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USNRDL-TR-67-64

16 May 1967

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**CORROSION STUDIES OF HAYNES 25 ALLOY IN
SEAWATER USING ELECTROCHEMICAL TECHNIQUES**

CESAT PRICES

by

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HQ \$ 3.50, MN 65

**U.S. NAVAL RADIOLOGICAL
DEFENSE LABORATORY**

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ADMINISTRATIVE INFORMATION

The work reported is part of a project sponsored by the Atomic Energy Commission under Contract No. AT(49-5)-2084.

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ABSTRACT

Electrochemical corrosion rate measurements have been made on uncoated, emissively coated and thermally shocked and uncoated specimens of Haynes 25 alloy in seawater at room temperature and 90°C. The corrosion rates of the emissively coated specimens were about an order of magnitude higher than those of the uncoated specimens. No significant difference between the corrosion rates measured at room temperature and at 90°C was observed for either the uncoated or the emissively coated specimens. Thermal shocking of uncoated specimens (by quenching from 500°C to room temperature) did not increase their corrosion rates. The corrosion rates observed were on the order of 3×10^{-3} mils per year (mpy) for the uncoated and the thermally shocked uncoated specimens and 4×10^{-2} mpy for the emissively coated specimens.

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SUMMARY

The Problem

Haynes 25 alloy has been selected for use as the fuel container material in several SNAP systems for space applications. Because of possible accident situations which could place the fuel container in a seawater environment, it is necessary to examine the corrosion behavior of this material in seawater to fully assess the radiological hazard from the radionuclide fuel.

Findings

The present report describes the electrochemical corrosion rate measurement of uncoated, emissively coated, and thermally shocked uncoated specimens of Haynes 25 in seawater at room temperature and at 90°C.

No significant difference between the corrosion rates measured at room temperature and at 90°C was observed for either the uncoated or the emissively coated specimens. Thermal shocking of uncoated specimens did not increase their corrosion rates. The corrosion rates observed were on the order of 3×10^{-3} mils per year (mpy) for the uncoated and the thermally shocked uncoated specimens and 4×10^{-2} mpy for the emissively coated specimens.

I. INTRODUCTION

For the past several years, this Laboratory (USNRDL) has been investigating the potential release of radionuclide fuels to the seawater environment from re-entering space SNAP (Systems for Nuclear Auxiliary Power) units, operational and proposed.

This program requires, in addition to the measurement of the dissolution rate of the radionuclide fuels themselves in seawater, a knowledge of the seawater corrosion rate of the fuel container material. Previous reports have described the measurement of the corrosion rates of Haynes 25, Hastelloy C and Hastelloy N in seawater by the application of electrochemical and radioactive tracer methods.^{1,2} These studies, on polished specimens, were carried out at or below room temperature. The present report describes the electrochemical corrosion rate measurement of polished (uncoated), emissively coated, and thermally shocked uncoated specimens of Haynes 25 in seawater at room temperature and at 90°C.* The emissive coating, nominally 60 % iron titanate, 30 % calcium titanate and 10 % silica, serves to increase the thermal emissivity of the fuel capsule during re-entry, and hence prevent burn-up.

II. EXPERIMENTAL

The techniques and equipment used to perform the galvanostatic polarization measurements have been described in an earlier report.¹ The specimens of Haynes 25 were fabricated here into electrodes from bar stock obtained from the Stellite Division, Union Carbide Company, Kokomo, Indiana. Table 1 shows the manufacturer's analysis of the alloy.

*The exposure condition of 90°C was examined since it is possible that the fuel in the container may raise the temperature of the local seawater environment this high. The thermally shocked specimens were used to simulate the result of re-entry heating and subsequent seawater quenching of the fuel container.

TABLE 1

Manufacturer's Chemical Analysis of Haynes 25 Alloy

Cr	19.60 %	Ni	10.73 %
W	14.70	Mn	1.24
Fe	2.65	P	0.016
C	0.15	S	0.010
Si	0.04	Co	balance (50.86)

The test specimens were fabricated in the shape of a right circular cylinder 0.5 in. in height and 0.25 in. in diameter with a 4-40 threaded stud extending from one end for mounting on the electrode assembly of the polarization apparatus. After fabrication each specimen was polished successively with #2, #0 and #00 emery paper and then sent to the Space and Missiles Division of General Electric Company, Valley Forge, Pennsylvania, for application of the emissive coating. In addition to completely coated specimens, specimens coated over only half their surface were also prepared to determine if there would be any difference in corrosion behavior between them and the completely coated or the uncoated specimens. For the half-coated specimens, the emissive coating was applied to the bottom half of the specimen (the top being the end with the threaded stud).

