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MEASUREMENT OF SEAWATER CORROSION OF SNAP
CONTAINER ALLOYS USING RADIOACTIVE TRACER
TECHNIQUES

by

D. A. Kubose

M. G. Lai

H. A. Goya

H. I. Cordova

RELEASED FOR ANNOUNCEMENT
IN NUCLEAR SCIENCE ABSTRACTS

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

SAN FRANCISCO • CALIFORNIA • 94135

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NUCLEAR APPLICATIONS BRANCH

D. L. Love, Head

NUCLEAR TECHNOLOGY DIVISION

R. Cole, Head

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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 Haynes 25, Hastelloy C and Hastelloy N in seawater have been measured using two radioactive tracer techniques. These techniques involved (1) measurement of the radioactivity leached into seawater from radioactive alloy specimens and (2) performing neutron activation analysis of corrosion products in seawater in which inactive alloy specimens had been placed. The second technique was used to determine whether the gamma recoil from the thermal neutron activation of the alloys in the first technique had affected their corrosion properties.

It was found that the gamma recoil from neutron activation had only a small effect on the corrosion rate of the alloys.

The average corrosion rates obtained for Hastelloy C and Hastelloy N were 0.5×10^{-4} mil per year and 1×10^{-4} mil per year, respectively. No appreciable differential leaching of the cobalt, nickel and chromium components of these alloys was observed. The results obtained for Haynes 25 indicated differential leaching had occurred. The corrosion rate calculated from the amount of cobalt released was 1×10^{-6} mil per year while that calculated from the amount of chromium released was 4×10^{-4} mil per year.

Results of experiments in which specimens of Haynes 25 in seawater were exposed to bremsstrahlung and cobalt-60 radiation indicated no significant effect of ionizing radiation on the corrosion rate for exposures up to 10^8 rads.

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SUMMARY

A program to study the release of SNAP (Systems for Nuclear Auxiliary Power) radionuclide fuels to an ocean environment requires, in addition to measurements of fuel solubilities in seawater, a knowledge of the corrosion rates in seawater of the material containing the radionuclide fuel. Initial indications of the corrosion rates of certain container materials, Haynes 25, Hastelloy C, and Hastelloy N, in seawater were obtained from radioactive tracer techniques.

The average corrosion rates observed for Hastelloy C and Hastelloy N were 0.5×10^{-4} mil per year and 1×10^{-4} mil per year, respectively. Differential leaching of alloy components, which was not observed for Hastelloy C and Hastelloy N, was observed for Haynes 25. The corrosion rate of Haynes 25 calculated from the amount of the cobalt component released was 1×10^{-6} mil per year while that calculated from the amount of the chromium component released was 4×10^{-4} mil per year.

I. INTRODUCTION

For several years personnel of the U. S. Naval Radiological Defense Laboratory have been conducting experiments on the release of SNAP (Systems for Nuclear Auxiliary Power) radionuclide fuels to the seawater environment.

Previous reports have described the results of studies involving measurement of the solubility rates of various isotopic fuel forms in seawater,^{1-4,*} the release of fission products from irradiated nuclear power reactor fuels to seawater,⁵⁻⁹ and the electrochemical measurement of corrosion rates of the isotopic fuel container material in seawater.¹⁰

In the last study, the container materials 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 electrochemical technique was the first of three such techniques instituted. The present report describes the study of alloy corrosion using the two other techniques; (1) measurement of the concentration of radioactivity leached into the seawater from thermal-neutron-activated alloy specimens, and (2) activation analysis of corrosion products in seawater from inactive alloy specimens which had been immersed in the seawater. The last technique was used to determine whether the gamma recoil from the thermal neutron activation of the alloys in the preceding technique had affected their corrosion properties.

*Determination of the chemical states of Pu²³⁸ in seawater can be found in a report by W. E. Lingren, "The Electrochromatography of Seawater Containing Dissolved Plutonium," U. S. Naval Radiological Defense Laboratory, USNRDL-TR (in preparation).

