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HISTORICAL RECORD AND FINAL STATUS REPORT
OF K REACTOR ZIRCALOY HYDRIDE PROBLEM

W. K. Alexander

March 26, 1971

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HISTORICAL RECORD AND FINAL STATUS REPORT
OF K REACTOR ZIRCALOY HYDRIDE PROBLEM

INTRODUCTION

This document is intended to serve both as an historical record and final status report concerning the K Reactor hydriding problem which has been under study since its discovery in 1965. If neither of the K reactors are restarted, this will serve as an historical record which might be of some use to those using Zircaloy components in either the power reactor or chemical industries. (Its classification would limit such use.) If either of the K's are reactivated, hydriding will again become an active subject since its deleterious effects on the tubes will have a significant bearing on future reactor operations. No attempt will be made to review or report on all work done on this problem but a complete list of references is appended. It is assumed that copies, or micro-films, of these references will be available for some time to come.

SUMMARY AND CONCLUSIONS

A great deal of technology was developed although a complete explanation of the hydriding mechanism(s) was not achieved. Development of an engineered solution to the hydride problem was emphasized. An economic method of arresting hydrogen absorption in the K Zircaloy tubes was developed and practically proven. Final proof was to be obtained in June 1971. The following listing of established facts, as well as unanswered questions, summarizes the final status of the program:

- Hydriding of the K Reactor tubes was caused by the galvanic coupling between the tubes and 6063 aluminum alloy spacers. It did not occur in the active zone where the tube was in contact with X-8001 aluminum alloy clad fuel elements. This difference in behavior was not explained.
- Hydriding occurred in two forms: a layer of almost pure ZrH_2 platelets on the inner surface (commonly called the case layer) and a uniformly distributed increase of hydrogen content in the underlying base metal. Case layers up to 15 mils in thickness were found. The average was 3-4 mils. Maximum base metal content was 175 ppm, with an average of ≈ 125 ppm at the time of shutdown. All tubes had both forms of hydriding to some extent.

- Elimination of the galvanic couple by use of anodized aluminum spacers or spacers made from metals cathodic to Zircaloy, prevented hydrogen absorption in new tubes. Conversion to anodized aluminum spacers occurred in early 1968.
- The use of anodized aluminum spacers may have retarded, but did not eliminate, hydrogen uptake in the base metal in tubes containing hydride case layers. This was believed to be due to diffusion from the hydrogen-rich case layer.
- Sandblasting to remove surface hydrides with subsequent use of anodized spacers eliminates all further hydriding but it is not practical to remove the case layer in this manner without excessive removal of sound metal. This results from the nonuniform distribution of the case layer.
- Electrochemical removal of the case layer by a two step process--anodic stripping followed by electro etch--leaves a bright, highly specular surface which will not rehydride when anodized spacers are used. However, practical application of this technique was deferred because of budget restrictions.
- Although a reliable prediction of when, and at what rate, tubes would fail as the result of hydriding cannot be made in the absence of failure statistics, it is concluded that any tube subjected to impact or flexure could fail because of the extreme brittleness of the base metal. Failure at the Van Stone flanges appears most likely because of their vulnerability to bending as the result of thermally induced movement, charging loads, or nozzle impacts.
- Up to the time of the KE shutdown, five Zircaloy tube failures had occurred. Four of these failed at Van Stone flanges. The location of the failure in the fifth tube was not found. None of these were solely related to hydriding, although hydrogen embrittlement may have contributed to two failures which occurred in 1970.
- An analysis of available data leads to the conclusion that removal of hydride case layers is economically justified if the K Reactors were to be reactivated for a production campaign exceeding 3 to 4 years.
- The cost of electrochemical hydride removal is estimated at 10 percent the cost of replacement with Zircaloy tubes and 50 percent the cost of replacement with aluminum tubes.

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DISCUSSION OF PERTINENT RECORDS

Table I lists all of the tubes in the K reactors from which data were obtained for this study. A number of these tubes have been operated with anodized spacers since replacement and would not require cleaning if it were ever decided to remove the case layer from all of the tubes. Table II lists these tubes.

There were five known Zircaloy tube failures at the K reactors as listed in Table III.

