Hastelloy C, Hastelloy N and Haynes 25, respectively. The corresponding values in aerated seawater were -0.168V, -0.125V, and -0.052V.

In deaerated seawater solutions, stirring had little effect on E_{corr} for Haynes 25 and Hastelloy N. For Hastelloy C, E_{corr} became approximately 50 millivolts less negative than without solution stirring. For aerated seawater E_{corr} for all three alloys became approximately 50 millivolts less negative with solution stirring.

Table 2 summarizes the polarization data and presents the values of the corrosion rates, expressed in mils per year (mpy), for each of the alloys. i_{corr} was converted to mil per year using Faraday's Law

Fig. 4 Galvanostatic cathodic polarization curves for Haynes 25, Hastelloy C and Hastelloy N in deaerated, stirred seawater.

Fig. 5 Galvanostatic cathodic polarization curves for Haynes 25, Hastelloy C and Hastelloy N in deaerated, unstirred seawater.

Fig. 6 Galvanostatic cathodic polarization curves for Haynes 25, Hastelloy C and Hastelloy N aerated, stirred seawater.

Fig. 7 Galvanostatic cathodic polarization curves for Haynes 25, Hastelloy C and Hastelloy N in aerated, unstirred seawater.

Fig. 8 Galvanostatic anodic polarization curves for Haynes 25, Hastelloy C and Hastelloy N in deaerated, stirred seawater.

Fig. 9 Galvanostatic anodic polarization curves for Haynes 25, Hastelloy C and Hastelloy N in deaerated, unstirred seawater.

Fig. 10 Galvanostatic anodic polarization curves for Haynes 25, Hastelloy C and Hastelloy N in aerated, stirred seawater.

Fig. 11 Galvanostatic anodic polarization curves for Haynes 25, Hastelloy C and Hastelloy N in aerated, unstirred seawater.

Fig. 12 Linear plot of cathodic overvoltage vs. current density for Hastelloy C in aerated, stirred seawater.

TABLE 2
Summary of Electrochemical Data

Conditions*	β_c (v)	β_a (v)	$\Delta E/\Delta i$ (v/ μ amp/cm ²)	i_{corr} (μ amp/cm ²), $\times 10^{-2}$	Corrosion Rate (mpy), $\times 10^{-2}$
<u>Hastelloy C</u>					
A, NS	.110	.155	2.15	1.3	2.7
A, S	.115	.171	4.45	.70	1.4
D, S	.376	.223	4.07	1.5	3.1
D, NS	.250	.145	2.58	1.6	3.3
D, NS	.332	.235	6.80	.88	1.8
D, NS	.325	.230	4.55	1.2	2.5
(Ave. for D,NS)				(1.2 $\pm$.4)	(2.5 $\pm$.8)
<u>Hastelloy N</u>					
A, NS	.183	.207	7.00	.60	1.4
A, S	.130	.113	2.25	1.2	2.9
D, S	.585	.231	4.37	1.7	4.1
D, NS	.600	.162	4.10	1.4	3.4
<u>Haynes 25</u>					
A, NS	.132	.352	5.60	.75	1.4
A, S	.135	.205	10.5	.34	.64
D, S	.680	.180	6.2	.99	1.9
D, NS	.300	.120	6.6	.56	1.1

* A, NS = aerated, no stirring; A, S = aerated, stirring; D, NS = deaerated, no stirring; D, S = deaerated, stirring.

and the assumption that the equivalent weights of the alloys are half their molecular weights (since bivalent ions would be expected from the major components). Included in Table 2 are the results of three runs, using Hastelloy C in unstirred deaerated seawater, made to obtain a measure of reproducibility.

The results shown in Table 2 indicate that within the experimental error, there is no significant effect of aeration and solution stirring on the corrosion rates of the alloys. The average value of the corrosion rate for all the stirred solutions is $(2.3 \pm 1.0) \times 10^{-2}$ mpy* and for the nonstirred solutions is $(2.1 \pm 0.7) \times 10^{-2}$ mpy. The average value of the corrosion rate for all the aerated solutions is $(1.7 \pm 0.6) \times 10^{-2}$ mpy and for the deaerated solutions is $(2.7 \pm 1.0) \times 10^{-2}$ mpy.

The averaged results for the three different alloys were: Hastelloy C, $(2.4 \pm 0.8) \times 10^{-2}$ mpy; Hastelloy N, $(3.0 \pm 1.2) \times 10^{-2}$ mpy; and Haynes 25, $(1.2 \pm 0.4) \times 10^{-2}$ mpy.

The value for Hastelloy C compares well with a literature value of 10^{-2} mpy.⁸ No literature values could be found for the other two alloys.

As mentioned above, values of E_{corr} were difficult to obtain. The difference in the steady state potential before and after polarization varied from about 20 millivolts to as much as 150 millivolts. This observation, and the consistency of the corrosion rates shown in Table 2, indicate that the corrosion rate is essentially independent of E_{corr} ; at least within the range of variation of E_{corr} observed here.

CONCLUDING REMARKS

Since the results presented here were obtained under laboratory conditions it is not intended that they be extrapolated to an actual ocean environment. Rather, these results serve to provide a baseline to which results of future studies, more realistic in nature, can be compared. The results presented here are based on the assumption that the corrosion is uniform. Pitting or localized corrosion would be undetected by this method.

* The value of the percentage standard deviation for each value of the corrosion rate is assumed to be the same as the measured value determined for the triplicate deaerated, nonstirred solution of Hastelloy C (Table 2).

Perhaps of more importance, these results show the feasibility of using electrochemical techniques to provide indications of the corrosion behavior of container alloys under various conditions. These conditions include use of thermally shocked specimens (to simulate reentry heating and subsequent quenching in the ocean) and use of galvanically coupled specimens of the container alloy with other metal components of the container. The results obtained from these studies will provide an indication of what may be expected from long-term ocean exposure studies.

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