II. EXPERIMENTAL

A. SEAWATER EXPOSURE APPARATUS

The alloy specimens were exposed to seawater in an apparatus that was designed, in accordance with ASTM Standard Designation A279-44T,¹¹ to provide control of the seawater temperature and oxygen content and the velocity of seawater past the specimen. All components of the apparatus in contact with the seawater were non-metallic.

Twenty test units, each designed to expose one specimen at a time were fabricated. Figure 1 shows the general arrangement of these twenty test units.

Test Units

A test unit and its components are detailed in Fig. 2. Each unit consists of an outer lead-shield container (to minimize radiation hazard to personnel from the radioactive specimens) and an inner pyrex test cell. A wax fill between supports the test cell within the lead shield and serves as thermal insulation. The test cell cover is of pyrex and is removable. Four 24/40 taper seal openings in it provide access for a specimen holder, thermometer well, gas aerator, and gas exhaust. The bottom of the test cell has a pyrex tubulation leading to a teflon-plug stopcock to permit draining of the seawater from the cell.

Rotating Specimen Holder

The specimen holder was fabricated from high-density polyethylene in the form of a miniature vise (see Fig. 2). This vise assembly is mounted on a pyrex rod which passes through a teflon stirrer bearing; this in turn fits into the center opening of the test cell cover. The configuration of the holder is such that the specimen, which was mounted in a vertical position, sweeps out a circle with a radius of 1-1/2 inches when the holder is rotated. This radius, coupled with a rotation speed of 55 RPM, provides a water velocity of approximately 0.7 ft/sec past

Fig. 1 General Arrangement of Test Units (Drawing No. CT66-652, Sheet 1)

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Fig. 2 Construction Details of a Test Unit (Drawing No. CT66-652, Sheet 3)

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the specimen. This velocity of the water past the specimen, which corresponds to about 0.4 knot, approximates the current velocity at depth off the California coast.^{12,13} The velocity of the mass of the water rotating in the test cell was not significant.

The holder is rotated by a synchronous electric motor-gear box assembly mounted atop the lead shielded container. A flexible coupling between the gear box output shaft and the pyrex rod allows for slight misalignment when assembling a test unit.

Temperature and Aeration Systems

The seawater in the test cell is maintained at the desired temperature by circulating chilled water through "Calorex" tubing wrapped around the pyrex cell. Because the tubing is flat on one side and is elastic and a good thermal conductor, it provides good cooling efficiency. The circulating water was maintained at $6 \pm 2^\circ\text{C}$ by a refrigeration unit. This temperature approximates that observed in the basins off the Southern California coast.¹⁴ The temperature of the seawater in each test cell was monitored by a Weston dial-type thermometer. The thermometer fits in a pyrex thermometer well in one of the cell cover openings.

The test gas, air or nitrogen, is introduced into the seawater by means of a fritted pyrex bubbler in one of the openings in the cell cover. The flow rate was approximately 50 cc/min. The concentration of oxygen in the air saturated seawater, $6.5 \pm 1 \text{ ml O}_2/\ell$, represents the highest concentration of oxygen normally expected in seawater.¹⁴

Figure 3 shows the general arrangement of the plumbing systems for cooling water and test gas for each of the twenty test units.

B. SPECIMENS

Specimens of Haynes 25, Hastelloy C and Hastelloy N, $1.4 \text{ in.}^2 \times 1/16 \text{ in.}$ thick, were machine cut from sheet stock obtained from Stellite Division, Union Carbide Corp. Samples of each alloy were analyzed.* Table 1 shows their composition.

Each specimen was treated in the following manner. The surfaces were polished with 80, 180, 400, and 600 grit paper, in that order, using a Precision Scientific Metal Polisher. After polishing, the

*Metallurgical Laboratories, Inc., 1142 Street, San Francisco, Calif.

