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ORNL
CENTRAL FILES NUMBER

CF 58-6-92

DATE: June 18, 1958
SUBJECT: Tabular Summary of Zircaloy-2 In-Pile
Rocking Autoclave Corrosion Data.
TO: E. G. Bohlmann
FROM: R. J. Davis

COPY NO. 126

ABSTRACT

A non-critical tabulation of Zircaloy-2 in-pile rocking autoclave data is presented. Exposure conditions, corrosion data, analytical results and references to other reports on the data are tabulated.

NOTICE

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This report is a tabular resume of the data from thirty seven in-pile, Zircaloy-2 rocking autoclave experiments performed since August of 1954 in an effort to understand the radiation corrosion of Zircaloy-2. A previous report describing the procedures has been written¹. The table presents the available data pertinent to the solutions used both before and after the reactor exposure, preirradiation treatment, the conditions during the exposure to irradiation, corrosion data from oxygen consumption and from specimen weight losses, and other data from specimens such as scale analyses. References to previous reports of the data are given. There are several instances of disagreement between calculated results listed here and those previously reported. These result from either revised calculations² or corrected errors.

The following paragraphs explain in some detail what may be found in the various columns in the table.

I. Design, Facility, Design Type.

The experiment number or designation is given. The experimental access hole in the LITR in which the experiment was exposed is listed. The HB-5 or HB-6 holes in the LITR are the two facilities used. The design type (I, II, or III) refers to the design of the autoclave used. These have been described in detail elsewhere.¹

II. Dates In and Out.

Under the second column are listed the dates on which the experiment was installed in the reactor facility and on which it was removed.

III. UO_2SO_4 Concentration.

The initial (synthetic) uranyl sulfate concentration, the final (analytical) concentration as indicated by a uranium determination on the irradiated solution and the isotopic enrichment are listed.

IV. CuSO_4 Concentration.

The initial (synthetic value) and final (analytical value from a copper determination) copper sulfate concentrations are listed.

V. H_2SO_4 Concentration.

The initial, as synthesized, sulfuric acid concentrations are listed. The second numbers, listed in parentheses, are calculated final sulfuric acid concentrations. From the total sulfate concentration (as shown by a sulfate determination on the irradiated solution) the analytical concentration of each metallic ion was subtracted to give an estimate of the final free acid concentration. Negative values for the final sulfuric acid concentration are listed in some cases. These simply signify that in those cases there were fewer equivalents of sulfate ion than of uranyl plus copper plus any other metallic ion.

VI. Additive Concentrations.

The initial and final concentrations and the formula of any additives are listed as well as the isotopic enrichment in the case of the Li_2SO_4 additive. In the case (Z-20) where D_2O was employed as solvent, that fact is indicated in column VI. In the case of D_2O solutions all concentrations are given in molar (rather than molal) units.

An unusual situation arose in the case of L6Z-114. About 6 times as much nitrogen was found in the irradiated solution (as ammonia) as was initially put into the solution (as nitric acid). The final analyses also showed excess sulfate which is reported in column V as H_2SO_4 . A reasonable interpretation is that the nitrate was nearly all converted to ammonia and the final solution contained $(\text{NH}_4)_2\text{SO}_4$ (~ 0.04) and NH_3 (~ 0.02).

VII. pH Values.

The pH's of the solution before and after irradiation are recorded here. In general the initial pH's were measured on a solution that was subsequently diluted by about 10% by the addition of 30% hydrogen peroxide. Subsequently, the peroxide was decomposed to water and oxygen. The solution in those experiments in which tank oxygen (column XXVIII) rather than peroxide was used did not undergo this dilution.

VIII. Length of Pretreatment.

This column gives the duration of the preirradiation corrosion treatment. First the number of hours during which the autoclave was subjected to high temperature water are listed. Note the letter "w" in parenthesis following this number indicates that that exposure was to water. Next the hours duration of the exposure to the fuel solution is listed.

IX. Pretreatment Temperature.

Column IX gives the temperatures of the pretreatment fluids, first the temperature of the water during the initial high temperature hydro-

static pressure test then the temperature of the fuel solution during the out-of-pile run are given..

X. Exposure Temperature.

The nominal temperatures during the exposure to irradiation are listed. In the case of several temperatures for one experiment, the order listed is the chronological order of exposure.

