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USNRDL-TR-67-109

19 August 1967

MASTER

SEAWATER CORROSION STUDIES OF EMISSIVELY-COATED HAYNES 25; RADIOACTIVE TRACER TECHNIQUES

by

D. A. Kubose

H. A. Goya

M. G. Lai

H. I. Cordova

*Not AEC
Refused*

U.S. NAVAL RADIOLOGICAL
DEFENSE LABORATORY

SAN FRANCISCO • CALIFORNIA • 94135

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NUCLEAR TECHNOLOGY DIVISION
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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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Eugene P. Cooper

Eugene P. Cooper
Technical Director

D. C. Campbell

D. C. Campbell, CAPT USN
Commanding Officer and Director

ABSTRACT

Corrosion rates of uncoated, half- and completely-emissively-coated Haynes 25 have been measured in seawater by use of radioactive tracer techniques. The corrosion rates observed for the (1) uncoated and (2) half- and completely-coated specimens were nominally 3×10^{-6} and 1×10^{-4} mil per year (mpy), respectively. It was established that the observed corrosion rates of the half- and completely-coated specimens were due entirely to corrosion of Haynes 25 and not to the corrosion of the emissive coating. The higher corrosion rates were attributed primarily to the fact that the actual surface area of the Haynes 25 was much larger than the geometric surface area because of surface preparation prior to application of the emissive coating.

A re-examination of the corrosion rate based upon the amounts of cobalt and chromium released to the seawater indicated that the previously reported differential leaching of these components of Haynes 25 was an experimental artifact due to errors in counting extremely low amounts of chromium-51. Present observations show that the cobalt-and-chromium-based corrosion rates are the same and agree with the previous cobalt-based corrosion rate.

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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 corrosion rate measurement of uncoated, half- and completely-emissively-coated Haynes 25 in seawater by use of radioactive tracer techniques.

It was found that the corrosion rates of the half- and completely-coated specimens were higher than that of the uncoated specimens. This increase was attributed to the fact that the actual surface area of the Haynes 25 for the half- and completely-coated specimens was much greater than the geometric surface area because of surface preparation prior to application of the emissive coating.

A re-examination of the corrosion rate based upon the amounts of cobalt and chromium released to the seawater indicated that there was no significant differential leaching of these components of Haynes 25; this contradicts earlier results.

I. INTRODUCTION

For the past several years the U. S. Naval Radiological Defense Laboratory 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.

In addition to examination of the solubility behavior of the radionuclide fuels themselves in seawater,¹⁻⁶ experiments have been carried out for determination of the corrosion rates of various fuel container materials in seawater.⁷⁻⁹ A recent report described the corrosion rate determination of emissively coated Haynes 25 in seawater by use of electrochemical techniques.^{9*} One of the results of that work indicated that the emissively coated specimens of Haynes 25 had a higher corrosion rate than that of the uncoated specimens. The electrochemical method, however, could not conclusively show whether this increase in corrosion rate was due to the corrosion of the coating itself.

The present report describes the measurement of the corrosion rates of emissively coated Haynes 25 in seawater by use of radioactive tracer techniques. Radioactive (tagged) specimens of Haynes 25 are immersed in seawater and the seawater is analyzed for radioactive corrosion products. This method of corrosion rate measurement provides the means to determine specific corrosion products and therefore is able to show if the increase in corrosion rate of the emissively coated over the uncoated Haynes 25 observed with the electrochemical method is due to the corrosion of the coating itself.