TABLE 1
Composition of Alloys

Haynes 25

Fe - 1.48 %	Cu - 0.08 %
Ni - 9.86	Cr - 20.24
Al - 0.03	Pb - 0.005
Sn - 0.003	Zn - 0.01
Co - 52.10	Mn - 0.90
Mo - 0.04	Si - 0.02
Ag - 0.0001	V - 0.05
Mg - 0.003	Ca - 0.001
Ti - 0.002	W - 14.88

Hastelloy N

Fe - 4.42 %	Cu - 0.06 %
Ni - 69.54	Cr - 7.23
Al - 0.004	Pb - 0.005
Sn - 0.004	Zn - 0.01
Co - 0.40	Mn - 0.75
Mo - 16.71	Si - 0.25
Ag - 0.0001	V - 0.22
Mg - 0.02	Ca - 0.001
Ti - 0.003	

Hastelloy C

Fe - 4.89 %	Cu - 0.05%
Ni - 56.42	Cr - 15.41
Al - 0.02	Pb - 0.005
Sn - 0.003	Zn - 0.01
Co - 1.08	Mn - 0.50
Mo - 16.02	Si - 0.25
Ag - 0.0001	V - 0.28
Mg - 0.006	Ca - 0.002
Ti - 0.002	W - 4.81

Fig. 3 Water-Cooling and Gas-Aeration Systems of Test Units (Drawing No. CT66-652, Sheet 2)

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specimens were cleaned in ethanol using an ultrasonic cleaner. Surface profile measurements were made using a Proficorder (Micrometrical Division, Bendix Corp., Ann Arbor, Michigan). Figure 4 shows typical profiles obtained before and after exposure to seawater. Finally, each specimen was weighed to within 1 mg on a Mettler Type S Balance.

Neutron Irradiation of Specimens

Figure 5 shows one of the aluminum irradiation capsules used. Polyethylene spacers were used to hold the specimens and standards in place within the capsule. Seven specimens of each alloy were irradiated, each set of seven in one of three capsules used.

The standards of cobalt metal (sheet), nickel metal (sheet) and chromium metal (powder) were irradiated simultaneously with the alloy specimens. The purity of the cobalt was 99.99 %, the nickel 99.97 % and the chromium 99.99 %. The cobalt and nickel standards were the same size as the alloy specimens except that the cobalt was 0.005 in. thick and the nickel was 0.010 in. thick. The chromium powder was placed in specially fabricated aluminum holders. These holders were the same size as the alloy specimens but had a 0.5 inch diameter by $3/64$ in. deep counterbored cavity in one side. A $1/64$ in. thick aluminum cover was screwed over the cavity after the powder was put in.

The irradiation was carried out in the Mark III Reactor Pool Facility at General Atomics, San Diego, California. The neutron flux profile, both radial and vertical, to which the specimens were exposed was determined by placing 0.05 % cobalt-aluminum flux wires in appropriate positions within the irradiation capsules.

The initial activities of cobalt-60, chromium-51, and nickel-63 in each of the three alloys were the following: Haynes 25, 30 mc of cobalt-60 and 18 mc of chromium-51; Hastelloy C, 3 mc of cobalt-60, 73 mc of chromium-51 and 0.1 mc of nickel-63; Hastelloy N, 2.5 mc of cobalt-60, 70 mc of chromium-51 and 0.3 mc of nickel-63.

C. 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 the seawater into 5-gallon polyethylene carboys. Prior to use, the seawater was filtered through an 0.8 μ membrane filter (Millipore) to remove particulate matter.

HASTELLOY - C

HASTELLOY - N

HAYNES - 25

Fig. 4 Representative Surface Profiles Before and After Exposure to Seawater

D. SEAWATER EXPOSURE PROCEDURE

Inactive Specimens

Two specimens of Haynes 25 and one each of Hastelloy C and Hastelloy N were immersed in seawater contained in the test units. Utmost care was exercised in cleaning the components of the test units and in handling the specimens to avoid any contamination. The test conditions imposed on each specimen were: seawater temperature of 6°C, seawater saturated with air, and no movement of the seawater past the specimen. After successive intervals of 37 and 57 days (total exposure time was 94 days) the seawater from each test unit was drained and fresh seawater put in. Neutron activation analysis was performed on aliquots of the seawater from each exposure interval to determine the amounts of cobalt, chromium and nickel leached from the alloy specimens.

