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REACTORS—POWER

ARGONNE NATIONAL LABORATORY
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Lemont, Illinois

FACTORS WHICH AFFECT FORMATION AND DEPOSITION OF TRANSPORT CORROSION PRODUCTS IN HIGH-TEMPERATURE RECIRCULATING WATER LOOPS

by

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Reactor Engineering Division

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FACTORS WHICH AFFECT FORMATION AND DEPOSITION OF
TRANSPORT CORROSION PRODUCTS IN HIGH-TEMPERATURE
RECIRCULATING WATER LOOPS

by

C. Wohlberg and F. W. Kleimola

ABSTRACT

Deposits of corrosion products form on heat transfer surfaces and in radiation flux zones at temperatures around 500F in stainless steel systems operating with circulating water. The report considers the possible harmful effects of such deposits on heat transfer and fluid flow, as well as factors involved in the origin of these corrosion products and in the mechanisms of deposition. The prevention of deposition by chemical, mechanical, and electrostatic methods is discussed.

1. INTRODUCTION

An attempt has been made to gather information bearing on the sources and the mechanisms of deposition of transport corrosion products in stainless steel, high-temperature water systems. These products, chiefly magnetite (Fe_3O_4), deposit preferentially on surfaces in radiation and heat flux fields. This phenomenon has been generally noted in all high-temperature, recirculating water, stainless steel systems. Transport corrosion products and other transport products have been found on fuel elements in Pilot Channel tests at Oak Ridge, Hanford, and Chalk River. In the course of the heat transfer work conducted at Argonne, and subsequently in the Heat Transfer Projects at UCLA and MIT, data were obtained verifying these effects.

Some of the undesirable effects of the deposition of corrosion products on heat transfer surfaces are:

- (1) The heat transfer coefficients are lowered by the formation of an insulating layer on the primary heat exchange surfaces.
- (2) The increased pressure drop resulting from the roughening of the surfaces can affect the pump capacity. For example, it has been

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found that at water velocities of 25 fps, a thickness of deposited product of 14 to 20 microinches in a 1/4-inch tube may cause increases in pressure drop of 20 to 50 %.⁽¹⁾

Under the worst conditions, both effects may cause the burnout of fuel plates.

(3) The deposition of radioactive products throughout the circulating loop can hinder the servicing of equipment, such as pumps.

For a constant rate of heat removal, the heat transfer coefficient of the deposit will determine, for any given power level, the surface temperature of the zirconium-clad fuel plates. The higher the operating power level, the higher will be the metal surface temperature and hence the greater the corrosion rate. Consequently, the life of the reactor will be shortened. Some calculations have been made of the surface temperatures expected as a function of number of days operation at various power levels.^(2,3)

Estimates made on the reduction of the film heat transfer coefficient are given in Table I. A more detailed tabulation is shown in Appendix C, Table VIII.

Table I

THICKNESS OF DEPOSIT AND OVER-ALL HEAT TRANSFER COEFFICIENT (U_0) FOR VARIOUS VOLUME PERCENTAGES OF Fe_3O_4 IN DEPOSIT		
Fe_3O_4 in Deposit, % by Volume	Thickness of Deposit, mils	U_0 Btu/(hr)(sq ft)(F)
0.0	0.0	10,000
38.0	0.15	8,400
(Deposit has one-half the density of Fe_3O_4)		
25.0	0.23	7,450
10.0	0.57	4,800

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With a corrosion rate of $0.05 \text{ mg}/(\text{cm}^2)(\text{mo})$ for AISI type 347 stainless steel, there will be produced in 6 months of steady-state operation of the Mark I system (area about 6000 sq ft or $5.6 \times 10^6 \text{ cm}^2$) about 1700 gm of magnetite, all assumed to deposit on the fuel plates. If the total corrosion product, assumed to be 38 volume % magnetite (corresponding to one-half the density of magnetite, $\frac{5.18}{2} \text{ gm}/\text{cm}^3$), deposits on the total fuel plate area of 2400 sq ft ($2.25 \times 10^6 \text{ cm}^2$), the thickness of the layer will be 0.15 mil ($38 \times 10^{-5} \text{ cm}$). This will give a "scale" coefficient of $53,000 \text{ Btu}/(\text{hr})(\text{sq ft})(F)$. Since the Mark I film coefficient is about $10,000 \text{ Btu}/(\text{hr})(\text{sq ft})(F)$, the over-all coefficient of heat transfer will be about $8400 \text{ Btu}/(\text{hr})(\text{sq ft})(F)$. The 0.15-mil deposit therefore reduces the over-all coefficient by 16%. Westinghouse⁽²⁾ has estimated that the corrosion product is only 10 volume % magnetite. On this basis and with the assumed corrosion rate of $0.05 \text{ mg}/(\text{cm}^2)(\text{mo})$, the calculated thickness of deposit is 0.6 mil , and the over-all coefficient is $4800 \text{ Btu}/(\text{hr})(\text{sq ft})(F)$, a reduction of 52%. Comparison of the over-all coefficients calculated for 10, 25, and 38% by volume magnetite shows that the effect of the same total weight of magnetite varies greatly with the assumed magnetite percentages in the deposit.