The uncoated test specimens used for the thermal shock experiments were heated to 500°C in a muffle furnace and quenched in seawater before being run.

The seawater used for the corrosion tests was aerated or deaerated by continuous purging with air or nitrogen, respectively. In instances where experiments were conducted in heated seawater, the pyrex vessel containing the sample was placed in a heating mantle. The power to the mantle was controlled by a variac. The temperature, monitored by a dial type Weston thermometer, was $(90 \pm 2)^{\circ}\text{C}$ for the heated samples. Room temperature was about 23°C.

III. THEORY

Values of the corrosion current, i_{corr} , were calculated from the following considerations.

For low values of applied current, i , expressed in units of amperes per square centimeter of the metal electrode surface (i.e., the test specimen of Haynes 25) and referred to as the current density, the dominant process controlling polarization is the reaction occurring at the electrode. For values of overvoltage (i.e., the magnitude of the displacement, caused by the applied current, of the electrode potential from its equilibrium potential) greater than about 50 millivolts the relation between overvoltage and current density is

$$E = \beta \log i/i_0 \quad (1)$$

where E represents the overvoltage*, β and i_0 (the exchange current density) are constants for a given system and i is the applied current density. Eq. (1) is referred to as the Tafel equation** and β is the Tafel slope, determined by plotting E against $\log i$.

At high values of i the electrode reaction becomes diffusion-controlled and a plot of E against $\log i$ becomes non-linear.

Stern and Geary^{3,4} have shown that for a system describable by Eq. (1) (i.e., for low values of applied current) the following relation applies if the overvoltage is less than about 50 millivolts.

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

The quantity $\Delta E/\Delta i$ is the slope near the region of the equilibrium potential for which the change of E with i is linear, and β_a and β_c refer to the Tafel slopes for anodic and cathodic polarization, respectively. This relation is valid for both anodic and cathodic polarization but in practice the cathodic polarization curve is used since it is experimentally easier to obtain in the region below 50 millivolts. i_{corr} was calculated from Eq. (2) and (1) by use of the experimentally determined values of β_a , β_c and $\Delta E/\Delta i$.

Values of i_{corr} were also obtained by extrapolation of the cathodic Tafel slope, β_c , to the equilibrium potential of the test electrode. The point of intersection represents the dissolution rate of the metal electrode and hence its corrosion rate in terms of current density.

* Either anodic or cathodic.

**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)).

IV. RESULTS AND DISCUSSION

Figures 1 and 2 show the Tafel curves (E vs. log i plots) obtained for uncoated specimens of Haynes 25 in 90°C seawater. Figures 3 through 8 show the Tafel curves obtained for half-coated and completely coated specimens of Haynes 25 under various conditions of seawater temperature and aeration. Figures 9 and 10 show the Tafel curves obtained for the uncoated thermally shocked specimens of Haynes 25. Values for β_a and β_c were obtained (at overvoltages > 0.05 v) from Figs. 1 through 10.

In most instances the determination of the Tafel slopes, β_a and β_c , was somewhat subjective. This subjectiveness does not affect the values of i_{corr} beyond a factor of about two since the term $\left(\frac{\beta_a \beta_c}{\beta_a + \beta_c}\right)$ in Eq. (2) is relatively insensitive to small variations in the values of β_a and β_c .

The linear plots of E vs. i for cathodic polarization gave relatively good straight lines in all instances. Representative plots are shown in Figs. 11 through 13 for uncoated, coated and thermally shocked specimens of Haynes 25. The value for $\Delta E/\Delta i$ for each situation was obtained from the appropriate plot of the type shown in Figs. 11 through 13. i_{corr} was then calculated for each situation, with the use of the appropriate values of β_a and β_c .

Table 2 summarizes the polarization data and presents the values of the corrosion rates expressed in terms of mils per year (mpy). Values of i_{corr} and the corresponding corrosion rates obtained graphically are shown in parenthesis. These corrosion rates were calculated from the values of the corrosion current by application of Faraday's Law and the assumption that bivalent ions are formed as the main corrosion products.

The values of i_{corr} obtained by use of Eq. (2) are considered better than the values of i_{corr} obtained graphically since the use of Eq. (2) does not reflect the subjectiveness of the Tafel slope as much as the graphical extrapolation does.