One specimen of Haynes 25 immersed in seawater in a pyrex container was exposed to bremsstrahlung radiation (2 Mev electrons at 150 μ A, impinging on a gold target) using the USNRDL Van de Graaff Facility. The dose rate to the seawater was approximately 10^3 rads per hour as determined by the Fricke dosimeter.* During the test period of twenty days the test specimen was irradiated on the average of about five hours each day. Another specimen of Haynes 25 immersed in seawater was used as a control specimen.

The USNRDL 1500 curie Cobalt-60 Source Facility was used in an analogous experiment. The Haynes 25 test specimen was under continuous gamma irradiation for a period of 35 days at a dose rate of 2×10^5 rads per hour to the seawater as determined by Fricke dosimetry.

After termination of both experiments, aliquots of the seawater were subjected to neutron activation analysis for the determination of cobalt and chromium.

Active Specimens

Experiments with the active specimens were made after completion of the experiments with the inactive specimens to preclude contamination of the apparatus.

*See, for example, J. W. T. Spinks and R. J. Woods. An Introduction to Radiation Chemistry. New York, John Wiley and Sons, Inc., p. 106, 1960.

Specimens of Haynes-25, Hastelloy C and Hastelloy N and the metal standards were irradiated with thermal neutrons prior to exposure to seawater. After return of the irradiation capsules to USNRDL, the specimens, standards and neutron flux monitoring wires were removed. Their relative positions within each capsule were noted to permit corrections for any flux variations. Each specimen was cleaned by dipping in dilute hydrochloric acid for a few seconds and then rinsing in distilled water. The radioactivity of the specimens was measured in a high pressure gamma ionization chamber and a gamma pulse height spectrum was obtained for one specimen of each alloy. After the radioactivity measurements, the samples stood overnight in separate beakers containing distilled water. They were then mounted on the specimen holders and immersed in the test units.

The parameters of stirring, aeration (air purge) and deaeration (nitrogen purge) were chosen such that their individual effects on the corrosion rate of each alloy could be determined.

At successive intervals of 18, 34 and 46 days (total elapsed time of 18, 52 and 98 days) the seawater in each test unit was drained and fresh seawater put in. Aliquots of the seawater from each exposure interval were analyzed to determine the amounts of cobalt-60, chromium-51 and nickel-63.

The Cobalt-60 Source Facility was again used, this time with an active specimen of Haynes 25, to determine whether cobalt-60 gamma radiation would affect its corrosion rate. At successive intervals of 20, 22 and 42 days (total elapsed time of 20, 42 and 84 days) the seawater in the control and test units was drained and replaced with fresh seawater.

E. CALIBRATIONS - ACTIVE SPECIMENS

Specific Activity

To obtain a corrosion rate from the amount of activity leached into the seawater, the specific activity of each isotope of interest expressed as cpm per μg of alloy must be known. These specific activities were determined by dissolving an accurately weighed piece of each irradiated standard sample in the minimum amount of an appropriate acid (cobalt

and chromium in concentrated hydrochloric acid, and nickel in concentrated nitric acid), diluting each of the resulting solutions to 100 ml in a volumetric flask and then counting aliquots of these 100 ml solutions in a gamma pulse height analyzer or a liquid scintillation counter.

Flux Profile Determination

To make valid comparisons of the corrosion rates of the specimens irradiated within a capsule, correction must be made for any differences in flux density between the standard and each specimen due to the capsule orientation to the flux. For this purpose flux wires were placed in each of the three capsules.

The relative positions of seven flux wires placed in each capsule are shown in Fig. 5. Following irradiation the flux wires were removed and cut; each of the six radial flux wires into three equal segments and the one vertical flux wire into ten equal segments. Each segment was counted and the relative average flux profile obtained. Additional relative flux profile information was obtained from the gross activities of each specimen. Corrections were made by multiplying the specific activity of the standard by a factor which was determined for each position by the flux profile; i.e., the factor, in effect, normalized the specific activity at each position to the position of the standard. Most of the corrections were of the order of 5 - 10 %; the largest correction which had to be made was about 30 %.