XI. The number of hours exposure at the indicated temperature (X) and thermal neutron flux (XII) are listed here. Short periods of reactor power level greater or less than the intended nominal operating level are accounted for on an equivalent energy basis. (One hour exposure with the reactor at 1.5 Mw during a period when the intended continuing power level was 3 Mw, would be counted as one-half hour exposure time). In most cases the nominal reactor (LITR) power level was 3 Mw but on special request a few experiments were exposed with the reactor at less than the nominal 3 Mw.

XII. Thermal Neutron Flux.

The average thermal neutron flux values (over the volume covered by specimens and over the total period of exposure) are listed here. In most cases these values were calculated from Zr^{95} activity in Zr-2 specimens. A few data from Zr^{95} in niobium containing alloys and from Cr^{51} activity in stainless steel specimens were used.

XIII. Solution Power Density.

These values (in w/ml) were calculated presuming that the initial uranium concentration continued throughout the run, that 200 Mev of energy are released per fission and that the nuclear cross section for U^{235} fission is 580 barns.

XIV. Average Radiolytic Gas Pressure.

During the course of each exposure, the increase in pressure each time the reactor went from zero to full power and the decrease on reactor power from full to zero, was noted on the pressure recorder. The linear average of all these values is listed. No correction for the affect of overheating of thermocouples by the γ flux during reactor operation was made.

XV. Initial Oxygen Pressure.

The partial pressures of oxygen at the exposure temperature at the beginning of each exposure are listed. The entries in parentheses are values calculated from pressure measurements made at 25° or 100°C rather than at the exposure temperature.

XVI. Final Oxygen Pressure.

The oxygen partial pressures at the end of each exposure are listed. Again these pressures correspond to the exposure temperature, and the values in parentheses were calculated from measurements made at 25° or 100°C.

XVII. Corrosion Rate from Oxygen Consumption.

These values are in general the slope of the least squares line through a plot of corrosion penetration vs. exposure time. As indicated, the corrosion penetration values in these cases were calculated from oxygen pressure measurements. In taking the least squares line through the data points the initial point was not included and the line was not forced to pass through the origin.

XVIII. Corrosion Penetration from Oxygen Consumption.

The difference in the oxygen partial pressures measured at a particular temperature before and after the entire exposure gives a measure of

the oxygen consumed and the depth of metal penetrated by corrosion. Many times measurements were made at different temperatures, thus providing more than one measure of penetration. The values listed in the table are penetration values from these pressure measurements (or averages when more than one value was available) to which were added the slight amount of corrosion estimated to have occurred during the pretreatment ($\ll$ 0.01 mils).

XIX. Specimen Material.

Here are listed the materials from which the specimens were made. The number of specimens of a given material is given in parenthesis following the material designation. In those cases where solid material was separated from the solution and analyzed an item termed precipitate is listed in this column. In one case (Z-12) the solution drained out of the autoclave during storage and only a solid material (powder from bomb) was analyzed, and a corresponding item is listed in column XIX to identify the sample analyzed.

XX. Weight Loss Corrosion Rates.

In those experiments where the entire exposure was made at one condition of temperature and thermal neutron flux it can reasonably be presumed that the corrosion occurred linearly during the exposure period, and the corrosion penetration from specimen weight losses divided by the exposure times yield the corrosion rate. Average values are given in the cases of two or more similar specimens.

XXI. Weight Loss Penetrations.

After the irradiation the specimens were treated to remove the corrosion scale. For Zircaloy-2 this treatment consisted of soaking .

in acetone, drying, cathodically defilming, drying and weighing. The weight loss before and after the experiment is presumed to be equal to the weight of metal corroded. Corrosion penetration can then readily be calculated. Average values from available specimens are reported.

XXII. Ratio Alloy/Zr-2.

As an index of the quality of the various metals and alloys tested, the ratios of the average corrosion penetration on the various metal or alloy specimens to that on the Zircaloy-2 specimens exposed in the same autoclave are reported here.

XXIII-XXVIII. Scale and Precipitate Analyses.