In addition to studies on emissively coated specimens, tracer-technique corrosion rate determinations on uncoated specimens of Haynes 25 were repeated. The latter were to determine whether the differential leaching of the cobalt and chromium components of Haynes 25 observed in an earlier work, also with radioactive tracer techniques, was a real effect or was due to inaccuracies in counting extremely low activities of chromium-51.⁸

*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

A. TEST APPARATUS

The radioactive specimens of Haynes 25 were exposed to seawater in test cells designed to provide control of the seawater temperature, oxygen content and velocity of seawater past the specimens. A detailed description of these test cells can be found elsewhere.⁸

B. SPECIMENS

The Haynes 25 specimens used in the radioactive tracer studies were machine cut into 1.4-inch-square specimens from 60 mil sheet stock obtained from the Stellite Division of Union Carbide Company, Kokomo, Indiana. Table 1 shows the manufacturer's analysis of the alloy. After

TABLE 1

Manufacturer's Analysis of Haynes 25

Cr - 20.45 %	Ni - 9.93 %
W - 15.10	Al - 1.03
Fe - 1.95	P - 0.016
C - 0.07	S - 0.011
Si - 0.07	Co - balance (51.37)

being cut, the specimens were polished successively with 80, 180, 400 and 600 grit paper and then cleaned with benzene and ethanol in an ultrasonic cleaner. Three of the specimens prepared in the above manner were used in the "uncoated" test. Six of the specimens prepared in the above manner were sent to the Missile and Space Division of General Electric Company, Valley Forge, Pennsylvania, for application of the emissive coating. Prior to applying the coating, GE grit-blasted the surface with #20 grit. In addition to completely coated specimens, specimens coated over only half their surface were prepared for determining whether 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 half the surface of each side, including the edges (i.e., as if the specimen had been dipped halfway into a solution).

C. NEUTRON IRRADIATION OF SPECIMENS

Figure 1 shows the aluminum irradiation capsule used. A total of nine specimens were irradiated - three completely coated, three half-coated and three uncoated. The specimens, along with standards, were stacked together, wrapped in aluminum foil, and then placed in the irradiation capsule. The specimens and standards were placed such that their faces were parallel to the long axis of the capsule. Additional aluminum foil was used to fill in the void areas within the capsule. A quartz tube containing some of the emissive coating powder was also placed in the capsule.

The standards of cobalt metal (sheet) and chromium metal (powder) were both 99.99 % pure. The cobalt standards were the same size as the Haynes 25 specimens except that they were only 5 mils in thickness. The chromium powder was placed in a specially fabricated aluminum holder. It was the same size as the Haynes 25 specimens but had a 0.5 inch diameter by $3/4$ inch deep counterbored cavity in one side. A $1/4$ inch thick aluminum cover was screwed on over the cavity after the chromium powder had been inserted.

The irradiation was carried out in the General Electric Test Reactor at Vallecitos, California. The neutron flux profile to which the stack of specimens and standards were exposed was determined by subsequent counting of the cobalt standards which were placed at various positions within the stack.

The nominal initial activities of cobalt-60 and chromium-51 in the Haynes 25 specimens, obtained by measuring the specific activities of the cobalt and chromium standards, were 110-260 mCi and 65-155 mCi, respectively.

D. SEAWATER

Seawater for the test solutions was collected on a biweekly schedule. Collection was made at least one mile from the nearest land mass (Farallon Islands off San Francisco) by dip-filling of the seawater into 5-gal polyethylene carboys. Prior to use the seawater was filtered through an 0.8 μ Millipore filter for removal of particulate matter.

E. EXPOSURE PROCEDURE

After removal of the specimens from the irradiation capsule, they were rinsed several times in seawater before being placed in the test cells.

Fig. 1 Neutron Irradiation Capsule.

The parameters of seawater velocity (0.7 ft/sec obtained by stirring the seawater), aeration (air purge) and deaeration (nitrogen purge) were chosen such that their individual effects on the corrosion rates of the different specimens (i.e., completely coated, half-coated and uncoated) could be determined. The seawater temperature in all the test cells was maintained at $6 \pm 2^{\circ}\text{C}$. This temperature approximates that observed in the basins off the southern California coast.

At successive intervals of 6, 13, 14, 15, 20, 51, and 41 days (total elapsed exposure times of 6, 19, 33, 48, 68, 119, and 160 days), the seawater in each test cell was drained and fresh seawater put in. Aliquots of the seawater from each exposure interval were analyzed for radioactive corrosion products.