F. ANALYTICAL PROCEDURES

Inactive Specimens

Aliquots of the seawater, in which inactive specimens of Haynes 25, and Hastelloy C and N, were exposed, were processed to isolate cobalt, chromium and nickel. After separation the material was sealed in a quartz ampoule and sent to Oak Ridge National Laboratory for neutron activation. The following are the analytical procedures used to separate the elements from seawater before neutron activation.

Seawater blank analyses were performed for each isotope using the same procedures.

Fig. 5 Irradiation Capsule Showing Relative Positions of Alloy Specimens, Standards, and Flux Wires

Cobalt

Cobalt was extracted from the seawater with α -nitroso- β -naphthol. The precipitate was collected on a Millipore filter, the filter and precipitate were ashed in perchloric acid and the residue was transferred to a quartz ampoule and evaporated to dryness. After neutron activation the analysis for cobalt-60 was made from a gamma pulse-height distribution.

Nickel

Nickel was complexed by addition of dimethylglyoxime to the seawater. The complex was extracted with chloroform then back-extracted into the aqueous phase with hydrochloric acid. The resulting solution was evaporated to dryness after transfer to a quartz ampoule. After neutron activation the quartz ampoule was broken and the radioactivity was leached out with hydrochloric acid. The nickel was then determined by counting the cobalt-58 peak in a gamma pulse-height distribution.

Chromium

Chromium was reduced to the +3 state with sulfur dioxide gas and co-precipitated with aluminum hydroxide. The precipitate was heated to dryness and the resulting powder transferred to a quartz ampoule. After neutron activation the ampoule was broken and the powder dissolved in hydrochloric acid. The following analysis for Chromium-51 was determined by the following solvent extraction method.¹⁵ Chromium carrier was added to the sample and 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 sodium hydroxide-ammonium chloride. 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.

Active Specimens

Radioactive cobalt, chromium and nickel in the seawater in which the active alloy 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 seawater 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.

Nickel-63

Nickel-63 was extracted from the seawater by the following method.¹⁶ The nickel was complexed by addition of dimethylglyoxime to the seawater. The complexed nickel was extracted with chloroform then back-extracted into the aqueous phase with dilute sulfuric acid. The resulting solution was evaporated to an appropriate volume, mixed with a scintillant solution and counted in a liquid scintillation spectrometer.

Chromium-51

The analytical procedure for chromium-51 was essentially the same as that used to determine chromium-51 in the neutron-activated samples described above. The only difference was that a 3-inch well gamma counter was used instead of the pulse height analyzer system. This was necessary because of the lower level of chromium-51 activity (after decay) for the active specimens.

G. COUNTING EQUIPMENT

Well Counter

The well counter consisted of a 3 x 3 inch NaI(Tl) crystal mounted on an EMI 95785 photomultiplier tube which in turn was connected to an USNRDL-designed preamplifier. The crystal, photomultiplier tube and preamplifier were enclosed in a lead cave. Other units of the Counter included a John Fluke Model 405 DC power supply and a Systron-Donner Model 1099 scaler.

Pulse Height Analyzer System

The components enclosed in the lead cave were the same as those in the well counter except that a modified Technical Measurement Corp. (TMC) preamplifier was used. The output from the preamplifier was fed to a TMC Model 404-6 400 channel pulse height analyzer which in turn was connected to a TMC Model 522 Resolver-Integrator. Data output was recorded on a TMC-Hewlett-Packard Model 500 tape printer.

Liquid Scintillation Spectrometer

The instrument used was a Tri-Carb 314A (Packard Instrument Sales Corp.) coincidence-type liquid scintillation spectrometer. It was operated at a voltage of 1080 V with a window setting of 500-510. The freezer section was maintained at 0°C.

III. RESULTS AND DISCUSSION

Several corrosion rates under given seawater exposure conditions were calculated for Haynes 25, Hastelloy C and Hastelloy N on the basis of the cobalt, nickel and chromium analyses. For determining whether differential leaching of the elements of an alloy had occurred, the corrosion rate calculated on the basis of one element was compared to that calculated on the basis of another element of the same alloy.

