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CORROSION IN SIMULATED PRTR
FUEL ELEMENT SURFACE CREVICES

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CORROSION IN SIMULATED PRTR
FUEL ELEMENT SURFACE CREVICES

By

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CORROSION IN SIMULATED PRTR
FUEL ELEMENT SURFACE CREVICES

ABSTRACT

Two out-of-reactor tests were conducted to study corrosion of Zircaloy-2 in crevices with geometries similar to those existing on heat transfer surfaces of Plutonium Recycle Test Reactor fuel elements. The tests were conducted in high temperature (293 °C), lithiated water at a pH of about 10 and used electrically heated, Zircaloy-2 clad test assemblies. Heat flux associated with heated crevices was about 1100 W/in². Examination after 70 days of loop exposure revealed test assembly cladding penetration, up to 0.007 in., in heated crevices. It is felt that the accelerated attack was the result of the concentration of lithium hydroxide by local boiling.

TABLE OF CONTENTS

I. INTRODUCTION	1
II. SUMMARY	1
III. DESCRIPTION OF TESTS	2
A. Test Assembly Description	2
B. Test Conditions	4
IV. TEST RESULTS.	5
V. DISCUSSION OF TEST RESULTS	12
VI. ACKNOWLEDGEMENTS.	14
APPENDIX A: TF-3 Water Analyses (December 1964 and January 1965)	15
APPENDIX B: Calculated Temperatures	18

CORROSION IN SIMULATED PRTR FUEL ELEMENT SURFACE CREVICES

I. INTRODUCTION

This report describes tests conducted to study corrosion in crevices with geometries similar to those existing on the heat transfer surface of Plutonium Recycle Test Reactor (PRTR) fuel elements. The main objective of the tests was to determine if crevice geometry is sufficiently pronounced to contribute to accelerated, localized corrosion when reactor parameters are adjusted to initiate local boiling on fuel element surfaces.

Zircaloy-2 is used as cladding material for the fuel elements of PRTR, a vertical pressure tube reactor, moderated and cooled with heavy water, with a thermal power rating of 70 MW. The primary coolant is maintained in an alkaline condition, primarily to reduce the concentration of corrosion products in the coolant. Lithiated coolant, at a pH of 10 ± 0.5 , is used. Components, which can create crevices on heat transfer surfaces, are added to clad fuel elements to maintain spacing, promote coolant mixing, and increase the strength of 19-rod cluster fuel assemblies.

Previous investigations using lithiated water at pH 10.5 and test conditions resulting in nucleate boiling on specimen surfaces has revealed that "either Zircaloy-2 or Zircaloy-4 will undergo crevice corrosion...".⁽¹⁾

II. SUMMARY

Test results indicate that Zircaloy fuel element surface crevices which will allow the concentration of chemical additives by local boiling should be avoided if lithiated coolant is used.

Examination after exposure revealed corrosion product deposits in almost all of the heated crevices. Microscopic examination of polished, mounted sections showed that the tests resulted in heating unit cladding penetration, up to 0.007 in., in areas immediately adjacent to those where bands had contacted the heating unit.

Individual test assemblies used in the two out-of-reactor tests consisted of cartridge type, Zircaloy-2 clad, electric heating units with PRTR fuel element components attached to the heating unit surface. Each of the heating units was equipped with two helically wound wire wraps and a circumferential band since the use of similar components can result in crevices on the heat transfer surface of actual fuel elements.

Test assemblies were installed in vertical test sections of out-of-reactor, stainless steel, recirculating water loops; temperature, pressure and surface heat flux was 293°C , 1365 psia, and 1100 W/in^2 , respectively. Tests were conducted in lithiated water at a pH of about 10; exposures attained were 60 and 70 days. Calculations indicate that test conditions resulted in local boiling in the heated crevices.

III. DESCRIPTION OF TESTS

Two out-of-reactor tests have been conducted to study corrosion in crevices with geometries similar to those existing on the heat transfer surface of PRTR fuel elements. Both tests were conducted in high temperature, high pressure lithiated water at a pH of about 10 and used electrically heated Zircaloy-2 clad, test assemblies.

