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EVALUATION OF ZIRCALOY-2 PROCESS TUBES CONTAINING NONSTANDARD SPACERS

B. Griggs

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EVALUATION OF ZIRCALOY-2 PROCESS TUBES CONTAINING NONSTANDARD SPACERS

INTRODUCTION

The discovery of a hydride case on the inner surface of the K Reactor Zircaloy tubes in 1965 led to an extensive investigation of its cause and remedial measures.[1,2,3] The most probable mechanism determined for the phenomenon was the galvanic coupling of the 6063 alloy aluminum spacers to Zircaloy-2 process tubes.[4,5] This coupling allows the corrosion produced electrons to migrate from the corroding aluminum spacer to the Zircaloy tubes where the excess electrons result in the reduction of hydrogen ion from water molecules on the surface to produce nascent hydrogen. Part of this nascent hydrogen then diffuses into the tube material precipitating as zirconium hydride. The balance of the hydrogen atoms recombine to form hydrogen gas which is dissolved or swept away by the flowing water. This simplified presentation ignores all other potential cathodic reactions such as $Fe^{+3} \rightarrow Fe^{+2}$, $O_2 \rightarrow OH^-$.

Three remedial actions were indicated which would interrupt this reaction: (1) electrically insulate the spacer from the process tube, thus interrupting the flow of electrons; (2) stop the corrosion of the spacers and thus the corrosion produced electrons; (3) make the spacers from a material which would have insufficient potential to reduce hydrogen on the surface of the Zircaloy, such as stainless steel, nickel, etc. All of these approaches were tested.[3] The purpose of this document is to present the hydrogen absorption and corrosion data relative to this testing.

EXPERIMENTAL

Alternate spacer concepts were charged in newly installed Zircaloy-2 tubes and operated in a standard manner.[2,3] When an experimental tube had completed its exposure with a special type of spacer charge, the tube was discharged and the rear five feet of the tube delivered

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to the Battelle-Northwest laboratory by Douglas United Nuclear for analyses. Frequently, the spacers were also sent in for examination. The region of the tube between 5 feet and 15 feet from the downstream end was sampled by making duplicate 9/32 inch punchings every longitudinal foot. These punchings were delivered to Battelle-Northwest for analyses. In addition, a 1 to 2 foot piece of the upstream end of the tube was salvaged for use as a blank to estimate the as-installed hydrogen content of the tube.

The downstream 5 foot section and the upstream 1 foot section were analyzed by cutting a 1/4 ring from the tube at appropriate positions with a wheeled pipe cutter or hack saw. The rings were cleaned of reactor deposits in boiling 10% oxalic acid and flash etched in standard nitric-hydrofluoric acid etch solution at room temperature for 15 to 30 seconds. The rings were then heated to approximately 1100°C in vacuum and the evolved hydrogen measured. The punchings were analyzed in a similar apparatus by the analytical section. Metallographic examination of the Van Stone flange was usually conducted.

The stainless steel and nickel plated spacers were cleaned for examination by treatment in boiling 10% oxalic acid. The anodized aluminum spacers were cleaned for examination in hot 40% nitric acid for a sufficient time to just remove the reactor formed film. Carbon steel spacers were cleaned with inhibited hydrocloric acid.

Results

The hydrogen analyses data for tubes are shown in Table I.

Metallographic examination of the downstream Van Stone flange was conducted for all the tubes with anodized spacers except 3172-W-2* and 4372-E-6. No hydriding was observed even though this flange is close to the untreated aluminum nozzle and frequently an aluminum gasket. Examination of the flanges of 3074-W-3, 2048-W-2, 1554-W-2, and 3181-W-2

*Tube numbers show the row and column in the reactor in the first four digits. The letter indicates the reactor W for K West and E for K East. The final digit indicates the numbers in the sequence of Zircaloy tubes in this location.

revealed no hydriding. Light hydriding was observed in 3667-W-2 and 2966-W-2 which were loaded with untreated aluminum spacers in this position. Tubes 4372-E-4, 2970-W-3, 3074-W-2, and 3075-W-2 were not examined.

The examination of spacers supports the following general observations.