INACTIVE SPECIMENS

Table 2 shows the corrosion rates for duplicate specimens of Haynes 25 and single specimens of Hastelloy C and Hastelloy N. The exposure conditions were: seawater at 6°C, saturated with air and no movement of seawater past the specimen.

The corrosion rates for Haynes 25 based on the cobalt analysis are somewhat erratic, suggesting possible contamination of the seawater prior to activation analysis. The corrosion rates for Haynes 25 based on the chromium analysis are more consistent. The average corrosion rate of the two Haynes 25 specimens was 0.6×10^{-4} mil per year (mpy) for 95 days of immersion. Due to the poor cobalt results it was not possible to establish if differential leaching of the elements of Haynes 25 had occurred.

Comparison of the corrosion rates of Hastelloy C and Hastelloy N based on their respective nickel and chromium analysis indicates that there is very little, if any, differential leaching. Both the nickel and the chromium results indicate that the corrosion rates of the two alloys decrease with increasing time of exposure. The average corrosion rates during 38 days exposure were 1.3×10^{-4} and 3.5×10^{-4} for Hastelloy C and Hastelloy N, respectively. For an additional period of 57 days (95 days total) the average corrosion rates decreased to 0.75×10^{-4} mpy and 1.9×10^{-4} mpy.

TABLE 2

Corrosion Rates (mils per year (mpy)) of Inactive Alloy Specimens in Seawater

	mpy $\times 10^4$ (based on Co analysis)*	mpy $\times 10^4$ (based on Cr analysis)*	mpy $\times 10^4$ (based on Ni analysis)*	Exposure time-days**
Haynes 25 #1	2.4	1.1	-	38
Haynes 25 #2	-	0.2	-	38
Hastelloy C	-	1.0	1.7	38
Hastelloy N	-	2.8	4.1	38
Haynes 25 #1	0.01	0.8	-	95
Haynes 25 #2	0.3	0.5	-	95
Hastelloy C	-	0.7	0.8	95
Hastelloy N	-	2.4	1.4	95

* The seawater blank was significantly less than the seawater samples for all the samples tested.

**Seawater sampled and changed at end of 38 days followed by additional exposure period of 57 days.

Table 3 shows the results of experiments in which specimens of Haynes 25 immersed in seawater were exposed to ionizing radiation. Considering the experimental spread of the corrosion rates of Haynes 25 in Table 2 and the control specimens in Table 3 there appears to be no significant effect of ionizing radiation of the corrosion rate of Haynes 25, at least not for the doses absorbed.

Active Specimens

The corrosion rate of Hastelloy C and Hastelloy N, based on their respective cobalt, nickel and chromium analysis, are shown in Figures 6 through 11 for various conditions of aeration, deaeration and movement of seawater past the specimen. The circles, squares and triangles represent the corrosion rates calculated on the basis of the cobalt, nickel and chromium analysis, respectively. The experimental error shown in the figures represent the average deviation of the average of two or three replicate determinations.

Fig. 6 Corrosion Rate of Hastelloy C in Aerated Seawater

Fig. 7 Corrosion Rate of Hastelloy C in Aerated, Moving (0.7 ft/sec) Seawater

Fig. 8 Corrosion Rate of Hastelloy C in Deaerated, Moving (0.7 ft/sec) Seawater

Fig. 9 Corrosion Rate of Hastelloy N in Aerated Seawater

Fig. 10 Corrosion Rate of Hastelloy N in Aerated, Moving (0.7 ft/sec) Seawater

Fig. 11 Corrosion Rate of Hastelloy in Deaerated, Moving (0.7 ft/sec) Seawater

TABLE 3

Seawater Corrosion Rates of Inactive Haynes 25 in a Radiation Field

	mpy $\times 10^4$ (based on Co analysis)	mpy $\times 10^4$ (based on Cr analysis)	Exposure Time (days)	Absorbed Dose (rads)
<u>Cobalt-60 Irradiation</u>				
Test Specimen	14	3	35	1.7×10^8
Control Specimen	3.2	1.4	35	0
<u>Van de Graff Irradiation</u>				
Test Specimen	13	2	20	10^5
Control Specimen	10	4.8	20	0

The general feature that is most apparent is the decrease in corrosion rate with increasing time of exposure. There is also an overall decrease in the experimental error of replicate determinations at the longer times, suggesting that the specimens are corroding more uniformly at the longer exposure times.