In most cases samples of scale (usually from specimens only) have been analyzed for uranium, copper, sulfate, zirconium and for any ion which may have been included in the solution as an additive. When a dry sample weight that was considered valid was obtained the analyses are reported as weight per cent of constituent. Otherwise the total weight of constituents are reported. Analyses of any solid material separated from the irradiated solution (precipitate) are reported similarly.

In several cases scale samples from specimens of different material were analyzed separately. Composite scale samples from more than one specimen material are so indicated by brackets following column XXII.

XXIX. Remarks.

Various items considered to be important and peculiar to particular experiments are noted here.

XXX. References.

Lists of references to each experiment in HRP Quar. Prog. Reports and in Central Files memos are given.

I	II	III	IV	V	VI	VII	VIII	IX	X	XI	XII						XIII		XIV		XV		XVI		XVII	XVIII	XIX		XX	XXI				XXII	XXIII	XXIV	XXV	XXVI	XXVII	XXVIII		XXIX																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																												
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											UO ₂ SO ₄ (m) (% U ²³⁵)	CuSO ₄ (m)	H ₂ SO ₄ (m)	Other (m)	pH	Time Exposed to Water and Solution (hr)	Temp. (°C)	Temp. (°C)	Time (hr)	Thermal Neutron Flux (n/cm ² /sec x 10 ⁻¹²)	Solution Power Density (w/ml)	Average Radiolytic Pressure (psi)	Oxygen Pressure Initial (psi)	Final (psi)			Oxygen Consumption Corr. Rate (mpy)	Oxygen Consumption Penetration (mils)		Specimen Material	Weight Loss Corrosion Rate (mpy)	Weight Loss Penetration (mils)								Corrosion Rate Ratio Alloy/Zr-2	(% U or total wt U)	(% Cu or total wt Cu)	(% SO ₄ -- or total wt SO ₄ --)	(% Zr or total wt Zr)	(% other or total wt)	ORNL Report No.	Page Nos.																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																							
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Designation Facility Design Type	Dates In and Out	Solution (Concentrations Before and After Exposure)				pH	Time Exposed to Water and Solution (hr)	Temp. (°C)	Temp. (°C)	Time (hr)	Exposure Conditions and Oxygen Consumption Data										Specimen Material	Weight Loss Corrosion Rate (mpy)	Specimen Data					Remarks	References	
		UO ₂ SO ₄ (m) (% U ²³⁵)	CuSO ₄ (m)	H ₂ SO ₄ (m)	Other (m)						Thermal Neutron Flux (n/cm ² /sec × 10 ⁻¹²)	Solution Power Density (w/ml)	Average Radiolytic Pressure (psi)	Oxygen Pressure Initial Final (psi) (psi)	Oxygen Consumption Corr. Rate (mpy)	Oxygen Consumption Penetration (mils)	Weight Loss Penetration (mils)	Corrosion Rate Ratio Alloy/Zr-2	(% U or total wt U)	(% Cu or total wt Cu)			(% SO ₄ ⁻⁻ or total wt SO ₄ ⁻⁻)	(% Zr or total wt Zr)	(% other or total wt)	ORNL Report No.	Page Nos.			