Anodized and Sealed 6063 Aluminum (oxalic acid process). The spacers retained most of their anodized film even after exposures of 7 months. There were longitudinal scratches, especially on the supporting feet, where minor corrosion of the underlying aluminum had occurred. The decontamination of the spacers in the nitric acid was excellent with most spacers reduced to about 1000 cpm. The actual thickness of the anodized film remaining was not determined.

300 Series Stainless Steel Spacers. These spacers appeared relatively unaffected by their exposure. No weight change data were obtained.

Nickel Plated and Heat Treated Aluminum Spacers. These spacers displayed numerous spots where corrosion had penetrated the nickel coating. The corrosion of the base metal beneath the coating was frequently severe. Chemically plated nickel (~6% phosphorous) of about 1 and 2 mil thicknesses and electrically plated nickel were tested. Generally, the two types of plating behaved similarly. The 2 mil plate had fewer spots of penetration than the 1 mil plate (Figure 1). A weighed charge of nickel plated spacers was tested and the data shown in Table IV. Two heat treatments were tested, 400°C for 4 hours and 525°C for 1/2 hour, which gave different amounts of nickel diffusion into the base metal. Most plates were applied to standard 6063 aluminum alloy spacers, but a few 8001 aluminum alloy spacers were plated. The reactor performance of the various types was similar.

Carbon Steel Spacers. The carbon steel spacers were substantially pitted after cleaning. No original weight data was obtained so total corrosion cannot be determined. The cleaning of one of these spacers resulted in a 24 gram weight loss which would correspond to a penetration of 2 mils if the stripped oxide represented only corrosion product and the corrosion were uniform.

Five Special Anodized Spacers. Two charges of anodized spacers were exposed for 71 operating days, along with a control charge of plain, preweighed spacers to determine some of the important parameters of anodizing and sealing. The data from these charges is shown in Tables II and III.

DISCUSSION

The effectiveness of the various types of spacers in preventing hydrogen pickup in new tubes can be seen in Table I. Tubes 3075-W-2 and 3074-W-2 represent standard spacer charges of untreated 6063 aluminum alloy spacers for comparison. The hydriding of these tubes in four and seven months was substantially more than in tube 3966-W-2, which was charged with untreated 8001 aluminum alloy spacers. The different behavior of the two aluminum alloys was anticipated by laboratory tests.[6] The use of nickel plated aluminum, 300 series stainless steel, carbon steel, or anodized aluminum spacers prevented the hydriding in the downstream portion of the tube except for a few notable cases. Tube 3667-W-2 shows hydrogen pickup at 6 inches, which was traced to the inadvertent use of an untreated spacer at this position during part of its exposure.[7] Tubes 3667-W-2 and 3074-W-3 show hydrogen pickup in the region adjacent to the fuel charge beyond 104 inches. Tube 3667-W-2 has high results at 9, 10, and 13 feet, while tube 3074-W-3 is high only at 10 feet. Metallographic examination of 3667-W-2 at 10 feet showed a spotty hydride case.[7] The unexpected hydriding adjacent to the fuel charge suggests the stainless steel and nickel plated spacers might be shorting current leakage down the fuel column to the process tube. The electrical flow could then return via the low resistance tube to a place adjacent to its origin. Hydriding was not observed in tubes 4372-E-4, 2048-W-2, or 3181-E-2, indicating the mechanism of hydriding adjacent to the fuel charge was subject to interruption. An initial test of this hypothesis was tried in 1554-W-2 in which the electrical continuity of the spacers and the fuel was interrupted by a standard anodized aluminum spacer. This tube did not hydride adjacent to the fuel charge but a statistical number of such charges would have to be evaluated to prove the hypothesis. An additional side effect seen with the stainless steel spacer charges was the severe corrosion of the aluminum Van Stone flange gasket.[7]

The anodized aluminum spacer charges produced no observable hydride absorption in any of the nine tubes examined. Although there is a maximum variation of 4 ppm in the hydrogen content between front face piece of tube (blank) and the rear portion of the tube, there is probably sufficient variation in hydrogen content of the as-received tube in addition to analytical errors to account for this difference.