A. Test Assembly Description

The basic component of an individual test assembly, shown in Figures 1 and 2, was a cartridge type, Zircaloy-2 clad, electric resistance heating unit. Some individual rods of the 19-rod, Zircaloy-2 clad, cluster type fuel element assembly used at the PRTR are helically wrapped with wire. The wire wraps maintain rod spacing and promote coolant mixing. Bands, which surround the cluster at several elevations, are also used to increase rigidity. Since the use of these components can result in crevices on the heat transfer surface of actual fuel elements, each of the two heating units was equipped with two wire wraps and a single "cluster" band.

FIGURE 1
Crevice Corrosion
Test Assembly

FIGURE 2
Installed Test Assembly

Using 220 V, 60 cycle, ac, test assembly heat generation rate was about 2400 W. Heating unit diameter and heated length were $\frac{1}{2}$ and $1\frac{3}{8}$ in., respectively. These dimensions correspond to a surface heat flux (along the heated length) of approximately 1100 W/in^2 . The Zircaloy-2 wires (0.073 in. diam) were helically wrapped on the heating unit and then attached by spot welding the ends of the wire to the cladding. The wire wraps contacted both the heated and unheated length of the heating unit. The 0.19 in. wide "cluster" band, consisting of a double wrap of 0.015 in.

thick Zircaloy-2, was installed at the approximate mid-point of the $1\frac{3}{8}$ in. heated length. The double wrap formed a continuous crevice. Except at two contact points, the band was held away from the surface of the heating unit by the previously installed wire wraps. The band was held in position by small resistance welds where the ends of the band overlapped; the welds did not penetrate the inner wrap of the band (wrap closest to the heating unit).

Except for the wire wraps, Zircaloy surfaces were not autoclaved (prefilmed) prior to exposure.

B. Test Conditions

Both of the tests were conducted in an out-of-reactor, stainless steel, recirculating water loop (designated TF-3) located near KE-Reactor. Test assemblies were installed in vertical test sections of this high pressure, high temperature loop. The assemblies were inserted and removed from the top of the test section. A metallic compression fitting formed the pressure seal between the test section and the test assembly. As shown in Figure 2, flow entered and exited the 1 in. ID test section from the sides. The top or inlet connection was 1 in. below the top of the test section; the inlet and outlet connections were 6 in. apart. The "cluster" bands of the test assemblies were centered between the inlet and the outlet.

High water purity was maintained with a feed and bleed arrangement. Deoxygenated, deionized feed water was adjusted to the desired pH with cation ion exchange resin regenerated to the lithium form; this method maintained loop water pH near 10. Results of chemical and emission analysis of TF-3 samples for December, 1964 and January, 1965 are presented in Appendix A to illustrate the degree of water quality during the test.

Test section flow rate was about 14 gal/min. Assuming annular geometry this value corresponds to calculated values of 1.3×10^6 lb/(hr)(ft²) and 7.6 ft/sec for mass and linear velocity, respectively. Bulk water temperature and pressure were 293 °C and 1365 psia, respectively. The pressure corresponds to a saturation temperature of 307 °C; bulk boiling was suppressed during the tests.

Calculated values for test assembly surface temperature and temperature required for local (surface) boiling are 358 and 313 °C, respectively. The equations used to calculate these temperatures are presented in Appendix B. The method of calculation for surface temperature assumes nonboiling, forced convection conditions, and annular geometry. The assumption of annular geometry neglects the effect (on heat transfer of the wire wraps and the "cluster" bands). It is felt that calculations indicate that local boiling existed in all heated crevices, i.e., wall or surface temperatures were sufficiently above the saturation temperature to initiate local, subcooled boiling.

IV. TEST RESULTS

Both of the tests were terminated by heating element failure after 60 and 70 day exposures. The appearance of the two test assemblies after exposure revealed that, in general, loop exposure resulted in the formation of an adherent, shiny, black oxide film on Zircaloy surfaces exposed to the high temperature, flowing water. Weld areas at the end of wire wraps were usually filmed with a white deposit. The shiny appearance of the portions of the heated length exposed to flowing water was evident only after removal of a second, slight, film of a substance that could be removed easily by wiping.

Contact areas on the heated length were immediately apparent after removal of the wire wraps and the "cluster" bands. Some of the contact areas associated with the test assembly with the 60 day exposure are shown in Figure 3. The corrosion and penetration at the band contact area shown in this figure is the most extensive revealed by visual examination. Other band contact areas were obvious because of discoloration, but cladding penetration was not immediately apparent. The corrosion and penetration associated with the wire wrap contact area shown in Figure 3 is typical of that where wire wraps contacted the heated length.