The deposit will increase the relative roughness of the heat transfer surfaces. It is estimated that the coefficient of friction could be increased by about 30%.

A part of the transport corrosion product arises from steady-state corrosion of the system wall. At the present state of knowledge, the best method for limiting corrosion of stainless steel is the use of dissolved hydrogen in excess of about 50 ml of hydrogen per liter of water at standard temperature and pressure (STP). With such amounts of hydrogen present, the duration of reactor operation possible is estimated as 150 days at 100% power and about 320 days at 50% power.⁽²⁾ It is assumed here that tramp material does not contribute to the corrosion product formation.

Distribution of activity in the external system of an in-pile loop has been noted on several occasions. A discussion of this problem will be found under Section III-B, Argonne Pilot Channel at Hanford.

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II. SUMMARY

The following factors appear important in the formation and deposition of transport corrosion product, and in the prevention of these phenomena:

A. Formation

1. The following are the major sources of transport corrosion product:

- a. Dirt, such as metallic chips, originally present in loops. Chips oxidize, and the oxides flake off, becoming a transportable product.
- b. Mechanical introduction of particles, as from pump failures, etc.
- c. Oxides resulting from steady-state corrosion of the system.

2. The exact ratio between the transport corrosion product formed from debris and that formed by steady-state corrosion of system walls is not known for most of the systems mentioned in this report. A carefully assembled system which is freed of suspendable mechanical debris by filters during the early stages of operation should have only a minimum of suspended corrosion product present.

3. Water conditions, such as gas concentrations, resistivity, pH, and temperature, have a major influence on the amount of steady-state corrosion product formed.

4. The products are primarily Fe_3O_4 , with lesser amounts of Cr_2O_3 and NiO present. Any dirt, such as asbestos from packings, also appears in these products.

5. There is still considerable question as to the mechanism by which the transport corrosion oxides form. It may be that ions initially in solution are the precursors of transport corrosion products or that a thickening of surface oxide films is followed by subsequent flaking off; current evidence is in favor of the latter mechanism.

6. There is a related problem involving the transport of radioactive ions, rather than solid products, from surfaces in the flux zone to surfaces outside such zones. This activity becomes part of the adherent oxide layer.

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B. Deposition

1. Corrosion product deposition is markedly greater on areas of energy throughput, such as heat exchangers and on surfaces in radiation flux zones, than on surfaces outside such zones.
2. There seems to be a greater deposition on zirconium than on stainless steel in a radiation flux zone.
3. Radiation seems to be somewhat more effective than heat transfer as a precipitating force in the flux zone. There seems to be little difference between the amount of transport corrosion product caught on solid zirconium plates and that deposited on zirconium-clad fuel plates of varying percentage of U^{235} enrichment.
4. Radiation is known to have a coagulative effect on positively charged metal oxide particles.
5. Fine particles tend to migrate from rapidly flowing streams toward areas of low turbulence, such as bypasses. Because of their greater momentum larger particles tend to remain fluidized in rapidly flowing streams.
6. Some of the corrosion products found are magnetic, and their removal, to some extent, has been effected by magnets.
7. It appears that an electro-kinetic phenomenon may be responsible for the development of charges on the particles. Potentials can be developed between the particles and water, and between the pipe wall and water. Radiation and heat transfer upset the stable relation between these charges, causing deposition.

C. Prevention and Removal

1. The cleaning of loops by removing cutting oil from pipes and subassemblies results in the removal of most of the mechanical debris. Operation of assembled systems with suitable filters removes most of the material that may cause transport corrosion products to appear.
2. Mechanical traps, magnets, and charged plates all have some effectiveness in removing finely divided corrosion products from the flowing streams. All have limitations involving factors such as bulk, high pressure drop, and difficulty of sealing electrical leads into main flow. Most of them need extensive evaluation.
3. The presence of 50cc to 100cc hydrogen per liter of water at STP reduces the corrosion of the system appreciably.

4. From current data, it would seem that the monobed ion exchanger has a definite usefulness in removing from circulation the solubles that apparently dissolve from the components of a new system. Considerable clarification is still needed on the apparent ability of the ion exchanger to maintain (or to bring back to original value, depending on method of operation) the heat transfer coefficient in a system operating with transport corrosion product present. It would seem that, in these cases, the ion exchange bed may be acting as a mechanical filter.