The lack of hydriding in the Van Stone flange which is adjacent to an aluminum nozzle and frequently an aluminum gasket is fortunate but not wholly consistent with the effect of spacers on the Zircaloy. Part of this inconsistency might be explained on the basis that the nozzle already has a substantial corrosion film and is not corroding as rapidly as freshly cleaned or new spacers. The gasket only exposes a very small surface area to the water and thus its effects could be spread out over the much larger cathode area of the adjacent Zircaloy tube. In addition, the gaskets are of 8001 aluminum which is less active than 6063. Several of the gaskets employed were asbestos and thus not reactive. If the well oxidized aluminum nozzle is the correct explanation for the lack of tube hydriding, then hydriding might be produced in the Van Stone flange area should chemically cleaned or new nozzles be installed. Prior to any substantial rear face decontamination or nozzle replacement, the effect of rapidly corroding, lightly oxidized nozzles should be clearly understood.

The three tube test run with special anodized spacers shown in Tables II and III clearly established the superiority of D.C. oxalic acid anodizing followed by 170°C water sealing. The time of sealing was not investigated; however, the temperature of sealing was shown to be of importance. The unsealed (sealed in service) coating of both the sulfuric and oxalic acid process was very inferior. The sealing treatment at 122°C was poor for the sulfuric acid coating and only marginal for the oxalic acid coating. Comparison to Table III shows the corrosion of untreated spacers was similar to the unsealed coating. The data from both tables indicate that the corrosion is not a function of position in the charge.

An examination of Table IV and Figure 1 will disclose that nickel plated spacers were substantially corroded during service. This result agrees qualitatively with results obtained earlier on chemically nickel plated spacers,[7] (electroless plating). Note the severe pitting of the rib on Sample 4 of Figure 1. This is in marked contrast to previous experiences with nickel plated aluminum fuels exposed in aluminum.

tubes,[8] which displayed no accelerated corrosion of aluminum under the nickel plate.

CONCLUSIONS

It has been shown that properly sealed, anodized aluminum spacers will prevent the hydrogen pickup by new Zircaloy-2 process tubes in the K Reactors. Indirectly, this data tends to confirm the hypothesis that the hydride formation is an electrical phenomenon associated with the corrosion of the aluminum spacers.

Stainless steel spacers prevent the hydride formation in tube sections where they are used but may accelerate a hydriding process adjacent to the aluminum clad fuel and the corrosion of the aluminum Van Stone flange gasket. "Insulating" the stainless steel spacers with anodized aluminum spacers may stop the upstream hydriding but insufficient data is available to establish this hypothesis.

Carbon steel spacers did not cause hydriding in one test but were substantially corroded themselves.

Nickel plated aluminum spacers did not cause hydriding adjacent to their position but probably gave rise to a hydriding adjacent to the fuel column. The nickel plate and the aluminum it protected suffered substantial localized corrosion in four months service.

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TABLE I

HYDROGEN CONCENTRATION OF PROCESS TUBES LOADED WITH NONSTANDARD SPACERS (ppm)

Tube Number	Exposure Months	Spacer Type	Blank Front	Approximate Distance from Downstream End (inches) 6	12	18	24	36	48	60	72	84	96	108	120	132	144	156	168
1357-E-2	6	A-5	9	8	9	9	10	9	9	8	10	10	10	10	12	10	10	10	
3966-W-3	7	A-1	16	13	13	12	12	12	13	14	14	15	15	15	15	14	14	15	15
2970-W-2	7	A	10	11	12	10	10	11	10	9	11	10	9	10	10	9	10	10	10
3172-W-2	7	A-4	14	18	15	13	14	13	16	16	12	13	12	13	12	13			
4372-E-6	6	A-6	9		8	7	8	10	9	7	10	8	8	8	10	10	10	9	
2577-E-2	7	A-1	8	12	12	9	10	10	9	8	10	10	9	10	10	10	13	11	9
2970-W-4	8	A-3	8	7	7	7	8	8	8	8	8	10	10	10	10	10	10	10	10
2571-E-2	7	A-2	9	8	11	10	10	10	9	8	10	11	10	10	10	10	12	9	12
4372-E-5	8	A-1	14	10	9	10	9	9	8	9	10	9	9	9	9	9	9	9	9
3074-W-3	4	SS		9	14	9	10	8	8	8	10	9	8	10	152	10	8	8	
4372-E-4	7	SS-1	9	9	8		6	8	8	7	11	11	12	12	11	11	12	11	11
2048-W-2	7	SS	9	9	10	7	7	8	5	8	8	8	8	7	8	9	9	9	9
1554-W-2	9	SS-2	6	6	7	8	8	7	8	7	8	8	7	8	8	8	9	8	8
[illegible]970-W-3	7	CS	7	6	7				8	8									
3667-W-2	4	Ni		43	15	12	14	15	12	14	20	13	12	106	120	13	11	*	13
[illegible]181-E-2	6	Ni		8	4	4	12	12	13	14	12	13	13	12	13	14	14	13	13
[illegible]075-W-2	4	Al	12	525	602		263	179	234	195	14	12		11	10	12	22	*	13
[illegible]966-W-2	6	Al-1	7	142	181	190	151	133	101	80	71	59	31	7	7	7	7	7	8
3074-W-2	7	Al	11	200		744	648	391	311	229									