There appears to be some indication of differential leaching of the alloy components of Hastelloy C and Hastelloy N. Disregarding the data at 18 days because of the large errors involved, the average corrosion rate of the two alloys based on the cobalt analysis are lower than those based on the nickel and chromium analysis. In the case of Hastelloy C (again neglecting the data at 18 days) the average corrosion rates calculated on the basis of the chromium analysis are higher than those calculated on the basis of the cobalt and nickel analysis. The magnitude of the differential leaching of the cobalt, nickel and chromium components is not significantly large.

No significant effect of aeration, deaeration and movement of seawater past the specimen on the corrosion rate was observed for Hastelloy C and Hastelloy N.

The overall average corrosion rates for Hastelloy C and Hastelloy N, as a function of exposure time are presented in Fig. 12. These overall averages combine the average data points of the cobalt, nickel and chromium analysis for all sets of exposure parameters. It is felt that these

overall averages, together with their respective standard deviations, are of more useful significance than the single averages shown in Figures 6 through 11. The corrosion rates of the inactive specimens of Hastelloy C and Hastelloy N are included in the figure for comparison (large circles and squares).

Figures 13 and 14 show the corrosion rates of Haynes 25 calculated on the basis of the cobalt and chromium analyses, respectively. The circles, squares and triangles represent the corrosion rates for the exposure conditions of aeration, deaeration and movement of seawater past the specimen, respectively. The experimental error shown for the circles and squares represent the average deviation of the average of two replicate determinations; the triangles represent a single determination.

As in the case of the Hastelloys, the exposure parameters did not significantly affect the corrosion rate of Haynes 25. On the other hand, there appears to be significant differential leaching of the cobalt and chromium components of Haynes 25. The corrosion rates calculated on the basis of the cobalt analysis are about two orders of magnitude lower than those calculated on the basis of the chromium analysis. The low cobalt results for Haynes 25 are not an experimental artifact due to loss of cobalt activity by adsorption on the pyrex and polyethylene components of the test cell since larger amounts of cobalt activity, a factor of 20 higher, was released from the Hastelloy alloys.

The cobalt-based corrosion rate is in contrast to that found for the inactive Haynes 25. However, because of the extremely small amount of cobalt released to the seawater (10^{-3} $\mu\text{g/day}$ compared to 10^{-1} $\mu\text{g/day}$ for nickel and chromium) it is felt that the cobalt results for the active Haynes 25 are more reliable than those for the inactive Haynes 25 on the basis of the method of analysis. The amount of cobalt-60 from the active Haynes 25 was determined by direct counting of the seawater whereas the determination of cobalt from the inactive Haynes-25 involved numerous manipulations of the seawater. The difficulty in analyzing for the inactive cobalt is reflected in the experimental scatter of the cobalt data in Table 2.

The results of the experiment in which an active specimen of Haynes 25 immersed in seawater was exposed to ionizing radiation in the USNRDL Cobalt-60 Gamma Source is shown in Table 4. The corrosion rates based on the chromium analysis indicate that there is no significant effect of cobalt-60 gamma radiation on the corrosion rate of Haynes 25. The results based on the cobalt analysis are not included because of cobalt-60 contamination of the test seawater from the cobalt-60 gamma source.

The possibility of loss of activity by adsorption on the test cell components (which would lead to lower corrosion rate values) was investigated. A polyethylene specimen holder, a section of tygon tubing and a

Fig. 12 Overall Average Corrosion Rates of Hastelloy C and Hastelloy N. Large symbols represent inactive specimens, small symbols represent active specimens.