Z-20 HB-5 II	6 June 56 18 June 56	0.168M 0.156M 93.2	0.040M 0.031M	0.040M (0.023M)	Solvent D ₂ O	1.40 1.4	6 (w) 20	310 250	250 250	128.0 124.8	6.4 12.8	9.3 18.6	345 640	662 401	401 205	7.5 8.7	0.225	Zr-2 (6) Ppt.		0.208		6.25 400 y	0.47 281 y	0 0	4.81 281 y	Mo		2222 2148	107-111 98-104	
Z-21 HB-5 II	10 July 56 12 July 56	0.171 0.152 93.2	0.040 0.023	0.017 (0.017)	0.171 0.117 Li ₂ SO ₄ (Natural)	2.7 1.9	6.5 (w) 4	310 260	280	36.4	13.7	18.8	100	790				Zr-2 (6) Ppt.	10.0	0.042		105 y 100 y	0 0	0 0	1330 y 1180 y	0 0 Li		2222	107-111	
Z-22 HB-5 II	29 Jan. 57 5 Mar. 57	0.170 0.159 0.05	0.020 0.015	0.000 (0.000)		2.50 2.2	6 (w) 24	310 250	250 280	514.8 131.9	11.6 11.6	0.01 0.01		1223 1210	1119 1094	0.9 1.2	0.071	Zr-2 (6)		0.053		1.2 0.86	0 0		4.28	Spec. wt gain data used N ₂ excluded	2379 2331	115-121 120-123		
Z-23 HB-5 II	16 Jan. 57 17 Jan. 57	1.274 93.2	0.040	0.040		1.60	6 (w) 23	310 250	280	20.5	10.1	109	606	994	900	11.8	0.036	Zr-2 (6) Ppt.	20.5	0.048		16.7 780 y	1.32 17 y	9.46 1477 y	24.51 200 y		Irrad. soln. accidentally diluted N ₂ excluded	2331	120-123	
Z-24 HB-5 II	21 Aug. 56 25 Aug. 56	0.172 0.134 93.2	0.010 0.004	(-0.004)		2.35 2.4	7 (w) 24	310 250	280	81.1	10.3	14.0	288	642	158	29.2	0.260	Zr-2 (3) Zr-2 pickled (3) Ppt.	26.7 22.9	0.247 0.212	0.86	9.8 3800 y	2.57 383 y	0 0	31.8 750 y		N ₂ excluded	2222	107-111	
Z-25 HB-5 II	11 Sept. 56 20 Sept. 56	0.0091 0.0036 93.2		(-0.015) H ₂ CO ₃	0.129 0.064 Li ₂ CO ₃	9.5 9.0	7 (w) 20	310 250	260	133.7	11.9	0.91		1188	862	7.2	0.118	Zr-2 (6) Ppt.	5.6	0.086		3.3 170 y	0 0		72.9 500 y	0 0 Li	Tank O ₂ used Also press'd with 300 psi CO ₂ N ₂ excluded	2222	107-111	
Z-26 HB-5 II	2 Oct. 56 8 Oct. 56	0.173 0.163 93.2	0.040 0.023	0.040 (0.068)	0.170 0.113 MgSO ₄	1.50 1.40	6 (w) 17	310 250	250	109.2	11.2	16.4	305	643	398	9.3	0.108	Zr-2 (6)	9.7	0.115		2.9 0.96	1.66 0.54	0 2.70	68.7 42.93	0 Mg		2272	110-115	
Z-27 HB-5 II	30 Oct. 56 16 Nov. 56	0.044 0.026 93.2	0.005 0.001	0.031 (0.014)		1.45 1.7	9 (w) 19	330 300	250 280	119.9 64.2	12.3 12.3	4.5 4.3	360 120	1210 1030	933 700	8.5 16.2	0.376	Zr-2 (6) Ppt.		0.407		3.14 63 y	0.88 0	0 0	1.22 0		N ₂ excluded	2272	110-115	
L6Z-101 HB-6 II	9 Oct. 56 23 Oct. 56	0.172 0.144 93.2	0.040 0.027	0.040 (0.082)		1.1 2.4	6 (w) 17	310 250	250 280	128.3 120.2	5.9 5.9	8.7 8.2	265 20	636 437	421 28	5.1 10.3	0.237	Zr-2 (4) Zr, 20% Nb (2) Ppt.		0.255 0.197	0.77	66 y 840 y	0 681 y	0 0	4400 y 85 y		N ₂ excluded	2272	110-115	
L6Z-102 HB-6 II	13 Nov. 56 10 Jan. 57	0.169 0.148	0.040 0.014	0.018 (0.018)		2.50 3.5	6 (w) 16	310 250	250 280	350.5 733.2	6.6 6.6	1.2 1.2	30 5	1158 1025	921 346	2.0 2.3	0.307	Zr-2 (6) Ppt.		0.266		0.18 1270 y	10.18 853 y	0 708 y	43.33 2300 y		N ₂ excluded	2331	120-123	