The detailed results of findings from the 85 tubes listed in Table I are found in the referenced documents. The one production test (PT-204) which was still active at the time KE was shut down is reviewed briefly in the following pages.

STATUS OF PRODUCTION TEST-204 - ELECTROCHEMICAL HYDRIDE REMOVAL AND EVALUATION

This PTA provided for electrochemical cleaning of ten tubes in KE Reactor. Five were anodically stripped only and five received both the anodic strip and the secondary HF-HNO₃ electrochemical etch. This process is completely outlined in the authorizing document, DUN-6680.

The test was performed as authorized during the February 1970 outage and was reported by CH Gydesen in the form of an unclassified letter report dated March 9, 1970 (see references). Immediately following the test, two tubes, 2255-KE and 3056-KE, were removed for evaluation. The results of this evaluation are contained in BNWL-CC-2516. More recently a third tube from this test, 3058, was removed (see Table I) because of a leaking rear Van Stone flange. This tube had been given the two-step cleaning process similar to 3056. There was no evidence that the flange failure was in any way connected with the cleaning test since the flanges had been masked over by the stinger seal during the test. However, it is likely that failure was at least partly due to hydriding of the flange.

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The following tentative conclusions can be drawn from the work which was done.

1. The one-step anodic stripping of the case layer effectively removes the bulk of the hydride but leaves a thin (≈ 1 mil) layer which would still serve as a reservoir of hydrogen for later diffusion into the tube wall.
2. The two-step process, anodic strip plus electrochemical etch, completely removes all traces of the case layer and leaves a smooth, highly specular surface which will not rehydride when anodized spacers are used.
3. There were no observed deleterious effects from the cleaning operation. However, sufficient studies were not completed to assure this to be true over a longer time period. For example, the possibility of fluoride contamination and subsequent corrosion problems, was not fully evaluated.
4. It was concluded that it would be necessary to clean all of the flange surface which had been in contact with process water and possibly hydrided. This would probably require complete nozzle removal.
5. The time and cost for a full reactor cleaning was never firmly fixed, largely because the required disposition of the waste solutions was never fixed.
6. Cleaning after an extended outage would be significantly cheaper, both money and exposure wise, than during a short outage. For this reason, due consideration should be given to cleaning before startup of either of the K Reactors.

Seven tubes, of the ten cleaned, remain in the reactor and are available for further evaluation if, or when, KE is ever put back into operation. These tubes are 2251*, 2253*, 2257, 2259, 3052*, 3054, and 3161*. Those marked with an * were anodically stripped only. As mentioned above, the Van Stone flanges on these tubes were masked over and were not cleaned. It would be necessary to clean this area before they could be considered entirely clean.

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TABLE I. TUBE REMOVAL AND INSPECTION - K HYDRIDE PROGRAM

Number	Tube No.	Date Installed	Date Removed	Length of Service-Mo.	Type of Tube	Type of Spacers	Max. Total Hydrogen (ppm)	Max. Base Hydrogen (ppm)	Remarks or Reason for Removal
1	3065-KW	8/61	3/2/65	43	ALSC*	6063 Al	1210	125	Leaking front Van Stone-75% cold work
2	3075-KW	8/63	3/2/65	20	W**	" "	1380	53	Apparent leaker. Broke in two during removal. Location & cause of leak never determined.
3	3074-KW	2/63	3/29/65	25	W	" "	330	36	Removed to see if other tubes were hydrided.
4	0868-KE	2/64	3/29/65	12	W	" "	235	13	Removed to install autoclaved tube for tests.
5	2169-KE	5/63	4/2/65	22	W	" "	357	34	Removed to see if hydriding was common to both reactors.
6	4372-KE	1/64	4/13/65	15	W	" "	720	44	
7	3679-C	7/60	4/13/65	57	C-size	" "	215	---	Removed to see if hydriding was present in C Reactor test tubes.
8	3586-D	7/55	5/20/65	118	B-D-F-size Ribbed	" "	310	60	Oldest tube in existence
9	3075-KW-2***	3/2/65	7/9/65	4	W	" "	602	16	Check on hydriding in new tube
10	2557-KW	8/61	7/9/65	47	ALSC	" "	329	133	Oldest tube in K reactors.
11	3074-KW-2	3/29/65	10/20/69	7	W	" "	744	22	Check on buildup rate of new tube.
12	3667-KW	11/63	10/20/65	23	W	" "	209	54	Removed to accumulate additional data on distribution and buildup rate
13	3181-KE	10/63	10/20/65	24	W	" "	1162	91	
14	4372-KE-2	4/13/65	10/20/65	6	W-autoclaved	" "	65	---	The entire tube had been autoclaved. Hydriding seen in scratches.
15	2048-KW	8/63	11/4/65	27	W	" "	202	41	
16	3966-KW	11/63	12/22/65	25	W	" "	1200	76	Tube had damaged rear Van Stone - not leaking.

