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ELK RIVER REACTOR
 OPERATIONS ANALYSIS PROGRAM
 FINAL REPORT - TASK 615
CORROSION SAMPLES AND TESTS
EVAPORATOR WATER BOXES

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FINAL REPORT TASK 615
CORROSION TEST
EVAPORATOR WATER BOX

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SUMMARY

Type 304 stainless steel coupons and "U-bend" test samples were exposed to the wet steam-water environment within the evaporator water boxes of the Elk River Reactor. This report describes the results of evaluation of the test probes after 22 months exposure. Small corrosion rates of as-rolled and annealed specimens were found. Some intergranular attack of the sensitized coupons showed up during this period. All "U" bend samples exhibited stress corrosion cracking.

1. OBJECTIVE

The purpose of this task is to obtain information on the corrosivity of the Elk River Reactor primary steam and water by periodic evaluation of corrosion samples inserted into the upper and lower evaporator water boxes of the Elk River Reactor.

2. INTRODUCTION

In April of 1964, the ERR-OAP project recommended that corrosion specimens be inserted into the evaporator water boxes of the Elk River Reactor. This recommendation was made based on the results of the analysis of radiolytic gas in the primary steam during operation of the reactor at full power in the non-vented condition. The Chicago Operations Office, USAEC, concurred in the adviseability of this task, and the specimens were subsequently prepared and installed early in June of 1964. The test, as designed, is qualitative in nature and was intended to determine if the conditions in the waterboxes are conducive to abnormally high corrosion rates. In addition, stressed "U-bend" samples were inserted to determine if conditions exist which are conducive to stress corrosion cracking. Examination of the specimens after exposure has shown that while the corrosion rates are not abnormal, there is evidence that conditions exist which are conducive to stress corrosion cracking.

The initial installation of specimens is described in detail in reference 1, which is the first report on the subject. The corrosion specimens were removed from the lower water box of evaporator No. 1 and visually examined in October 1964. The results of this examination are reported in reference 2. Since there was no evidence of deleterious effects, the test specimens were re-inserted for further exposure on the same day. The next removal for inspection occurred on August 1, 1965. At that time, evaporator No. 1 was opened and the specimens were removed for examination. Two coupons of each type (as-rolled, annealed, and sensitized) were taken from each fixture. The "U-bend" sample was removed from the lower water box fixture. The remaining specimens were re-installed for further exposure. The results of a laboratory examination of the removed specimens are given in reference 3. These results gave the first indication that conditions might exist which are conducive to stress corrosion cracking. On April 26 and 27, 1966, all remaining specimens were removed for a laboratory examination. This report describes the results of the final examination of specimens originally installed.

A set of replacement specimens were prepared for installation into the evaporator water boxes. The fabrication history of these coupons is described in detail in Appendix A to this report.

3. DESCRIPTION OF TEST

The Elk River Reactor is constructed with Type 304 stainless steel as the principal material exposed to the primary coolant. Four identical test fixtures were placed in the waterboxes of the evaporators in May and June of 1964. One fixture was placed in each of the two hand holes of each evaporator. The fixtures are therefore paired, two in the inlet and two in the outlet water boxes as shown in Table 1. The coupons inserted with each fixture are also shown in Table 1 as well as the locations of the fixtures. (Note that the coupon locations were transposed in ACNP-64588).

Each fixture supported the following test specimens:

- 1) A type 304 stainless steel "U-bend". The sample is formed by bending a 3/4-in. x 1/4-in. bar over a 2-in. diameter rod until the included angle is approximately 160°. The legs are then drawn parallel to one another when attaching the specimen to the fixture. This assures the presence of a full yield stress while the specimen is exposed to the test environment.
- 2) Nine type 304 stainless steel coupons having dimensions of 2-in. x 2-in. x 1/16-in thick. Three of the coupons were in the "as-rolled" condition, three in the "annealed" condition, and three in the "sensitized" condition. The coding of the coupons is shown in table 2.

TABLE 1

AS ASSEMBLED COUPON POSITION

Figure No.	1	2	3	4
Location	Inlet	Inlet	Outlet	Outlet
Evaporator No.	1	2	1	2
Coupons	U-Bend	U-Bend	U-Bend	U-Bend
	63*	66	53*	56
	43*	46**	13*	16
	33*	36**	3	6
	62*	65**	52*	55**
	42*	45	12*	15
	32*	35	2*	5
	61**	64	51**	54
	41**	44	11**	14**
	31**	34	1**	4

Coupons marked * were removed for examination August 1, 1965.
 Results were reported in Interim Report #2, September, 1965.
 Coupons marked ** were examined after removal of all remaining
 specimens in April, 1966.