5. Corrosion inhibitors, such as some dissolved substances and high pH water, have been tried. SiO_2 in crystalloidal solution and some other ions have shown some promise under static conditions, but have not been sufficiently evaluated. Water at pH 10 reduces deposition of transported products in irradiation fields.

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III. SOURCES OF TRANSPORTABLE PRODUCTS

There are 3 major sources of transportable products: (A) Initial dirt, (B) Mechanical failures (pumps, heaters, etc.), and (C) Steady-state corrosion.

A. Initial Dirt in Loops

Even though special care is usually taken to have all components of a loop kept clean during fabrication and assembly, appreciable amounts of drawing compound, core sand, turnings, metal chips, cutting oil, weld slag, asbestos, pipe-threading compounds, etc. may remain. Examples of dirt in various loops are cited to show that this initial dirt in loops is significant with respect to transport corrosion product formation.

1. Argonne Small Scale Heat Throughput Loop⁽⁴⁾

Deposit was still being collected on heat transfer tubes after one year's operation despite repeated "flashing off" and draining. Spectrographic analyses of the deposits collected on heat transfer tubes (see Appendix C, Table IX) show significant amounts of aluminum, magnesium, and silicon, which were probably introduced as sand and packing material, i.e., asbestos.

A 100-mesh filter placed in the main stream after the first year of operation collected stainless steel chips and turnings, asbestos fibers, sand, balls of copper and zirconium from burnouts, and welding slag. The chips and sand show conclusively that this material was in the loop from the start. The loop was operated for 40 hr between changes of screen. Six changes of screen were required to clean the loop; about a half-pint of dirt was collected. The type of material collected is shown in Fig. 1. After this effort, the deposits on heat transfer tubes were markedly minimized.

2. Argonne Large Scale Heat Throughput Loop

By the time this loop was ready for operation, it had become apparent that a new loop required a mechanical clean-up, that is, a means of filtering out the remaining dirt. Therefore, cone-shaped 100-mesh stainless steel filters were mounted in the pump inlet lines of this loop. The loop was then operated until a significant change was observed in the flow rate; at this time the filter was removed and cleaned. This was repeated until very little additional dirt was collected. The dirt consisted largely of metallic chips, sand, and miscellaneous debris, and amounted to approximately one pint. One metallic chip one-half inch in length was recovered. Analyses of the dirt are included in Appendix C, Table IX.

FIG. 1
DEBRIS RETAINED ON 100-MESH FILTER IN ARGONNE SMALL SCALE
HEAT THROUGHPUT LOOP AFTER OPERATION FOR ONE YEAR

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Following mechanical clean-up, very little deposit was collected on heat transfer surfaces during subsequent operation.

3. Argonne Corrosion Loop No. 2⁽⁵⁾

This loop was used for a simulated wear test of bearings in a Westinghouse 100A pump. Zirconium wear chips (2.2 gm) were inserted into the system. On completion of the ten-day test, the loop was disassembled and all components were scrubbed with acetone. Assorted solids totaling 4.4 gm were recovered. The loop was reassembled with a Neva-Clog filter in the main stream. After operation for one week, 0.15 gm of chips were found on the filter. This is an appreciable amount in view of the effort made to get quantitative cleaning in this test. The difficulty of complete removal of tramp material without a filter is illustrated by this test.

4. Westinghouse W Pump Loop

The results obtained by Westinghouse⁽⁶⁾ on their W pump loop give quantitative proof of the importance of dirt originally present in a loop. An analysis was made of the recirculated water after 7-day operating period, 4 days with an ion exchanger and 3 days without an ion exchanger. The water contained 7.6 ppm total solids of which 5.1 ppm were non-volatile solids and at least 4.3 ppm were suspended solids. Of the total solids 32.9 to 52.7 % was silica.

The test equipment during this period included an AISI type 316 stainless steel Micrometallic filter ahead of the ion exchange cells.

"The filter was in operation nearly 20 days throughout the clean-up and build-up periods, at a flowrate between 0.3 and 0.6 gpm and at 100F when it was first backwashed according to the procedure described above. An estimated 10 to 15 gm of sediment were removed. This was a surprisingly large quantity of material. Despite all this the main W loop still contained a large quantity of this sediment, for when the loop was drained and opened, sediment was found to have settled out all along the bottom of the piping.

"Part of the sediment expelled from the filter floated on the water, but the majority settled quickly. All of it was extremely black, and most of the heavy material was very magnetic with less than half of the lighter material being magnetic. The chemical analysis shows that 6.2% of the initial water backwashed sediment was carbon, while 5.5% of the HNO₃-backwashed sediment was carbon. This represents nearly 1 gm of carbon in the 10 to 15 gm of sediment."