The corrosion rate found for the uncoated specimen of Haynes 25 in 90° seawater, 2.6×10^{-3} mpy (2.0×10^{-3} mpy), compares well with the value found in room temperature seawater, 3.5×10^{-3} mpy.^{1,*} The fact that the rate at 90°C is not higher than the rate at room temperature is probably due to formation of a stable surface film. It is noteworthy

*A computational error was discovered in the corrosion rates reported in Ref. 1. The reported rates for Hastelloy C, Hastelloy N and Haynes 25 are $(2.4 \pm 0.8) \times 10^{-2}$, $(3.0 \pm 1) \times 10^{-2}$ and $(1.2 \pm 0.4) \times 10^{-2}$ mpy, respectively. The corrected rates are $(6.0 \pm 2) \times 10^{-3}$, $(5.1 \pm 2) \times 10^{-3}$ and $(3.5 \pm 1) \times 10^{-3}$ mpy, respectively. These values now agree more closely with the values, determined by radiochemical techniques, reported in Ref. 2.

Fig. 1 Anodic Polarization Curve for Uncoated Haynes 25 in 90°C Seawater

Fig. 2 Cathodic Polarization Curve for Uncoated Haynes 25 in 90° Seawater

Fig. 3 Anodic Polarization Curves for Half and Completely Emmissively Coated Haynes 25 in Aerated Seawater at Room Temperature

Fig. 4 Cathodic Polarization Curves for Half and Completely Emissively Coated Haynes 25 in Aerated Seawater at Room Temperature

Fig. 5 Anodic Polarization Curves for Half and Completely Emissively Coated Haynes 25 in Deaerated Seawater at Room Temperature

Fig. 6 Cathodic Polarization Curves for Half and Completely Emissively Coated Haynes 25 in Deaerated Seawater at Room Temperature

Fig. 7 Anodic Polarization Curves for Half and Completely Emissively Coated Haynes 25 in 90°C Seawater

Fig. 8 Cathodic Polarization Curves for Half and Completely Emissively Coated Haynes 25 in 90°C Seawater

Fig. 9 Anodic Polarization Curves for Uncoated Thermally Shocked Haynes 25 in Aerated and Deaerated Seawater at Room Temperature

Fig. 10 Cathodic Polarization Curves for Uncoated Thermally Shocked Haynes 25 in Aerated and Deaerated Seawater at Room Temperature

Fig. 11 Linear Plot of Cathodic Overvoltage vs. Current Density for Uncoated Haynes 25 in 90°C Seawater

Fig. 12 Linear Plot of Cathodic Overvoltage vs. Current Density for Completely Coated Haynes 25 in 90°C Seawater

Fig. 13 Linear Plot of Cathodic Overvoltage vs. Current Density for Uncoated, Thermally Shocked Haynes 25 in Deaerated Seawater at Room Temperature

TABLE 2

Summary of Electrochemical Data for Haynes 25

Specimen Condition	Seawater Condition	β_c (v)	β_a (v)	$\frac{\Delta E}{\Delta i}$ (v/ μ amp/cm ²)	$i_{corr} \times 10^{+2}$ (μ amp/cm ²)	Corrosion Rate $\times 10^{+2}$ (mpy)
half-coated	room temp. aerated	0.182	0.184	0.84	4.7 (4.5)	2.5 (2.4)
completely coated	room temp. aerated	0.172	0.276	0.69	6.7 (5.6)	3.6 (3.0)
half-coated	room temp. deaerated	0.212	0.310	1.44	3.8 (2.8)	2.0 (1.5)
completely coated	room temp. deaerated	0.195	0.236	0.44	10.6 (8.1)	5.6 (4.3)
uncoated	90°C	0.169	0.340	10	0.49 (0.38)	0.26 (0.20)
half-coated	90°C	0.174	0.302	0.47	10.2 (7.5)	5.4 (4.0)
completely coated	90°C	0.196	0.330	0.29	18.4 (10.0)	9.8 (5.3)
thermally shocked	room temp. aerated	0.150	0.226	11.2	0.35 (0.32)	0.19 (0.17)
thermally shocked	room temp. deaerated	0.365	0.384	13.5	0.60 (0.90)	0.32 (0.48)

to mention here that in an experiment in which plutonium dioxide beads were exposed to seawater at 120° and 190°C, the lower dissolution rates observed (compared to room temperature) was attributed to a coating of calcium sulfate film on the beads at the high temperature.⁵ It is very likely that the same situation occurred in the present case of Haynes 25 in heated seawater. In fact, a coating (most likely calcium sulfate because of its negative solubility temperature coefficient) formed on the inside of the pyrex polarization vessel following each experiment in which the seawater was heated to 90°C.