Table I (cont'd)

Number	Tube No.	Date Installed	Date Removed	Length of Service-Mo.	Type of Tube	Type of Spacers	Max.Total Hydrogen (ppm)	Max. Base Hydrogen (ppm)	Remarks or Reason for Removal
17	4453-KW	11/63	1/7/66	26	W	6063 Al	847	62	Failed rear Van Stone flange.
18	3074-KW-3	10/20/65	2/17/66	4	W	Stainless Steel	164	---	Highest H ₂ content near Van Stone.
19	3667-KW-2	10/20/65	2/17/66	4	W	Electroless Nickel plate on 6063	114	---	Highest H ₂ content found 14" from Van Stone.
20	4273-KE-3	10/20/65	2/15/66	4	W	6063 Al	296	---	Control tube for 3074 KW-3 and 3667 KW-2
21	3181-KE-2	10/20/65	5/2/66	6	W	Electroless Nickel Plate on 6063	12	---	Spacers plated by Battelle; high quality
22	2970-KW-2****	11/22/65	5/15/66	7	W	Anodized 6063 Al	12	---	Dummies anodized by Battelle
23	1554-KW	5/63	5/15/66	36	W	6063 Al	1400(est)	80	Analyzed after sandblasting.
24	5351-KE	1/64	5/24/66	28	W	6063 Al	----	39	(These tubes not removed from reactor. 4" samples removed from rear for analysis. Screw on flanges were used to replace Van Stone. Still in service at final shutdown.
25	5353-KE	1/64	5/24/66	28	W	" "	----	37	
26	5458-KE	1/64	5/24/66	28	W	" "	----	35	
27	3966-KW-2	12/22/65	6/7/66	6	W	8001 Al	190	20	Comparison of 8001 with 6063 Al.
28	2048-KW-2	11/4/65	6/15/66	7	W	Stainless	10	---	Further check on stainless.
29	4167-KE	10/63	8/29/66	34	W	6063 Al		50	Further test of screwed flange. Only 4" removed. Tube still in place.
30	1357-KW	5/63	9/10/66	40	W	6063 Al	995	93	Sandblasted. Analyses on masked area.
31	4372-KE-4	2/25/66	10/18/66	8	W-Grit Blasted before installed	Stainless	10	10	Evaluation of effect of grit blasted surface.
32	5651-KW	2/64	11/1/66	32	W	6063 Al	458	30	Tube damaged by graphite drill. Had operated at .5 power factor.

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TABLE I (cont'd)