The composition of the materials from which the corrosion samples were prepared is shown in table 3.

TABLE 2
CODING OF COUPONS

<u>Condition</u>	<u>Set 1 Coupon Nos.</u>	<u>Set 2 Coupon Nos.</u>
As Rolled	1 thru 7	31 thru 37
Annealed	11 thru 17	41 thru 47
Sensitized	51 thru 57	61 thru 67

TABLE 3
MATERIALS CHEMISTRY (w/o)

Type Sample	C	Mn	P	S	Si	Cr	Ni	Mo	Cu	Co
U-Bend	.062	1.78	0.023	0.011	0.37	18.30	9.03	0.36	0.16	--
Coupons	0.039	0.62	0.018	0.016	0.49	18.28	9.01	0.22	0.30	0.19

4. TEST CONDITIONS

The corrosion coupons and the stressed specimens were exposed to near saturated steam conditions of 936 psig and 539 F in the water boxes of the evaporators. The amount of moisture present in the incoming steam to the evaporators is of the order of 1/2%. The water solids carryover in the steam does not exceed one part per billion. The

outlet water boxes contain steam condensate with an atmosphere of steam and non-condensibles.

The steam leaving the reactor is sampled and analyzed routinely for oxygen and hydrogen (Ref. 4). The results of these analyses are shown in Table 4. The oxygen and hydrogen content of the steam in the water box are not measured on a routine basis, however, data taken during recent recombiner tests (Ref. 5) indicate that the concentrations are on the order of 1100 ppm oxygen and 130 ppm hydrogen. Some observers (Ref. 6, 7) have postulated that higher oxygen concentrations (greater than 5,000 ppm) may have occurred during the earlier days of reactor operation.

The chloride concentrations in the reactor water have been maintained at non-detectable levels, with an analytical technique sensitive to about 35 ppb, during the entire reactor operational period. The pH of the water has ranged from 6 to 8.

TABLE 4

RADIOLYTIC GAS CONCENTRATIONS IN REACTOR EFFLUENT STEAM

<u>DATE</u>		<u>OXYGEN</u>		<u>HYDROGEN</u>	
<u>Mo.</u>	<u>Year</u>	<u>Maximum</u>	<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>
Dec.	1964	131	32	---	---
Jan.	1965	64.4	29.3	---	---
Feb.	1965	33.4	---	---	---
Mar.	1965	45.4	12.44	3.55	1.1
Apr.	1965	168	11.6	4.74	1.1
May	1965	24.8	16.4	2.0	1.5
June	1965	66.9	13.9	3.3	1.53
July	1965	17.56	15.9	1.71	1.4
Sept.	1965	29.34	29.34	1.96	1.96
Oct.	1965	27.6	11.2	4.2	1.4
Nov.	1965	4.14	4.11	2.22	1.29
Dec.	1965	5.86	2.03	2.18	1.15
Jan.	1966	8.05	2.02	1.69	1.4
Feb.	1966	7.5	6.03	1.52	1.4
Mar.	1966	6.05	1.63	1.57	0.76

5. TEST PROCEDURE AND RESULTS

The fixtures were inserted May 27 and June 8, 1964 and were allowed to remain in place until August 1, 1965. At that time, Evaporator #1 was opened and Fixtures 1 and 3 were removed for examination. One "U" bend and 12 coupons (see Table 1) were removed for examination. Corrosion rates were small except for the sensitized specimens. Stress corrosion cracking of the "U" bend was observed. The results of the examination are reported in detail in Interim Report II of this task. The balance of the specimens were left on the fixtures and returned to the original locations for further exposure.

After a reactor shutdown in April, 1966, the balance of the coupons and "U" bends were removed from the evaporators and brought to Allis-Chalmers Greendale Laboratory for examination. In general, the appearance of the samples was the same as reported previously.

Immediately upon receipt, a gamma spectrum was obtained for the set of coupons from each evaporator. A change in the spectra has been noted from that observed in September, 1965. There is a small difference in the spectra between the upper and lower water boxes as shown in Fig. 1. A new peak at 1.7 Mev, possibly antimony 124, has appeared.