The emissively coated specimens of Haynes 25, both half-coated and completely coated, show corrosion rates about an order of magnitude higher than that of the uncoated Haynes 25. Since the electrochemical method measures the total corrosion current, it could not determine what fraction of the total current was due to the corrosion of Haynes 25. However, an indication that part of the corrosion current is due to the corrosion of the emissive coating can be obtained from a comparison of the corrosion rates of the half-coated and completely coated specimens. For each experimental condition of aeration, deaeration and temperature, the completely coated specimens showed a slightly higher corrosion rate than the half-coated specimens. This observation would be expected if the coating were corroding since the rates for the half-coated specimens were calculated on the basis of the total surface area (coated plus uncoated) of the specimen. A consideration which must be taken into account for the emissively coated specimens is that the geometric surface area was used in expressing the current per unit area. This geometric surface area is a relatively good measure of the actual surface area for the uncoated specimens of Haynes 25 because of their smooth surface. However, it is not a good measure of the actual surface area in the case of the emissively coated specimens because of the roughness of the coating. Therefore, since the actual surface area of the emissively coated specimens is greater than the geometric surface area, the values of the corrosion rates of the emissively coated specimens shown in Table 2 are conservative (i.e., higher than the true rates).

Comparison of the corrosion rates of all the emissively coated specimens indicates that there is only a slight effect of aeration, deaeration, partial coating or temperature. The fact that the rate at 90°C is not higher than the rate at room temperature indicates, as in the case of uncoated Haynes 25, the formation of a calcium sulfate coating on the specimens run at 90°C.

The corrosion rates of the thermally shocked specimens of Haynes 25 compare well with the corrosion rate of specimens not thermally shocked (3.5×10^{-3} mpy). This good agreement indicates that subsequent heating (bar stock is heat-treated at 1230°C and water quenched by the manufacturer) to 500°C followed by quenching in seawater does not affect the corrosion properties of Haynes 25 to a significant degree.

V. CONCLUSION

Electrochemical corrosion rate measurements made on uncoated, emissively coated and thermally shocked uncoated Haynes 25 in room temperature and in 90°C seawater indicate that: (1) there is no significant increase in the corrosion rate of uncoated or emissively coated Haynes 25 in 90°C seawater; (2) the corrosion rates of the half-coated and completely emissively coated specimens are about an order of magnitude higher than that of uncoated specimens. Although it could not be proved conclusively, there was evidence that this increase reflects the corrosion of the emissive coating itself; (3) there was only a slight effect of aeration, deaeration, temperature or partial coating on the corrosion rates of the emissively coated specimens; (4) thermal shocking of uncoated specimens from 500°C to room temperature by quenching in seawater did not increase their corrosion rates.

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D. A. Kubose and H. I. Cordova 16 May 1967
32 p. tables illus. 5 refs.
UNCLASSIFIED

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and thermally shocked and uncoated specimens
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2. Corrosion
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Security Classification

DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author) U.S. Naval Radiological Defense Laboratory San Francisco, California 94135		2a. REPORT SECURITY CLASSIFICATION UNCLASSIFIED	
		2b. GROUP	
3. REPORT TITLE CORROSION STUDIES OF HAYNES 25 ALLOY IN SEAWATER USING ELECTROCHEMICAL TECHNIQUES			
4. DESCRIPTIVE NOTES (Type of report and inclusive dates)			
5. AUTHOR(S) (First name, middle initial, last name) Donald A. Kubose Herman I. Cordova			
6. REPORT DATE 22 August 1967		7a. TOTAL NO. OF PAGES 32	7b. NO. OF REFS 5
8a. CONTRACT OR GRANT NO. AEC Contract No. AT(49-5)-2084		9a. ORIGINATOR'S REPORT NUMBER(S) USNRDL-TR-67-64	
b. PROJECT NO.			
c.		9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)	
d.			
10. DISTRIBUTION STATEMENT This document has been approved for public release and sale; its distribution is unlimited.			
11. SUPPLEMENTARY NOTES		12. SPONSORING MILITARY ACTIVITY Atomic Energy Commission Washington, D.C. 20545	
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14 KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Corrosion Seawater Haynes 25						