Number	Tube No.	Date Installed	Date Removed	Length of Service-Mo.	Type of Tube	Type of Spacers	Max.Total Hydrogen (ppm)	Max. Base Hydrogen (ppm)	Remarks or Reason for Removal
33	3181-KE-3	5/2/66	11/30/66	6	W-Autoclaved	Ni-Plate & Autoclaved	10	10	
34	2970-KW-3	5/15/66	12/27/66	7	W	Carbon Steel	7	7	Alternate dummy study
35	2571-KE	5/63	1/11/67	44	W	6063 Al	510	74	(Base tubes for establishment of hydriding rates - to be compared later with tube charged with anodized. Further evaluation of alternate spacers.
36	2577-KE	5/63	1/11/67	44	W	6063 Al	425	78	
37	1554-KW-2	5/15/66	2/7/67	7	W	Stainless & Anodized	10	10	Base tubes for establishment of hydriding rates - to be compared later with tube charged with anodized.
38	1571-KW	5/63	2/7/67	44	W	6063 Al	1400	115	
39	1578-KW	5/63	2/7/67	44	W	" "	2480	112	
40	1766-KE	8/63	2/15/67	42	W	" "	510	97	
41	1773-KE	8/63	2/15/67	42	W	" "	750	98	
42	3966-KW-3	6/7/66	2/23/67	6	W	Reused Anodized	10	10	Evaluation of anodic coating deterioration.
43	3160-KW	8/63	3/17/67	43	W	6063 Al	827	95	(Base tubes for establishment of hydriding rates - to be compared later with tube charged with anodized.
44	3172-KW	8/63	3/17/67	43	W	" "	1227	106	
45	1357-KW-2	9/10/66	3/17/67	6	W	Reused Anodized	(DATA LOST)		
46	1960-KE	5/63	3/23/67	46	W	6063 Al	225	94	(Sandblasted on PT-052. Case layer not completely removed.
47	1972-KE	5/63	3/23/67	46	W	" "	184	90	
48	4372-KE-5	10/18/66	5/12/67	7	W	Reused Anodized	10	10	
49	2970-KW-4	12/27/66	7/24/67	7	W	Anodized	10	8	Test of front charged spacers.
50	2571-KE-2	1/11/67	7/27/67	6	W	Sanford Hardcoat	11	10	Test of proprietary anodizing process.
51	2577-KE-2	1/11/67	7/27/67	6	W	Low Resistivity Reused anodized	12	10	Test of anodic coating in bad condition.

TABLE I (cont'd)

Number	Tube No.	Date Installed	Date Removed	Length of Service-Mo.	Type of Tube	Type of Spacers	Max. Total Hydrogen (ppm)	Max. Base Hydrogen (ppm)	Remarks or Reason for Removal
52	3761-KW	11/63	10/6/67	47	W	40-Mo. 6063 7 Mo. Anodized	4560	175	Tube contained E metal rupture
53	3172-KW-2	3/17/67	10/25/67	7	W	Reused Anodized	18	12	The spacer rails had been scraped bare.
54	4372-KE-6	5/12/67	1/3/68	8	W	Nonsealed Anodized	9	8	Check on value of sealing anodic coat.
55	2461-KE	5/63	1/3/68	55	W	Anodized for last 12 mo.	613	111	The following 16 tubes (8 each) were to compare hydride content of tubes which had been operated with anodized spacers for 12-17 months and those which had used only 6063 Al for entire life.
56	2465-KE	5/63	1/3/68	55	W	6063 Al	410	94	
57	2479-KE	5/63	1/3/68	55	W	6063 Al	739	128	
58	2483-KE	5/63	1/2/68	55	W	12 Mo. with Anodized	611	105	
59	2479-KW	8/63	1/9/68	52	W	" " "	1050	111	
60	2473-KW	8/63	1/9/68	52	W	6063 Al	862	131	
61	2461-KW	8/63	1/9/68	52	W	6063 Al	574	104	
62	2462-KW	8/63	1/9/68	52	W	12 Mo. with Anodized	1500	99	
63	1667-KE	8/63	3/20/68	55	W	6063-Al	1188	152	
64	1673-KE	8/63	3/20/68	55	W	15 Mo. with Anodized	2185	142	
65	1761-KW	5/63	4/9/68	58	W	6063 Al	1510	143	
66	1773-KW	5/63	4/9/68	58	W	" "	1860	131	
67	1760-KW	5/63	4/9/68	58	W	15 Mo. with Anodized	795	132	
68	1774-KW	5/63	4/9/68	58	W	" "	1820	139	
69	1677-KE	8/63	5/29/68	57	W	6063 Al	550	112	
70	1672-KE	8/63	5/29/68	57	W	17 Mo. with Anodized	1178	134	

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TABLE I (cont'd)