F. CALIBRATIONS

Specific Activity

To obtain a corrosion rate from the amount of activity leached into the test seawater from the Haynes 25 specimens, one must know the specific activity of each isotope of interest (e.g., cobalt-60, chromium-51) expressed as cpm per μg of alloy. These specific activities were determined by solution of accurately weighed portions of the irradiated cobalt and chromium standards in the minimum amount of concentrated hydrochloric acid, dilution of each of the resulting solutions to an appropriate volume in a volumetric flask and then counting of aliquots of these solutions. The specific activity for Haynes 25 in terms of the cobalt and chromium activities was then calculated by use of the known percent cobalt and chromium content of Haynes 25.

Flux Profile

To make valid comparisons of the corrosion rates of the various specimens, one must make corrections for any differences in the neutron flux between the standards and each specimen due to neutron flux attenuation in the stack.

These corrections were obtained from the cobalt standards placed at various positions within the specimen stack. The relative positions of the cobalt standards with respect to the Haynes 25 specimens are shown in Fig. 2. The relative neutron flux profile, also shown in Fig. 2, was obtained by determining the specific activity of cobalt for each cobalt standard. The relative flux profile across the face of the specimen stack was obtained by determination of the specific activity of pieces cut off opposite corners of the cobalt standards. The results of the latter determination showed that the neutron flux was essentially the same over all areas of the face of the specimen stack.

Fig. 2 Neutron Flux Profile for the Various Positions of Standards and Specimens.

Therefore, the only correction required was that for the flux profile shown in Fig. 2.

G. ANALYTICAL PROCEDURES

Radioactive cobalt and chromium in the seawater in which the Haynes 25 specimens had been immersed were determined by the following procedures.

Cobalt-60

Analysis for cobalt-60 was made by direct counting of a 400 ml aliquot of the test seawater solution in a gamma pulse-height analyzer system. The amount of cobalt-60 present in the aliquot was determined by comparison with a series of standard samples containing known amounts of cobalt-60.

Chromium-51

Analysis for chromium-51 was made by the following solvent-extraction method.¹⁰ Chromium carrier was added to the sample and was precipitated with ammonium hydroxide. The precipitate was dissolved and the chromium was oxidized to the +6 state by vigorous heating in perchloric acid. The hexavalent chromium was then complexed with n-tetrabutylammonium hydroxide followed by extraction with hexone. The chromium was stripped from the organic phase with a sodium hydroxide-ammonium chloride mixture. Impurities were removed by several ferric hydroxide scavenging steps. The purified chromium was measured spectrophotometrically for chemical yield and the chromium-51 was counted in a gamma pulse-height analyzer system.

Analysis of seawater blanks was made in all cases.

III. RESULTS AND DISCUSSION

A previous report, based on the same radioactive tracer techniques described here, indicated that there was differential leaching of the cobalt and chromium components of Haynes 25 in seawater.⁸ The corrosion rate of Haynes 25 based on the amount of chromium released to the seawater was about two orders of magnitude higher than that based on the amount of cobalt released. It is felt that the high chromium result might have been due to inaccuracies in counting extremely low levels of the chromium-51 in the seawater. However, at that time it was the only information available and it was reported as such.

TABLE 2

Comparison of Corrosion Rates of Haynes 25 Based on
Cobalt and Chromium Analyses

	mpy (based on Co)	mpy (based on Cr)	Exposure Time Days*
Haynes 25 #1	3.4×10^{-5}	1.8×10^{-5}	7
Haynes 25 #2	1.4×10^{-5}	9.7×10^{-6}	7
Haynes 25 #1	1.1×10^{-5}	9.2×10^{-6}	22
Haynes 25 #2	9.2×10^{-6}	5.9×10^{-6}	22

*Seawater sampled and changed at end of 7 days followed by additional exposure period of 15 days.

For re-examination of the cobalt-and-chromium-based corrosion rates of Haynes 25, two of the uncoated specimens used in this work were exposed to seawater prior to the initiation of the test schedule outlined in section II E. Table 2 shows the corrosion rates of these two specimens of Haynes 25 based on the amounts of cobalt and chromium released to the seawater.