It was necessary to section the heating units to determine the extent of cladding penetration in the crevice regions. As shown in Figure 4, the sections allowed the examination of the portion of the heating unit surface immediately adjacent to areas where the wire wraps and "cluster" band

FIGURE 3

Areas where "Cluster" Band and Wire Wraps
Contacted Heating Unit

FIGURE 4
Locations of Sections

had contacted the heating unit, i.e., areas where the wire wraps and band formed surface crevices. Microscopic examination of polished sections revealed gray-white corrosion product deposits in almost all of the heated crevices. The examination showed that the tests resulted in heating unit cladding penetration, up to 0.007 in., in areas immediately adjacent to those where bands had contacted the heating unit.

The extent of cladding attack associated with two "cluster" band contact areas is illustrated in Figures 5 and 6, photomicrographs of sections from the test assemblies with 70 and 60 day exposures, respectively. Maximum surface penetration shown in Figures 5 and 6 is about 0.007 and 0.004 in., respectively. Penetration associated with the remaining band contact areas was 0.001 to 0.002 in. Figure 6 is a section through the same corroded area shown in Figure 3.

Microscopic examination of sections from the test assembly with the 70 day exposure revealed sharply defined imprints, up to 0.002 in., where wire wraps contacted the heated length of the heating unit; however, no corrosion product deposits were found in or adjacent to the imprints. Examination of sections from the test assembly with the 60 day exposure revealed similar imprints or penetration which, as shown in Figure 7, were almost filled with corrosion product deposits. The depth of penetration into the heating unit cladding, shown in Figure 7, is about 0.002 in.

Visual examination of the "cluster" band from the test assembly with the 60 day exposure revealed localized corrosion and discoloration on the band where it had contacted the heating unit. Corrosion products were found on the inner and outer side of the inner wrap and on the inner side of the outer wrap, i.e., in the crevice formed by the two wraps of the band as well as in the crevice formed by the inner wrap of the band and the heating unit. The corrosion associated with the inner wrap is illustrated in Figure 8, a photomicrograph of a section through one of the localized corrosion areas; the maximum thickness of corrosion product deposit shown is about 0.003 in.

Corrosion Product
Deposit

Cladding Surface
Before Corrosion

Heating Unit
Cladding

Neg. 56595B (~100X)

FIGURE 5
Crevice Corrosion Associated with "Cluster" Band Contact,
70 Day Exposure

Neg. 465661C (~75X)

FIGURE 6

Crevice Corrosion Associated with "Cluster" Band Contact,
60 Day Exposure

Corrosion Product
Deposit

Heating Unit
Cladding Surface

Neg. 465660C (~75X)

FIGURE 7

Crevice Corrosion Associated with Wire Wrap Contact,
60 Day Exposure

Inner Wrap Contacted Outer
Wrap on this Side

Corrosion
Product
Deposit

"Cluster" Band
Inner Wrap

Inner Wrap Contacted Heating Unit on this Side

Neg. 465662 (~75X)

FIGURE 8

Inner Wrap of "Cluster" Band,
60 Day Exposure

The deposits of gray-white corrosion product in the various crevices appear identical to those, identified as zirconium oxide, formed during similar tests except that crevice geometry was slightly different. Etching sectioned mounts to define hydride platelets revealed that the hydride content of metal adjacent to corroded areas of the cladding and of the "cluster" band was <10 and 20 to 35 ppm, respectively. The hydride concentration adjacent to the localized corroded areas was not significantly different from that found in uncorroded areas.

V. DISCUSSION OF TEST RESULTS

Extensive corrosion was limited to the narrowest portion of heated crevice regions. Except for weld areas, the corrosion resistance of surfaces away from crevice region (i. e., surfaces exposed to high temperature, flowing water) appeared to be excellent. Cladding corrosion, where wire wraps contacted the unheated length of the heating unit, was negligible. The double wrap of the "cluster" band formed a continuous crevice; corrosion was limited to the portion of the crevice where the band contacted the heated length of the heating unit.

Although tests were not designed to define the corrosion mechanism, it is felt that the accelerated corrosion can be attributed to the concentration of lithium hydroxide, by boiling, in the restricted flow area of the crevices.