Results of spectrographic analyses of the sediment back-washed from the filter and gravimetric analyses for carbon in the sediment are tabulated.

<u>Material</u>	<u>Per Cent</u>
C*	5.5 6.2 9.9
Fe ₂ O ₃	major
Cr ₂ O ₃	18.0
NiO	8.0
SiO ₂	0.25 - 0.7

*Gravimetric

There were lesser amounts of other oxides. The analysis of the regenerating acid used on the cation resin in this loop contrasts with this analysis. The spectrographic analysis of the acid, for the major components, is shown below.⁽⁷⁾ Thirty grams of dissolved solids as chlorides were obtained.

<u>Material</u>	<u>Per Cent</u>
Na ₂ O	62.00
CaO	30.00
K ₂ O	3.50
MgO	2.50
Al ₂ O ₃	1.00
Fe ₂ O ₃	0.50
SiO ₂	0.01
NiO	0.20
Cr ₂ O ₃	0.005

Two things notable in these runs, aside from the large amount of materials recovered, are: the high alkali and alkaline earth content, and the low iron, chromium and nickel content.

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5. Westinghouse D Loop

The loop is made mostly of forged pipe (rather than cast) and has about 180 sq ft (1.6×10^5 sq cm) of surface. The flow rate is 4000 gpm at 475F. Total time of operation was 1250 hr under substantially degassed conditions. On elution of the ion exchanger a total of 29.78 gm of "metals" was recovered. Chemical analysis showed the following:

<u>Element</u>	<u>Per Cent</u>
Mg	36.2
Ca	34.8
Fe	13.0
Al	4.6
Mn	3.8
Na	1.4
Si	5.5
Cu	0.32
Ni	0.27
Cr	0.013
Zr	0.40

The iron content is relatively low, yet the possibility exists that it was colloidal oxide rather than ionic iron that passed through the filters to the ion exchanger, since some corrosion product passed through one Micrometallic filter to be caught on the one in series with it.

A further significant fact derived from this run is that in successive periods, under degassed conditions, the rate of formation of solubles decreased as shown:

<u>Run</u>	<u>Rate of Formation of Solubles, mg/(cm²)(mo)</u>
1	0.25
2	0.222
3	0.206
4	0.188
5	0.162

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Since the amounts of alkaline earths, aluminum, manganese, sodium, and silicon are too high to result from corrosion alone and since all of these metal ions in the amounts found could not have come from make-up water, the following conclusions were drawn:⁽⁸⁾ "....it is evident that in the system studied the solubles were formed largely from extraneous materials, the most likely being welding slag, packing and gaskets. The decrease of the rate of formation of solubles with time is then to be expected and the solubles are seen to play only a minor role in the corrosion process."

6. Argonne Pilot Channel Loop at Oak Ridge^(9,10)

Numerous pump failures obscure the picture on initial dirt in the original loop. However, an analysis of the system water showed an unusual amount of heavy metals. This material was traced to pipe-bending compound used in one of the pipes. These heavy metals became quite radioactive and were distributed throughout the system.

There was a somewhat similar experience at start-up of the second loop used at Oak Ridge. After a 2-week test, the system contained large quantities of iron in the form of black iron oxide, along with copious quantities of grease. These materials were assumed to have resulted from machining and drilling operations and had not been removed prior to assembly.

7. Argonne Pilot Channel Loop at Hanford⁽¹¹⁾

Considerable effort was made to clean the loop prior to assembly and also during the run-in period. Following the initial tests at Argonne, on disassembly prior to shipment to Hanford, a considerable amount of chips and sand was present in the loop. Analysis of the sediment showed the principal components to be:

Carbon (by combustion)	1.7%
Iron (or other metal)	24.5%
Fe_3O_4	73.0%

Mechanical brushing, acetone rinsings, and water flushing were used to remove the loose material from the loop.

At Hanford, after a 2-week operation period (with in-pile tube by-passed), a leak developed in the top flange of the flowmeter. On removal of the flange, it was found that a large amount of black material had collected in the flowmeter well. At this time, stainless steel drill turnings were found at the check valve in the make-up water line.

On installation of the pressure tube in the pile, a 20-mesh screen was inserted in the main flow stream at the water inlet to the pressure tube. This screen caught black oxide and "fibrous-looking" metal chips, as shown in Fig. 2. This screen was cleaned after each test.