The data show good agreement between the cobalt and chromium-based corrosion rates (i.e., no significant differential leaching). These corrosion rates are in agreement with the cobalt-based corrosion rates in Ref. 8. The chromium data obtained here are felt to be more valid than that obtained in the previous work because of the much higher specific activity of chromium-51 in the present seawater samples at the time of analysis (e.g., the present chromium activity had decayed only 1.5 half-lives compared to 5 half-lives previously; moreover, the initial chromium activity in the present specimens was an order of magnitude higher than that in the previous specimens). This higher level of activity would greatly lessen the percent contribution of impurities to the count rate (previous samples were counted in a well counter, not in a pulse-height analyzer) and would therefore provide a more valid measure of the true amount of chromium present in the seawater sample.

Figures 3 through 5 present the values of the corrosion rates (averaged over the total exposure periods) of uncoated, half-coated and completely-coated Haynes 25, respectively, as a function of exposure time for the various conditions of aeration, deaeration and seawater velocity. All values of the corrosion rates are based on the amount of cobalt released to the seawater. The vertical bars for each

Fig. 3 Corrosion Rate of Uncoated Specimens of Haynes 25 in Aerated, Deaerated and Stirred, Aerated Seawater.

Fig. 4 Corrosion Rate of Half-Emissively Coated Haynes 25 in Aerated, Deaerated and Stirred, Aerated Seawater.

Fig. 5 Corrosion Rate of Completely-Emulsively Coated Haynes 25 in Aerated, Deaerated and Stirred, Aerated Seawater.

exposure interval serve to indicate the general decrease of the corrosion rates as a function of exposure time and the attainment of relatively constant values at long exposure times. It should be noted that the individual points do not represent instantaneous corrosion rates but represent the average value over a particular exposure interval.

In all instances (i.e., coated, uncoated, half-coated) there appears to be no significant (in the operational sense) effect, particularly at the longer exposure times, of the exposure parameters of aeration and seawater velocity on the observed corrosion rates. There appears to be a slight lowering effect of deaerated seawater in all instances.

Table 3 summarizes the corrosion rates observed for the longer exposure times. Comparison of the corrosion rates of the uncoated, half- and completely-coated specimens shows that the corrosion rates of the completely-coated specimens are significantly higher than those of the uncoated specimens. The corrosion rates of the half-coated specimens were also higher than those of the uncoated specimens but were slightly lower than those of the completely-coated specimens. This same relationship had been obtained when electrochemical techniques were used to measure the corrosion rates of uncoated, half- and completely-coated specimens of Haynes 25.⁹ The electrochemical technique, however, could not conclusively determine whether the increase in corrosion rate of the completely coated specimens over that of the uncoated specimens was due to the corrosion of the coating itself or was due to an inherent increase in the corrosion rate of the Haynes 25 due to the presence of the coating.

One of the unique advantages of the radioactive tracer technique is that it can provide information of that nature. The following considerations indicate that the increase in corrosion rate of the completely-coated specimens over that of the uncoated specimens is due to the Haynes 25 itself and not to the corrosion of the coating. Since Haynes 25 contains approximately 50 % cobalt, the characteristic peak of cobalt-60 (1.17 Mev) in the gamma pulse height spectrum of the seawater sample was used as the measure of its corrosion. The gamma pulse-height spectrum of the irradiated emissive-coating powder showed a characteristic peak at approximately 0.8 Mev (most likely manganese-54) in addition to the expected iron-59 peaks (at 1.10 Mev). The fact that the characteristic peak at 0.8 Mev was not detected in the gamma pulse-height spectrum of the seawater indicates that there was no detectable amount of the coating material in the seawater. In other words, all of the counts integrated under the cobalt-60 peak are coming from Haynes 25 with an insignificant contribution from the iron-59 peak (iron-59 and cobalt-60 have almost identical gamma spectra). There is the possibility of differential leaching of the manganese and iron. However, if there were, it would have to be on the order of 10^3 in favor of iron to account for