Tests by other investigators have shown that the use of concentrated, lithiated solutions can result in a marked decrease in the corrosion resistance of Zircaloy-2. According to Coriou,⁽²⁾ "...in lithia solutions at 360 °C the Zircaloy undergoes very rapid corrosion for lithia concentrations...in the neighborhood of 1 g/liter..."; after tests of Zircaloy in 360 °C water, Hillner and Chirigos⁽³⁾ stated "...with more concentrated lithiated solutions (5×10^{-3} molar and above) a severe loss of corrosion resistance was clearly indicated." The preceding suggests that the localized corrosion was solely the result of the environment existing in the crevice. The effect of differences in environment between crevice and noncrevice regions, e. g., concentration differences of ionic species, cannot be completely disregarded when

discussing possible contributing factors. The concentration of ionic species in heated crevices could have been orders of magnitude higher than that of surrounding environment. i.e., the water at the mouths of the crevices.

Corrosion-produced hydrogen pickup appeared to be slight. The low hydride concentrations adjacent to corroded areas is believed to be the result of hydride diffusion into the relatively large, often cooler, volume of uncorroded metal.

Other items that should be mentioned prior to ending the discussion concern crevice geometry, exposure times, and future tests. It is felt that crevice corrosion associated with wire wrap contact areas was not as extensive as that associated with "cluster" band contact areas because the crevice geometry associated with wire wraps was more open.

Longer test runs were desired; but as previously indicated, both tests were terminated by heating element failure. Tests are currently being conducted with lower surface heat fluxes in an attempt to obtain longer exposure times. Tests using surface heat fluxes higher than 1100 W/in^2 are also planned. The tests by Anderson and McGoff⁽¹⁾ to study crevice corrosion of Zircaloy-2 and Zircaloy-4 under nucleate boiling conditions revealed that "Much more drastic attack occurs in lithiated water than in ammoniated water." Tests similar to those described in this report, except that ammoniated water is used, may also be desired.

In conclusion, test results indicate that Zircaloy fuel element surface crevices of a geometry which will allow the concentration of chemical additives by boiling should be avoided if lithium hydroxide is used to adjust the pH of the reactor coolant.

VI. ACKNOWLEDGEMENTS

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APPENDIX A

TF-3 WATER ANALYSES

(December, 1964 and January, 1965)*

* Test assembly with 70 day exposure installed in TF-3 from 9-29-64 to 12-14-64; test assembly with 60 day exposure, from 12-14-64 to 2-14-65.

TABLE A-I
RESULTS OF CHEMICAL ANALYSIS

<u>Date</u>	<u>January, 1965</u>			<u>December, 1964</u>		
	<u>Cl⁻, ppm</u>	<u>pH</u>	<u>Specific Resistance (Ω)(cm)</u>	<u>Cl⁻ ppm</u>	<u>pH</u>	<u>Specific Resistance (Ω)(cm)</u>
1	<0.1	9.22	72,500			
2				<0.1	9.74	119,000
3						
4				<0.1	9.31	150,000
5	<0.1	9.79	98,000			
6	<0.1	9.95	86,500			
7						
8	<0.1	9.85	77,500			
9				<0.1	10.18	52,000
10						
11	<0.1	9.92	80,000	<0.1	10.05	67,000
12	<0.1	9.90	80,500			
13	<0.1	9.85	66,000			
14				<0.1	9.90	65,000
15	<0.1	10.00	52,000			
16				<0.1	9.92	76,000
17						
18						
19	<0.1	10.00	64,000			
20	0.38	9.88	59,500			
21						
22	<0.1	9.78	89,500			
23				<0.1	9.94	66,000
24						
25	<0.1	9.55	185,000			
26						
27	<0.1	10.00	47,200			
28				<0.1	9.73	73,000
29	<0.1	9.75	82,000			
30				0.8	9.55	98,000

TABLE A-II

RESULTS OF EMISSION ANALYSIS

<u>Date</u>	<u>12-9-64</u>	<u>12-23-64</u>	<u>1-5-65</u>	<u>1-13-65</u>	<u>1-27-65</u>
	<u>parts per billion</u>				
Iron	<10	<10	<10	<10	<10
Nickel	<10	<10	<10	<10	<10
Chromium	>500	<10	<10	<10	<10
Copper	13	14	<10	<10	<10
Aluminum	110	<10	<10	<10	<10
Zirconium	<4	<4	<4	<4	<4
Manganese	<4	<4	<4	<4	<4
Cobalt	<10	<10	<10	<10	<10
Calcium	<10	<10	<10	<10	<10
Magnesium	90	<10	<10	<10	<10
Lead	<20	<20	<20	<20	<20
Zinc	<10	<10	<10	<10	<10