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FIG. 2

DEBRIS RETAINED ON A 20-MESH FILTER IN THE MAIN FLOW STREAM AT
INLET TO THE PRESSURE TUBE. ARGONNE PILOT CHANNEL AT HANFORD

Table II shows the amount of transport corrosion product found on various test sections run in this loop. It indicates that the longer the loop was run (and presumably the more initial dirt was removed), the less product was deposited.

8. Argonne-Chalk River Loop

After six months operation of this loop, one of the flow control valves in the main circulating stream, which was considerably more radioactive than other external components, was disassembled. The radioactivity was found to be due to stainless steel particles which were part of the debris (Fig. 3) removed from this valve.

B. Mechanically Introduced Impurities

Appreciable amounts of transport corrosion product may be formed by breakdown on pumps and subsequent introduction of wear particles produced by the friction of metal against metal. In general, these particles finally become magnetic oxide of iron, and nickel and chromium oxides.

1. Pump Failures

Generally in pump failures a great deal of metallic material and carbon from the bearings is thrown into the system, as indicated by the inky appearance of the water on draining.

Brief descriptions of the formation of debris following pump failures in various loops follow.

a. Argonne Pilot Channel Loop at Hanford

The first pump failure occurred during initial testing of the loop with the in-reactor tube bypassed. Stainless steel cuttings (turnings) were found in several places. Some large pieces were found wedged in the impeller passages. The remainder, small pieces, were found in the pump discharge chamber in the space between the nickel strip and the impeller end bearing housing and within the impeller end bearing housing. From this description and from the photographs of pump components after failure, Fig. 4, it may be seen that very large amounts of metal and carbon may have been introduced into the system.

After installation of the in-pile tube, a dummy test run with a dummy stainless steel cartridge was conducted for about three weeks. Analyses of transport corrosion products from this test run are given in Appendix C, Table X.

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Table II

DEPOSITION ON FUEL PLATES IN ARGONNE PILOT CHANNEL AT HANFORD

Medium: Degassed Water at 540F

Cartridge, ANL-140-	Plate Material	Loose Transport Corrosion Deposit,	
		mg/cm ²	mg/(cm ²)(mo)
2	Type 347	0.303	0.078
	U ²³⁵ core, Zr clad	2.109	0.541
	U ²³⁸ core, Zr clad	0.652	0.167
A	U ²³⁸ core, Zr clad	0.002	0.001
1	Type 347	0.690	0.105
	U ²³⁵ core, Zr clad	2.420	0.367
9	Zr-Sn Alloy	2.180	0.426
	Th core, Zr clad	1.470	0.289
11	Zr-Sn Alloy	1.940	0.381
	U ²³⁵ core, Zr-Sn clad	1.360	0.268
	Zr-Sn Alloy	0.035	0.007
C	U ²³⁸ core, Zr-Sn clad	0.022	0.004
	Type 347	0.105	0.048
	Zr-Sn Alloy	0.084	0.038
15	U ²³⁵ core, Zr-Sn clad	0.098	0.045
	Type 347	0.068	0.031
	Zr-Sn Alloy	0.017	0.008
E	U ²³⁸ Zr-Sn clad	0.008	0.004
	Type 347		0.006
	Zr		0.014
20	Type 347		0.000
	Zr		0.004
G	Type 347		0.000
	Zr		0.004

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FIG. 3
DEBRIS DEPOSITED AT INLET SIDE OF MAIN
CIRCULATING FLOW CONTROL VALVE. ARGONNE
LOOP AT CHALK RIVER. MAGNIFICATION X2.

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MOTOR HOUSING

ROTOR - IMPELLER END

THERMAL SPACER

GRAPHITAR
AND TITANIUM

FRONT BEARING HOUSING

FIG. 4

APPEARANCE OF DISASSEMBLED WESTINGHOUSE PUMP AFTER FAILURE

b. Argonne Pilot Channel Loop at Oak Ridge

Large amounts of transport corrosion products were coincidental with pump failures of a mechanical nature in this loop. The transport corrosion products deposited preferentially in low velocity areas and dead spots. Following one pump failure, graphite and metallic wear particles of nickel, Inconel, and stainless steel were found dispersed throughout the loop.

c. Argonne Corrosion Loop No. 2

In this loop, also, failure of pump bearings resulted in the introduction of particles into the system. The system was cleaned up, but subsequently about 0.12 gm of a dark brown solid was found in a static part of the loop.

Spectrographic analysis for the metals and chemical analysis for carbon showed:

C	2.6%
Fe	42.6%
Ni	7.3%
Cr	10.1%

which indicates for the metals alone the following percentage analysis:

Fe	71.0%
Ni	12.2%
Cr	16.8%

2. Abrading of Cartridges

During insertion and removal from test positions of spring-loaded test cartridges in the loops it has often been noticed that fine metallic chips and sometimes large score marks, are produced on these subassemblies. This abraded metal provided another source of transport corrosion product.