The individual coupons were descaled electrolytically and the amount of corrosion as well as thickness of desposit were measured. The results are given in Tables 5 and 6. With the exception of the sensitized coupons, the corrosion is small. The sensitized specimens differ from the others in the extreme difficulty in removal of the radioactivity by descaling. The reason for this can be seen in the intergranular attack observable in the metallographic sections shown in Fig. 2.

TABLE 5

RESULTS OF CORROSION TESTS AFTER 22 MONTHS EXPOSURE AT TEMPERATURE

<u>Coupon No.</u>	<u>Corrosion (mg/dm²)</u>	<u>Corrosion Product Film Thickness mg/dm²</u>	<u>Corrosion Rate (mg/dm²/mo) at 22 months</u>
1	12.4	15.3	0.70
5	cut up for examination		
11	15.5	17.25	0.80
14	13.6	13.1	0.60
31	45.6	25.3	1.15
36	6.8	10.98	0.50
41	15.1	35.10	1.60
46	9.2	13.24	0.60
51	134.8	125.10	5.70
55	113.4	152.9	6.95
61	875.2	598.24	27.20
65	232.4	296.6	12.25

TABLE 6

CORROSION TESTS
AVERAGE VALUES

	<u>Corrosion (mg/dm²)</u>	<u>Film Thickness (mg/dm²)</u>	<u>Normalized Activity 1.33 Mev Gamma</u>
As Rolled	21.6	17.19	1.0
Annealed	13.3	19.69	1.9
Sensitized	338.9	286.46	4.3

Examination of the "U" bends was as follows. A gamma spectrum was obtained from each "U" bend. The spectra did not differ significantly from that of the coupons. The "U" bends were then electrolytically descaled and sectioned parallel to the straight portion of the legs. All three "U" bends were found to have cracks. More cracks were present in the "U" bend from Evaporator #1 than from #2.

The appearance of several cracks are shown in Figures 3 through 7. The deepest cracks (12 mils) were found in the specimen removed from the top of Evaporator #1.

The results of corrosion tests of August 1965 and April 1966 are listed in table 7 for comparative evaluation. The corrosion rates are exceptionally low compared to the generally accepted corrosion rate for type 304 stainless steel which is about 10 mg/dm²/mo (0.06 mil/yr.). The corrosion rate is greatest during early exposure hours and slows down rapidly as the protective oxide is formed. Presumably, after the first 100 hours, the oxide coating retards corrosion and the weight does not change appreciably. This is evidenced by the decrease in corrosion rate values shown in table 7; i.e., when the constant film weight is divided by the increase in months of exposure. In general, the corrosion rate is greater in the inlet water box as compared to that of the outlet water box. This may be due to the fact that steam condensate enters the lower water box where a water or wet atmosphere is conducive to film formation and promotes the retardation of metal corrosion.

TABLE 7

RESULTS OF CORROSION TESTS AFTER EXPOSURE AT TEMPERATUREEVAPORATOR NO. 1
8 Months

Corrosion Rate (mils per year)

<u>Sample</u>	<u>Inlet Water Box</u>	<u>Outlet Water Box</u>
As Rolled	0.007	0.006
Annealed	0.015	0.008
Sensitized	0.225	0.080

EVAPORATOR NO. 1
22 Months

<u>Sample</u>	<u>Inlet Water Box</u>	<u>Outlet Water Box</u>
As Rolled	0.007	0.004
Annealed	0.010	0.005
Sensitized	0.166	0.035

EVAPORATOR NO. 2
22 Months

<u>Sample</u>	<u>Inlet Water Box</u>	<u>Outlet Water Box</u>
As Rolled	0.003	sample cut for inspection
Annealed	0.004	0.004
Sensitized	0.075	0.043

CONCLUSIONS AND RECOMMENDATIONS

A meeting was held on May 23, 1966 at the Chicago Operations Office, USAEC, Argonne, Illinois. In addition to representatives from the Commission, RCPA, NUS, and Allis-Chalmers, the attendance list included Mr. Robert Wylie of SwRI and Mr. Sutton, Mr. Balai and Mr. Stone of ANL who were present as metallurgical consultants. Since the results of this examination were discussed at the meeting, it is considered appropriate to report the conclusions and recommendations which resulted from these discussions. In the interest of accuracy, the following is quoted from the meeting summary prepared by the chairman, Mr. W. G. Albert:

"----Allis-Chalmers reported on the (three) stressed corrosion samples that had been removed from the water boxes in April of this year. These samples showed signs of stress corrosion cracking." "---Consultants agreed that no evidence was available to indicate that stress corrosion had ever, or could ever, result in catastrophic failure of piping. However, they also noted that regulatory people had not accepted this position. The conclusion was that the Elk River Reactor environment can lead to stress corrosion cracking since there are undoubtedly areas of high residual stress, high applied stress, or high oxygen concentration. The consultants recommend that an investigation of representative welds in the piping system be conducted prior to reactor startup. It was suggested that about 10 welds be selected for ultrasonic inspection.

If this inspection reveals any defects, the weld would then be radiographed and compared with the original radiographs made at the time of construction. Other questionable areas which could not be confirmed by radiograph would be tagged for routine ultrasonic surveillance. A more detailed and routine plan for piping inspection should be developed for future operation.---"

The general corrosion rates of stainless steel, type 304 exposed to the evaporator water box environment of the Elk River Reactor Plant were within the generally accepted value of 0.06 mil/year. Corrosion rates of the sensitized specimens were low enough to give assurance that the guaranteed tube life of twenty years would be exceeded in the absence of stress corrosion cracks.

REFERENCES

1. ACNP - 64588, Elk River Reactor Operations Analysis Program, Interim Report - Task 615, Corrosion Samples and Tests, Evaporator Water Boxes. July 1964.
2. SWRI - 1228-48 Consulting Report on the Inspection of 17-4 pH Mechanism Components, November 23, 1964.
3. ACNP - 65598, Elk River Reactor Operations Analysis Program, Interim Report II - Task 615, Corrosion Samples and Tests, Evaporator Water Boxes. October 1965.
4. Rural Cooperative Power Association - Elk River Reactor Monthly Operating Reports.
5. NUS - 263 Elk River Reactor Operations Safety Analysis Monthly Report #6, Sept. 1 to October 31, 1965.
6. GNEC - 388 Reactor Operations Safety Analysis Technical Report #9, Feb. 15 to March 15, 1964.
7. NUS - 264 Elk River Reactor Operations Safety Analysis Monthly Report #7, Nov. 1, 1965 to Jan. 31, 1966.

FIGURES

GAMMA SPECTRUM SHOWING DIFFERENCES BETWEEN UPPER AND LOWER WATER BOXES

FIG. 1

METALLOGRAPHIC SECTION OF SENSITIZED COUPON #65.
300X UPPER AS POLISHED, LOWER ETCHED. INTERGRANULAR
ATTACK HAS PENETRATED TO A DEPTH OF ABOUT 6.5 MILS

FIG . 2

TYPICAL CRACK IN SPECIMEN FROM EVAPORATOR #1 TOP.
400X

FIG. 3

GRAIN PATTERN OF SPECIMEN SHOWN IN FIG. 3 AT
CENTERLINE OF "U" BEND BAR. 100X

FIG. 4

ETCHING REVEALS TRANSGRANULAR AND BRANCHING NATURE OF THE CRACK. (TOP #1 EVAPORATOR, 100X ETCHED)

FIG. 5

TYPICAL CRACK FROM "U" BEND FROM BOTTOM OF EVAPORATOR #2. 400X

FIG. 6

CRACKS OBSERVED IN "U" BEND FROM TOP OF
EVAPORATOR #2. 400X

FIG. 7

A P P E N D I X A

APPENDIX A

DESCRIPTION OF REPLACEMENT SPECIMENS

On April 26 and 27, 1966 new specimens of slightly different design were attached to the fixtures and left with the RCPA personnel for installation into the evaporator water boxes. A documentation of the fabrication procedures and pretest examination of these specimens is given below.