Fig. 13 Corrosion Rate of Haynes 25 in Seawater Based on the Amount of the Cobalt Component Released to Seawater

Fig. 14 Corrosion Rate of Haynes 25 in Seawater Based on the Amount of the Chromium Component Released to Seawater

TABLE 4

Corrosion Rate of Active Haynes 25 in a Cobalt-60 Radiation Field

	mpy $\times 10^4$ (based on Cr analysis)	mpy $\times 10^4$ (based on Co analysis*)	Exposure Time (days)**	Absorbed Dose (rads)
Test Specimen	7.0	-	20	10^8
Control Specimen	4.9	0.23	20	0
Test Specimen	4.3	-	41	2×10^8
Control Specimen	3.7	0.085	41	0
Test Specimen	2.0	-	87	4×10^8
Control Specimen	3.0	0.065	87	0

* Cobalt-60 contamination of test solution.

**Seawater sampled and changed at end of 20, 41 and 87 days (incremental exposure times of 20, 21 and 46 days).

teflon plug stopcock, all from an operating test cell, were counted in the pulse height analyzer system. No significant amount of activity was found. Further, it has been reported¹⁷ that there is no significant amount of adsorption of tracer amounts of cobalt and chromium in seawater on pyrex glass for test periods up to 6 months.

No visible change in the surface texture of any of the specimens was observed at the end of 98 days. Comparison of surface profile measurements made before and after exposure (Fig. 4) showed no noticeable change in the surface roughness of the specimens.

Comparison of the corrosion rates obtained from the two techniques used, i.e., (1) neutron activation analysis of corrosion products in seawater in which inactive alloy specimens were exposed, and (2) measurement of the concentration of radioactivity leached into the seawater from active alloy specimens, indicates that there was only a small effect of the gamma recoil from neutron activation of the alloy specimens. This observation is significant in that neutron activated specimens can be used in future experiments with these alloys, thus avoiding the contamination problems always present in performing activation analysis.

Table 5 presents a comparison of the corrosion rates observed here with other reported values.*

TABLE 5

Comparison of Corrosion Rates with Other Reported Values

Corrosion Rate (mpy)			
Haynes 25	Hastelloy C	Hastelloy N	References
-	10^{-2}	-	18
2×10^{-1}	2×10^{-1}	-	19 ^a
1×10^{-2}	2×10^{-2}	3×10^{-2}	10
4×10^{-4} (Cr)	5×10^{-5}	1×10^{-4}	this work ^b
1×10^{-6} (Co)			

a. Seawater velocity past the specimens was 128 ft/sec.

b. Corrosion rate of active specimens at end of 98 days; Haynes 25 values reported for chromium and cobalt analyses.

The data from references 18 and 19 are based on weight loss measurements; the data from reference 10 are based on electrochemical measurements.

Since the corrosion rates of these alloys are so small, it is felt that the weight loss method is a relatively poor one in terms of its sensitivity. The electrochemical technique of corrosion rate measurement is more sensitive by comparison.

The relatively high values reported for Haynes 25 and Hastelloy C in ref. 19 are probably due to an erosion effect rather than corrosion because of the very high velocity of seawater past the specimen. The other literature values are more comparable to the present results. The difference between these corrosion rates is such that it is of crucial importance relative to the deployment of some SNAP devices. For example, if the containment of Pu^{238} (half life of 89 years) is considered, a container with a 40 mil wall will provide containment between 20 and 1000 half lives depending upon whether the uniform corrosion rate is 2×10^{-2} or 5×10^{-5} mpy, respectively.

*Corrosion rate measurements of Hastelloy C have been conducted by Martin Company. Reference to the work can be found in their monthly progress report for March 1965 (Classified).

It must be kept in mind, however, that the corrosion rates obtained in this work were measured under conditions that would tend to give low rates (i.e., uniform low specimen temperatures, no thermal stresses). More realistic exposure conditions such as thermal gradients within the specimen, thermally shocked specimens and effects of galvanic couples may very well produce higher corrosion rates. In this respect the corrosion rates measured in this work might be considered to approximate the minimum values for these alloys. There is clearly a need for further study involving the use of the more realistic exposure conditions mentioned above.

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