3. Miscellaneous Debris

Although metallic chips are a major mechanical source of transport corrosion products, non-metallic debris also gives rise to material which can deposit on heat transfer surfaces. Some of the earliest manifestations of the transport corrosion product problem were in the heat transfer study loops at MIT^(12,13) and at UCLA.⁽¹⁾ Analyses of the transport corrosion products deposited in electrically heated tubes in these 2 loops

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indicated that a major constituent was asbestos, which entered the system from sludge seals in the Hayward-Tyler pumps and from gaskets in the system. The data are included in the Appendix C, Table IX.

C. Steady-State Corrosion Product

1. General Problem and Theory

In many past experiences with loops, the effect of steady-state corrosion of the system on transport corrosion product formation was not appreciably noticed because it was obscured by the contribution from the various mechanical sources mentioned previously. However, the steady-state corrosion rate may be as high as $-0.05 \text{ mg}/(\text{cm}^2)(\text{mo})$ at oxygen concentrations of 0 to 2 ml/liter. Extrapolating this corrosion rate to a surface area approximating the STR, one would expect appreciable amounts of corrosion product to be formed.

In all of the work by the various collaborating laboratories, Fe_3O_4 has been found to be the major constituent of transport corrosion product; however, little data exist on the mechanisms by which the steady-state corrosion product finds its way from the system walls into the flowing stream. The following are 2 possible mechanisms:

a. The normally adherent oxide layer on the metal thickens slowly, until it reaches a sufficient thickness so that internal stresses in it are relieved by recrystallization. Since the specific volume of the oxide is greater than that of the metal from which it is formed, a breakup of the outermost layers of the oxide then occurs and some of the material is washed away.

b. Ferrous ions go into solution and, by one of several mechanisms, (14,15) are oxidized to Fe_3O_4 . However, in an experiment at Argonne, precipitated magnetite was injected into a loop at 550F with an oxygen content of 0.2 to 0.5 ml/liter. After 6 days, inspection showed the surfaces to be covered copiously with red oxide, which by X-ray diffraction was determined as $\alpha \text{ Fe}_2\text{O}_3$. The original material was poorly crystallized, and hence rather active Fe_3O_4 , which apparently gave rise to finely divided Fe_2O_3 . If a mechanism of solution and oxidation were correct for the formation of transport corrosion product, then the same type of red Fe_2O_3 would be the end product. This evidence indicates that mechanism (a) above is probably the correct one, since usually only well crystallized Fe_3O_4 is found.

2. Effect of Water Condition on Corrosion Product Formation

Water conditions, such as gas concentrations, resistivity, pH and temperature, have considerable influence on the amount of steady-state corrosion product formed.

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a. Gas Concentrations

(1) Hydrogen and Oxygen

Corrosion tests made in the external circuit of the Argonne Pilot Channel Loop at Oak Ridge⁽¹⁶⁾ showed that AISI type 347 stainless steel samples tested in 500F water containing 1000 and 1200 ml hydrogen per liter of water for 2 weeks retained substantially their original untested appearances with no significant weight changes.

Static autoclave tests at Battelle Memorial Institute showed that Type 347 stainless steel samples tested in 600F water containing 385 ml hydrogen per liter of water retained substantially their original untested appearances with no significant weight changes.

A large number of dynamic and static corrosion tests on an extensive group of alloys have been run at Babcock and Wilcox with a variety of gas concentrations in the water. Table III shows typical results on AISI type 347 stainless steel tested for 1500 and 2000 hr in 500F deionized water. The results may be summarized as follows:

Hydrogen concentrations over 50 ml H₂ per liter of water^(17,18,19) had a very definite effect in reducing the corrosion rates of the AISI type 300 series stainless steel to very low values. The corrosion rates were about 1 mg/(dm²)(mo) after 1500 to 2000 hr. The largest gain in weight (3 mg/(dm²)(mo)) was noted after 250 hr and 500 hr test; thereafter no changes occurred.^(18,20) The most significant criterion was appearance, since the actual weight changes were not appreciable.

A large number of other alloys, including some of the Monels, Inconels, and Stellites, also showed improved corrosion resistance in the presence of over 50 ml of hydrogen per liter of water. There is still some question about the stainless steels, AISI types 410 hardened and USS Stainless W, and Armco 17-4, precipitation hardened, in water containing dissolved hydrogen.⁽²¹⁾

Degassed water seemed to be somewhat more corrosive than water containing about 2 ml oxygen per liter and appeared to be particularly deleterious to Inconel.