REPLACEMENT "U" BENDS

The replacement "U" bends were prepared from 1/4 x 3/4 inch type 304 stainless steel bar in the annealed condition. Hardness testing of the bar showed it to be 80 R_B confirming the annealed condition. The bar was obtained from Central Steel and Wire Co. and certified to be Armco Heat No. 14024 with the following composition:

Cr - 18.57	Co - 0.22
Ni - 9.29	Cu - 0.18
Mn - 0.76	C - 0.062
Si - 0.64	P - 0.023
Mo - 0.26	S - 0.013

The procedure used for preparing the "U" bends was identical to that for the initial set of corrosion specimens as reported in ACNP-64588. The bar stock was cut to lengths, holes were drilled, and an initial bend was made to a 160° leg angle. At this point, the specimens were thoroughly cleaned of lubricant, etc. and all further handling was with clean white gloves. The "U" bends were then drawn down to the 180° condition, i.e., legs parallel, on the mounting bolt which is used for holding them in place in the evap-

orator. Dye penetrant tests and microscopic examination were performed and evidenced no cracking. The "U" bends were cleaned again, dried, and packaged in plastic bags for storage and transportation to the ERR.

CORROSION COUPONS

The coupons were prepared from a 20 gauge sheet of type 304 stainless steel in the annealed condition as confirmed by a hardness of 81 R_B.

The coupons were prepared by shearing the twenty gauge sheet into two-inch squares and filing the edges lightly to remove burrs. Center holes were bored and deburred in the next operation. A stress corrosion test cup was then formed using the jig pictured in Fig. A-1. The flat coupon was sandwiched between sheets of aluminum foil, which served as the drawing lubricant, and then mounted in a die held together with four bolts using 5 ft-lb. torque. A hardened steel ball of 0.394-in. radius was pressed into the coupon using a calculated force of 52,800 psi; a hydraulic pressure of 8,000 psi (see Fig. A-2) on a 1-in. plunger. The resultant hemispherical cup measured 0.394-in. I.R. x 0.168 in. deep. The cup forming operation thinned the bottom of the cup from the coupon thickness of 0.036-in. to 0.027-in. Fig. A-3 shows the front and reverse sides of a typical coupon.

The coupons were pickled lightly with cold 10 percent nitric acid for five minutes, to remove any adherent aluminum, and cleaned and dried to constant weight. The weights are tabulated in Table A-1. The coupon position as assembled on the holders is shown in Table A-2.

A test of the stress corrosion tendencies of the cups was performed in boiling 42% $MgCl_2$. Numerous cracks formed in one-half hour and became visible to the naked eye after four hours.

TABLE A-1

Pretest Corrosion Coupon Weight (gms)

1.	18.75706	17.	18.58850*	33.	18.54608
2.	18.76165	18.	18.74548	34.	18.75901
3.	18.93117	19.	18.62430	35.	18.79720
4.	18.66458	20.	18.43846	36.	18.74304
5.	18.43091	21.	18.55778*	37.	17.94300
6.	18.56679	22.	18.40725	38.	18.78421
7.	18.76599	23.	18.38599	39.	18.72878
8.	18.66114	24.	18.44346	40.	18.18029
9.	18.70908	25.	18.79501	41.	18.64827
10.	18.68706	26.	18.67370	42.	18.80260*
11.	18.74530*	27.	18.74738	43.	18.77676
12.	18.76283	28.	18.59885*	44.	18.69366
13.	18.77421	29.	18.33209	45.	18.61151
14.	18.65758	30.	18.77219	46.	18.69860
15.	18.80086	31.	18.65408	47.	18.76221
16.	18.72375	32.	18.78514*	48.	18.77170

* A TIG weld bead is present opposite the stress corrosion cup.

TABLE A-2

COUPON POSITION AS ASSEMBLED

<u>EVAP NO. 1</u> <u>Upper Water Box</u>	<u>EVAP NO. 1</u> <u>Lower Water Box</u>	<u>EVAP NO. 2</u> <u>Upper Water Box</u>	<u>EVAP NO. 2</u> <u>Lower Water Box</u>
U-Bend	U-Bend	U-Bend	U-Bend
1	29	19	20
2	28	18	10
3	27	39	11
4	26	30	12
5	25	31	13
6	24	32	14
7	23	33	15
8	22	36	16
9	21	37	17

JIG FOR FORMING STRESS CORROSION CUPS WITH A
FINISHED COUPON (280 F-O-3)

FIG. A-1

PRESS FOR MAKING HEMISPHERICAL STRESS CORROSION
CUPS (280-O-2)

FIG. A-2

REPRESENTATIVE COUPONS ARE SHOWN FRONT (ABOVE)
AND REVERSE SIDES (BELOW)

FIG. A-3

EXTERNAL DISTRIBUTION

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