TABLE 3

Corrosion Rate Data for Uncoated, Half- and Completely-Emissively Coated
Haynes 25 After 160 Days Immersion in Seawater

Specimen	Exposure Parameter	Corrosion Rate (mpy)
uncoated	aerated	3.4×10^{-6}
uncoated	deaerated	2.8×10^{-6}
uncoated	aerated, stirred	4.6×10^{-6}
half-coated	aerated	9.1×10^{-5}
half-coated	deaerated	7.6×10^{-5}
half-coated	aerated, stirred	9.1×10^{-5}
completely coated	aerated	3.0×10^{-4}
completely coated	deaerated	0.51×10^{-4}
completely coated	aerated, stirred	1.4×10^{-4}

even 10 % of the total counts under the cobalt-60 peak. This magnitude of differential leaching is very unlikely.

These results, however, do not imply that the coating is not corroding. Its detection sensitivity was simply too low to provide data (the specific activity of the cobalt-60 from Haynes 25 was three orders of magnitude greater than that of the 0.8 Mev peak from the coating material).

There are two possible causes for the higher corrosion rate of the completely-coated specimens. One, the emissive coating-Haynes 25 system functions as a galvanic couple with the Haynes 25 being the anode; and two, the higher corrosion rate is a surface area effect. The surface area effect is felt to be the more important of the two since the emissive coating has a relatively low electrical conductivity. Prior to the application of the emissive coating the surface of the specimen is grit blasted with #20 grit (for the half-coated specimens only the half to be coated was grit-blasted) and hence its surface area is much greater than the surface area of the uncoated specimens, whose surface has been polished with #600 grit paper. Since the corrosion rates reported here are based on the geometric surface area of the specimens, the reported corrosion rates of the half- and completely-coated specimens are much higher than if their actual surface areas were used in the calculation of their corrosion rates. This explanation is based on the assumption that the emissive coating does not affect the transport of cobalt and chromium ions from the Haynes 25 to the seawater.

IV. CONCLUSION

The results obtained indicate that a previously observed corrosion rate of Haynes 25 based on the amount of the chromium component released to the seawater was an experimental artifact due to inaccuracies arising from the counting of extremely low activities of chromium-51. The present results, considered more valid because of the much higher chromium-51 activities counted, show that there is no significant differential leaching of the cobalt and chromium components of Haynes 25; this contradicts the earlier results.

Comparison of the corrosion rates observed for uncoated, half- and completely-emissively-coated Haynes 25 indicated that the half- and completely-coated specimens had a significantly higher corrosion rate than that of the uncoated specimens. It was established that the increased corrosion rate was due only to the Haynes 25 and not to the corrosion of the emissive coating itself. The higher corrosion rates

of the half- and completely-coated specimens were attributed primarily to the fact that the actual surface area of the Haynes 25 for the coated specimens was much larger than its geometric surface area because of surface preparation prior to application of the emissive coating.

In all cases there was no significant (in the operational sense) effect of aeration or seawater velocity on the observed corrosion rates. The rates in deaerated seawater were slightly lower.

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DISTRIBUTION DATE: 31 October 1967

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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 SEAWATER CORROSION STUDIES OF EMISSIVELY-COATED HAYNES 25; RADIOACTIVE TRACER TECHNIQUES			
4. DESCRIPTIVE NOTES (Type of report and inclusive dates)			
5. AUTHOR(S) (First name, middle initial, last name) Donald A. Kubose Ming G. Lai Harry A. Goya Herman I. Cordova			
6. REPORT DATE 31 October 1967		7a. TOTAL NO. OF PAGES 29	7b. NO. OF REFS 10
8a. CONTRACT OR GRANT NO. AEC, Contract No. AT(49-5)-2084		9a. ORIGINATOR'S REPORT NUMBER(S) USNRDL-TR-67-109	
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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DD FORM 1473

1 NOV 65

(PAGE 1)

S/N 0101-807-6801

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KEY WORDS

Corrosion
Haynes-25

LINK A

LINK B

LINK C

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