Hydrogen did not cause a passivation of Type 300 series stainless steels persisting through tests with other water conditions.^(20, 21) Corrosion at 30 fps velocity was slightly greater than at one fpm velocity.⁽²²⁾

No substantial difference in corrosive attack was noticed for water initially containing 2.4 ml O₂ per liter and 110-180 ml H₂ per liter, as contrasted to water containing hydrogen only. The oxygen content dropped to negligible values in 3 to 12 hr under these conditions.

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Table III

CORROSION OF AISI TYPE 347 STAINLESS STEEL UNDER VARIOUS GAS CONCENTRATIONS
AT BABCOCK & WILCOX CO.

Temperature: 500F
Water Continuously Deionized

Gas Concentrations	Weight Change, mg/(dm ²)(mo)				Appearance
	Water Velocity, 30 fps		Water Velocity, 1 fpm		
	1500 hr	2000 hr	1500 hr	2000 hr	
Circuit #1	+ 1 (17)	---	-1 (17)	---	---
0.2 ml O ₂ /l	-40 (20)	---	-2 (20)	---	---
(0.01 - 0.5 ml O ₂ /l)	+ 1 (17)	---	-1 (21)	---	---
Circuit #1	+ 1 (20)	---	+2 (19)	0.0(22)	Pale blue to dark straw.
2 ml O ₂ /l	+ 2 (19)	---	---	---	Semi-static.
(0.5 - 3.4 ml O ₂ /l)	- 6 (22)	---	+3	---	Thin brown coating.
Circuit #2	+ 1 (18)	0.0(18)	0.0(20)	---	Almost metallic to glossy straw.
100-200 ml H ₂ /l	+ 1 (18)	+1 (22)	-1 (20)	---	
0.1 ml O ₂ /l	---	-1 (19)	---	---	
	---	-1 (19)	---	---	
Circuit #3	- 2 (19)	---	-2 (19)*	---	Glossy black.
25 ml H ₂ /l	- 2 (19)	---	-2 (19)*	---	
(24-40 ml/l)					
0.1 ml O ₂ /l					
Circuit #4	-23 (18)	-1 (22)	-3 (18)	11 (22)	
Degassed	-49 (20)	---	0.0(21)	---	
0.01 - 0.1 ml O ₂ /l	- 4 (21)	---	+4 (20)	---	
1.7 - 5.0 ml H ₂ /l	- 3 (20)	---	0.0(20)	---	
500 ml H ₂ /l	0.0(20)				Original metallic appearance.

*Welded.

The foregoing suggests that a relatively high hydrogen concentration should be effective in suppressing the formation of transport corrosion product. A free radical mechanism on a catalytic surface involving formation of free H radicals, with the latter acting as a "getter," would seem to be indicated. The fact that "degassed" water containing 0.01 to 0.10 ml oxygen per liter is the most corrosive environment would indicate that oxygen at this low concentration acts as a source of OH radicals which promote corrosion. At the higher concentration of about 2 ml oxygen per liter, oxygen acts as an anodic polarizer.

Tests by Battelle⁽²³⁾ indicate that hydrogen-saturated water has less corrosive action than degassed water, even on pure iron.

(2) Carbon Dioxide

The effect of CO₂ on the corrosion of stainless steels is not yet fully known. It is difficult to keep carbon out of a system if it is present in bearings. CO₂ may be present as a result of the oxidation of carbon by OH free radicals formed by the irradiation of water. It is expected that a monobed ion exchanger will remove CO₂ from the water; however, since a relatively small portion of the main flow goes to ion exchangers, some effects due to the presence of CO₂ are possible. It is known that CO₂ at appreciable concentrations will dissolve iron from stainless steels to form a clear solution. Under irradiation, the iron in solution will oxidize and deposit.

CO₂ may also rise from possible attrition and oxidation of ion exchange resins. M. Sivetz has noted appreciable losses in the capacity of ion beds due to such damage.⁽²⁴⁾ Further, CO₂ from the system water has been found to reduce appreciably the capacity of the anion resin.

Attention should be paid to the role of low concentrations of CO₂ in the formation of transport corrosion product. Possibly, the greater corrosion rates noted at Babcock and Wilcox in the presence of low concentrations of oxygen (0.25 ml oxygen per liter of water) are due to the presence of CO₂. CO₂ is known to be more aggressive toward mild steel in the presence of a low concentration of oxygen.⁽²⁵⁾

b. Temperature

Judging from the Babcock and Wilcox test results, temperature changes between 500F and 600F have little effect on the long-time corrosion rate.⁽²⁶⁾ The tests were made with a concentration of 30 ml of oxygen per liter; however, the effect may be different at low oxygen concentrations. Lower temperatures (440F to 470F) may show a markedly lower corrosion rate.