A = Standard oxalic acid anodized 6061 aluminum; sealed.
A-1 = Reused A.
A-2 = "Sanford hard coat".
A-3 = Front face charged A.
A-4 = Low resistance A.
A-5 = Reclaimed A.
A-6 = Unsealed A.
Al-1 = 8001 aluminum alloy spacers, untreated.
Al = Standard 6063 aluminum alloy spacers, untreated.

SS = 300 Series Stainless Steel.
SS-1 = SS charged in grit blasted tube.
SS-2 = SS charged with one A at each end.
CS = Carbon steel.
Ni = Nickel plated 6063 aluminum.
* = Conflicting analyses obtained.

[redacted]

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TABLE II

SPECIAL ANODIZED SPACER CORROSION DATA

Piece Number(3)	Position Number From Rear	Anodic Treatment	Sealing Conditions	Weight Change As-Removed (2) (grams)	Weight Change As-Cleaned (2)	Cleaning Time (Minutes (1)
2-C 2-D	2 9	Oxalic Acid DC-2 mil	2 hours 170°C	+0.322 +0.280	-0.066 -0.161	30 65
1-C 10-D	1 1	Oxalic Acid DC-1 mil	2 hours 170°C	+0.239 +0.269	-0.086 -0.623	30 65
1-D 10-C	10 10	Oxalic Acid DC-1 mil	2 hours 170°C	+0.277 +0.288	-0.205 -0.225	65 30
3-C 3-D	3 8	Oxalic Acid DC-1 mil	2 hours 122°C	+0.279 +0.287	-0.324 -0.272	30 65
4-C 4-D	4 7	Oxalic Acid DC-1 mil	no seal no seal	-0.413 -0.446	-2.692 -2.649	5 5
8-C 8-D	8 3	Oxalic Acid AC-1 mil	2 hours 170°C	+0.313 +0.333	-2.444 -2.806	20 53
5-C 5-D	5 6	Sulfuric Acid DC-1 mil	2 hours 170°C	+0.179 +0.167	-0.488 -0.684	10 24
6-C 6-D	6 5	Sulfuric Acid DC-1 mil	2 hours 122°C	-0.201 -0.171	-3.256 -4.184	10 12
7-C 7-D	7 4	Sulfuric Acid DC-1 mil	no seal	-0.481 -0.436	-2.933 -3.094	5 5
9-C 9-D	9 2	no anodize	2 hours 170°C	+0.270 +0.279	-0.629 -0.575	20 53

(1) Cleaned in 40% HNO_3-100°C-5 to 60 minutes.
(2) Spacers surface area 4.1 dM^2 of 6063-T6 aluminum.
(3) Pieces No. C were exposed 71 operating days in tube 2561-E;
Pieces No. D were exposed 71 operating days in tube 2560-E.