L6Z-104 HB-6 II	4 June 57 17 June 57	0.173 0.142 93.2	0.031 0.014	0.031 (0.020)		1.30 2.3	8 (w) 19	310 250	250 280	20.5 256.3	7.3 7.3	8.5 8.0	240 61	1064 1158	1048 97	2.6 9.6	0.384	Zr-2 (2) Zr, 15% Nb, 1.5% Sn (2) 347 SS (2) Ppt.		0.334 0.172 0.227	0.51 0.68	1.09 1040 y	0.71 261 y	4.08 50 y	37.03 440 y		N ₂ excluded	2432	127-128	
L6Z-106 HB-6 II	7 May 57 19 May 57	0.171 0.166 93.2	0.031 0.020	0.060 (0.059)		1.05 1.60	6.5 (w) 19	310 250	280	180.5	7.5	8.1	38	1073	404	8.7	0.235	Zr-2 (2) Zr, 15% Nb, 1.5% Sn (2) 347 SS (2) Ppt.	4.4 4.9 7.8	0.090 0.102 0.160	1.13 1.78	2.20 309 y	0.66 48 y	2.05 173 y	47.60 370 y		Zr-2 specimens attacked, not completely descaled N ₂ excluded	2432	127-128	
L6Z-112 HB-6 II	9 July 57 26 July 57	0.171 0.127 93.2	0.030 0.014	0.060 (0.026)		1.05 2.90	6.5 (w) 22	310 250	280	279.4	8.2	8.9	70	869	82	5.9	0.260	Zr-2 (2) Zr, 15% Nb, 1.5% Sn (2) 347 SS (2) Ppt.	10.1 6.2 8.9	0.321 0.198 0.283	0.61 0.88	2.48 0.55 0.51	0 0 0	0 1.19 0.92	0 0 0		N ₂ excluded	2432	127-128	
L6Z-113 HB-6 II	30 July 57 16 Aug. 57	0.157 0.077 93.2	0.037 0.010	0.037 (0.024)		1.35 2.75	6 (w) 21	310 250	280	344.8	6.5	6.3	39	1082	157	6.5	0.269	Zr-2 (3) Nb (3) ZrO ₂ (2) Scale scraped from bomb Ppt.	7.5 2.5	0.296 0.099	0.33	700 y 25.88	80 y 16.02	107 y 0.94	7250 y 5.65		N ₂ excluded	2432	129	
L6Z-114 HB-6 II	20 Aug. 57 28 Aug. 57	0.168 0.112 93.2	0.020 0.016	0.036 (0.036)	0.098 (HNO ₃) 0.634 (NH ₃)	1.00 2.95	6 (w) 17	310 250	250 280	149.5 16.5	7.0 7.0	7.8 7.3	370 0	683 469	451 173	6.9	0.186	Zr-2 (2) Zr-2 pickled (2) Zr, 15% Nb, 1.5% Sn (2) Ppt.	9.4 12.6 17.1	0.178 0.239 0.324	1.34 1.82	3.78 150 y	0.22 29 y	0.36 727 y	19.05 450 y		Atm. N ₂ excluded			
L6Z-115 HB-6 II	17 Sept. 57 1 Oct. 57	0.042 0.044 93.2	0.007 0.011	0.020 (0.051)	0.198 0.204 Li ₂ SO ₄ 99.9% Li ⁷	1.20 1.40	7 (w) 22	310 250	280	278.3	4.9	1.6	70	463	44	4.3	0.149	Zr-2 (1) Zr-2 pickled (2) Zr, 15% Nb (2) Ppt.	4.1 3.7 2.7	0.130 0.118 0.085	0.91 0.65	0.14 1380 y	1.88 372 y	7.89 0	36.84 400 y	0 0 Li	1 Zr-2 pin broken N ₂ excluded			
L6Z-116 HB-6 II	15 Oct. 57 8 Nov. 57	0.174 0.114 93.2	0.041 0.019	0.041 (-0.004)	0.183 0.111 Li ₂ SO ₄ (Natural)	1.80 1.4	7 (w) 18	310 250	280 250	256.7 212.0	4.8 4.8	6.7 7.1	13 474	1161 457	494 156	5.6 4.5	0.291	Zr-2 (1) Zr, 15% Nb (2) Nb (2) Ppt.		0.282 0.322 0.292	1.14 1.04	2.74 0.3 24.81	0.47 1.39 3.60		84.68 10.2 6.02	0.12 0 0.18 Li	1 Zr-2 pin broken N ₂ excluded			

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2. K. S. Warren, "In-Reactor Autoclave Corrosion Studies. I Outline of Methods and Procedures. Supplement A. Refinements in Treatment of Oxygen Data". (In Press).

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