APPENDIX B
CALCULATED TEMPERATURES

CLADDING (SURFACE) TEMPERATURE

The calculated surface temperature was based on a surface heat transfer coefficient obtained from the following correlation for predicting h for fluids flowing in an annular space:⁽⁴⁾

$$h = 0.029 C_p G \left(\frac{\mu}{D_i G} \right)^{0.2} \left(\frac{k}{\mu C_p} \right)^{0.67} \left(\frac{\mu}{\mu_s} \right)^{0.14} \left(\frac{D_o}{D_i} \right)^{0.15} \quad (1)$$

where,

h = the heat transfer coefficient

C_p = the heat capacity at the average bulk temperature of the fluid

G = the mass flow rate

μ = the viscosity at the average bulk temperature of the fluid

D_i = the diameter of the inner boundary annulus (the outside diameter of the heating unit)

D_o = the diameter of the outer boundary annulus (the inside diameter of the vertical test section)

k = the thermal conductivity at the average bulk temperature of the fluid

μ_s = the viscosity at the temperature of the heated surface.

The surface temperature was then calculated from the following:

$$T_s = T_b + (q/Ah) \quad (2)$$

where,

T_s = the temperature of the heated surface

T_b = the average bulk temperature

q/A = the surface heat flux.

The above equations assume that test assembly geometry is cylindrical, i. e., test assemblies were not equipped with "cluster" bands and were wraps. The radial temperature differences caused by the thermal resistance of these components and the gap between these components and heating unit surface are not included in calculated values for surface temperature. Axial temperature gradient in the vertical test section was also neglected because of the short heated length.

CALCULATED TEMPERATURE REQUIRED FOR SURFACE BOILING

Surface temperatures slightly higher than the saturation temperature would be required to initiate local surface boiling; an estimate of the superheat required was obtained from the following correlation:⁽⁵⁾

$$T_{\text{boil}} = T_{\text{sat}} + 1.9(q/A)^{0.25} e^{-P/900} \quad (3)$$

where,

T_{boil} = the heated surface temperature at local boiling, °F

T_{sat} = the saturation temperature of the bulk water, °F

q/A = the surface heat flux in Btu/(hr)(ft²)

P = the fluid pressure, psia.

SAMPLE CALCULATIONS

Test Conditions

$$T_b = 293 \text{ }^{\circ}\text{C} (560 \text{ }^{\circ}\text{F})$$

$$P = 1365 \text{ psia}$$

$$\text{Flow rate} = 14 \text{ gal/min} (7.6 \text{ ft}^3/\text{sec})$$

$$q/A = 1100 \text{ W/in.}^2 [550,000 \text{ Btu/(hr)(ft}^2)]$$

Parameters

$$T_{\text{sat}} = 307 \text{ }^{\circ}\text{C} (584 \text{ }^{\circ}\text{F})$$

$$C_p = 1.33 \text{ Btu/(lb)(}^{\circ}\text{F)}$$

$$\rho = 46 \text{ lb/ft}^3$$

$$\mu = 0.11 \text{ cP}$$

$$\mu_s = 0.095 \text{ cP (estimated)}$$

$$D_o = 0.0834 \text{ ft}$$

$$D_i = 0.0416 \text{ ft}$$

$$G = 1.26 \times 10^6 \text{ lb/(hr)(ft}^2)$$

$$k = 0.33 \text{ Btu/(hr)(ft)(}^{\circ}\text{F)}$$

$$k/\mu C_p = 0.94$$

$$\mu/D_i G = 5.09 \times 10^{-6}$$

From Equation (1)

$$h = (0.029)(1.33)(1.26 \times 10^6)(5.09 \times 10^{-6})^{0.2} (0.94)^{0.67} \\ (1.16)^{0.14} (2)^{0.15} = 4.65 \times 10^3 \text{ Btu/(hr)(ft}^2\text{)(}^\circ\text{F)}.$$

From Equation (2)

$$T_s = 560 + (550,000/4650) = 678 \text{ }^\circ\text{F} = 358 \text{ }^\circ\text{C}.$$

From Equation (3)

$$T_{\text{boil}} = 584 + (1.9)(550,000)^{0.25} e^{-(1365/900)} = 595 \text{ }^\circ\text{F} = 313 \text{ }^\circ\text{C}.$$

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