TABLE III

STANDARD SPACER CORROSION DATA

Piece Number(1)	Position No. from Rear	Wt. Change As-Discharged grams	Wt. Change As-Cleaned(2) grams	Penetration By Wt. Change(3) mils
78-A	1	-1.92	-4.33	1.54
78-B	2	-1.74	-4.14	1.47
78-C	3	-1.64	-4.05	1.44
78-D	4	-1.74	-4.25	1.51
78-E	5	-1.78	-4.30	1.53
78-F	6	-1.74	-4.14	1.47
78-G	7	-1.82	-4.31	1.53
78-H	8	-1.81	-4.20	1.49
78-I	9	-0.92	-3.44	1.22
78-J	10	-1.84	-4.28	1.52

(1) Standard 6063 aluminum spacers of K-5 design exposed 71 operating days in Tube 2562-E.
(2) Cleaned in 2% CrO_3-5% H_3PO_4 solution at 100°C 30 minutes.
(3) Spacer area 4.1 dm^2.

TABLE IV

NICKEL PLATED SPACER CORROSION

Piece Number	Position Number From Rear	Plate Description (1)	Wt. Change As-Discharged grams (2)	Wt. Change After Cleaning (3) grams
X-11	2	Commercial	-0.066	
X-13	3	Commercial Autoclaved	-0.148	-0.339
X-3	4	BNW Plate Autoclaved	-0.625	-1.185
X-14	5	Commercial Autoclaved	-0.033	
X-15	6	Commercial	-0.066	-0.210
X-5	7	BNW Plate	-0.706	
X-16	8	Commercial	-0.076	
X-4	9	BNW Plate Autoclaved	-0.534	

(1) Standard aluminum spacers nickel electroplated and heat diffused 400°C 4 hours. Autoclaving was for 6 hours at 170°C. BNW plate was applied to 8001 aluminum alloy spacers. Commercial plate was applied to 6063-T6 aluminum alloy spacers.
(2) Exposed in tube 2559-E for 52 operating days.
(3) Cleaned in boiling 10% oxalic acid solution for 30 minutes.

[redacted]

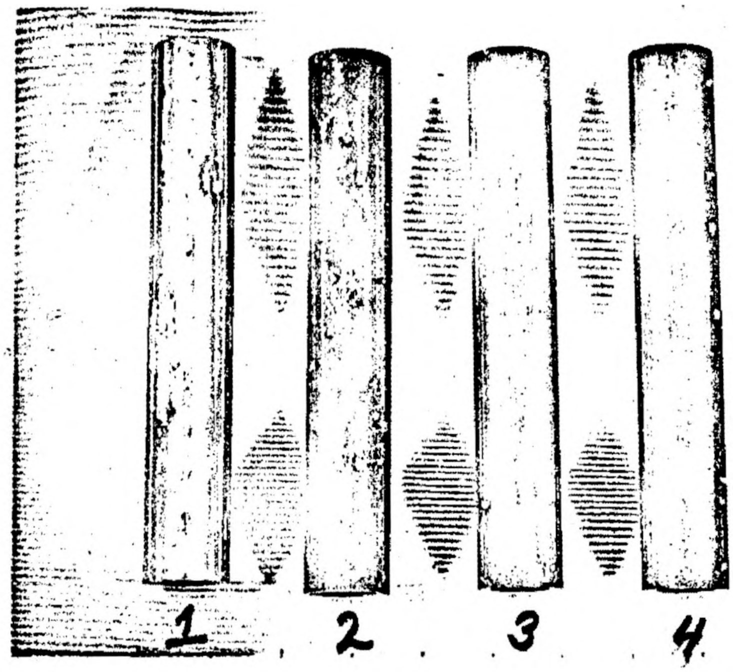

Figure 1

Nickel Plated Aluminum Alloy Spacers
After Exposure in Standard Reactor Usage

No. 1 - 1 mil plate applied on 8001 alloy at BNW; cleaned in 10% oxalic acid after 52 days in test.

No. 2 - 1 mil plate applied on 8001 alloy at BNW; as removed from test after 52 days.

No. 3 - 2 mil commercial plate on 6063 aluminum as removed from test after 52 days.

No. 4 - 1 mil commercial plate on 6063 aluminum as removed from test after 128 days. (Note severe pitting on rib.).

Samples 1, 2, and 3 were heat treated 400°C for 4 hours before test to diffuse the nickel electroplate partially into aluminum. Sample 4 was heat treated 1/2 hour at 525°C to diffuse the chemical nickel plate partially into the aluminum.

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