Number	Tube No.	Date Installed	Date Removed	Length of Service-Mo.	Type of Tube	Type of Spacers	Max. Total Hydrogen (ppm)	Max. Base Hydrogen (ppm)	Remarks or Reason for Removal
71	3560-KW	11/63	7/68	56	W	18 Mo. with Anodized	DATA NOT AVAILABLE	DATA NOT AVAILABLE	Tube which sustained loss of coolant.
72	3460-KW	11/63	7/68	56	W	18 Mo. with Anodized	DATA NOT AVAILABLE	DATA NOT AVAILABLE	Damaged during 3560 removal
73	1960-KE-2	3/23/67	12/12/68	20	W	Anodized	10	10	{ These tubes were removed to see if after nearly two years with anodized spacers they were still clean.
74	1970-KE-2	3/23/67	12/12/68	20	W	Anodized	10	10	
75	1966-KE	5/63	12/10/68	67	W	6063 Anodized	Variable	120	{ These three tubes were operated for 18 months after sandblasting with anodized spacers. Where case layer removal was complete there was no further hydriding.
76	1968-KE	5/63	12/10/68	67	W	" "	"	140	
77	2163-KE	5/63	12/10/68	67	W	" "	"	130	
78	3661-KW	11/63	12/19/68	61	W	Last 24 Mo. with anodized	575	140	This tube had history of high outlet temperature because of EN load.
79	3074-KW-4	2/17/66	12/19/68	33	W-Grit blasted before installed	Ni/anodized	35	10	Records indicate short time with old nonanodized 6063 dummies between Ni & anodized operation.
80	1354-KW	5/63	5/20/69	72	W	6063/anodized	DATA NOT AVAILABLE	DATA NOT AVAILABLE	{ Metallurgical data indicate these tubes similar to 75, 76, & 77. They were operated 32 months with anodized after sandblasting.
81	1355-KW	5/63	5/20/69	72	W	" "	" "	"	
82	2255-KE	5/63	2/20/70	81	W	" "	279	116	Cleaned by anodic stripping only. Minimum remaining wall - 34 mils.
83	3056-KE	10/63	2/20/70	75	W	" "	147	142	Anodic strip plus electro-etch. Minimum remaining wall - 28 mils.
84	2069-KE	5/63	5/70	84	W	" "	----	118	Removed because of flange broken by impact.
85	3058-KE	10/63	9/70	83	W	" "		117	This tube had been chemically cleaned in February 1970. Removed because of flange failure. No apparent connection between failure & cleaning operation.

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TABLE I (cont'd)

LEGEND

- Prototype tubes made by Allegheny-Ludlum Steel Co.
- ** Production tubes made by Wolverine Tube Co.
- *** The designation 3075-KW-2 indicates this to be the second tube removed from Channel 3075-KW.
- **** The original tube removed from channel 2970-KW.

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TABLE II - TUBES NOT REQUIRING HYDRIDE REMOVAL

<u>KW Reactor</u>		<u>KE Reactor</u>	
1354	2462	3058	1677
1355	2461	2069	1673
3074	2473	2255	1667
3661	2479	3056	2483
3560	3172	1966	2479
3460	3761	1968	2465
1774	2970	2163	2461
1760	1357	1960	4372
1773	3160	1970	2577
1761	3966	1672	2571
1554		1972	1960

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TABLE III. HISTORY OF K ZIRCALOY TUBE FAILURES

<u>Failure No.</u>	<u>Tube No.</u>	<u>Date</u>	<u>Exposure</u> <u>Mo.</u>	<u>Hydrogen Content</u>		<u>Failure Cause</u>
				<u>Case</u>	<u>Base</u>	
1	3065-KW	3/2/65	43	1210	125	Prototype tube - 65% C.W. - Cracked front Van Stone.
2	3075-KW	3/2/65	20	1380	53	Tube would not hold pressure. Defect never located. Tube broke off during removal ≈8 feet from flange.
3	4453-KE	1/7/66	26	847	62	Cracked rear Van Stone flange. Believed to have been undetected defective flange.
4	2069-KE	5/70	84	ND*	118	Cracked rear Van Stone believed to have resulted from impact on nozzle by falling KER tube section
5	3058-KE	9/70	83	(light)	118	This tube was electrochemically cleaned in February 1970 (both anodic & etch). Failure was in form of cracked rear Van Stone. No evidence of failure having been caused by cleaning since failure was in masked area.

*ND = not determined

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