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c. High pH

The studies at Babcock and Wilcox in tests of 500, 1000, and 1500 hr duration at 500F with water containing 2 ml oxygen per liter of water, and with pH controlled at 9 to 10 by additions of NaOH, showed that more loose powder deposited at high pH than in a system with an ion exchanger. At the end of the first 500 hr the water from the loop operated with an ion exchanger was much clearer than the water from the loop operated at pH 10, and there was very little deposit. The analysis of the 2 waters, made after removing precipitates, showed 94 ppm of solids (alkali and carbonates) in the high pH loop and 12 ppm of solids in the loop with the ion exchanger. Alkalinity is difficult to maintain in the presence of oxidizing agents and, presumably, of carbonaceous material, unless the cation resin in a monobed ion exchanger is operated as an alkali exchanger. After the first 500 hr operation in the tests at Babcock and Wilcox, (22) losses of 0.30 and 0.10 mg/(cm²)(mo) were noted for Type 300 series stainless steels in high pH water, as compared to losses of 0.08 to 0.16 mg/(cm²)(mo) noted for similar specimens in deionized water.

It should be pointed out that the use of alkali may have undesirable effects. Alkaline solutions in the presence of oxygen or H₂O₂ are relatively good solvents for Cr₂O₃. This solvent or dispersive effect has been noted at as low a pH as 7.5. (27) Such action might destroy a passive film on a stainless steel surface. Weiser (28) cites some work which tends to show that in the presence of other oxides, such as those of nickel and iron, chromic oxide is rather easily dispersed by alkali.

In the presence of oxidizing free radicals resulting from the irradiation of water, there is definite danger of losing chromium from the stainless steel surface by solution. Up to 33 ppm of chromium have been found in the water from the alkaline irradiation in the Pitt cyclotron after 50 hr at 500F. (29,30)

The formation of "ferrites" is also a possibility. Wells (31) discusses the formation of the "defect" structures of oxides of iron. The arrangement of oxygen ions, cubic and close-packed, is the same in FeO, Fe₃O₄, and γ -Fe₂O₃. As some Fe⁺⁺ ions are removed from FeO and replaced with two-thirds their number of Fe⁺⁺⁺, to maintain electrical neutrality, an FeO structure defective in iron is obtained. "If this process be continued until we have only twenty-four iron ions in the volume containing thirty-two oxygen ions, we have Fe₃O₄. Further removal of iron gives γ -Fe₂O₃ with an average of 21-2/3 iron atoms in the same volume.The defect γ structures are stabilized by the presence of such ions as Li⁺, the compounds containing Li⁺ being intermediate between the trioxides and compounds such as LiAl₅O₈, which also has the spinel structure." A similar discussion is given by Huckel. (32)

The foregoing suggests that, as the iron in the surface of the metal is oxidized to FeO , it can also progress to a more highly oxidized state in the presence of Li^+ to form a normally unstable $\gamma\text{-Fe}_2\text{O}_3$ structure. Since knowledge of the steps in the oxidation of the stainless steels at elevated temperatures is sketchy, these possible troublesome mechanisms should be considered before deciding upon the use of Li^+ ion in a reactor.

In the operation of the Pilot Channel Loop at Oak Ridge at pH 10, using Li^+OH , it was difficult to maintain this pH, probably because of the continuous generation of CO_2 in the solution. When this run was discontinued, repeated efforts at removing the alkali from the loop by filling with water and nitric acid and by washing were unsuccessful. Operation with an ion exchanger resulted in effective removal of the lithium from the system, but it also apparently resulted in the production of very colloiddally "active" Fe_3O_4 , which quickly plugged up a throttling valve in the system. A "ferrite"⁽³²⁾ was probably the source of the strongly held lithium in the system and its decomposition resulted in the formation of the easily transported Fe_3O_4 .

d. Process Water

It has been suggested that the use of process water in the Hanford loop has been a contributor to transport corrosion product. However, samples exposed to process water only in the Hanford reactor for periods up to 9-1/2 months showed little deposit, which consisted mainly of calcite.⁽⁵⁴⁾ The corrosion products of stainless steel were present as 0.01 % or less. It may therefore be concluded that process water operation of the Hanford loop made negligible contribution to transport corrosion product formation in that loop.

3. Effect of Surface Conditions on Corrosion

a. Welds

It is well known that surfaces around welds, if not properly cleaned, give rise to rather loose corrosion products.^(5,34) A series of welded samples that had been cleaned in 2 % hydrofluoric acid - 15 % nitric acid (by volume) at 140F showed markedly lower corrosion rate, judging by appearance, than a group that had not had such treatment, as may be seen by an examination of Fig. 5. The uncleaned welded specimens showed dark areas attributed to oxides near the welds; furthermore, these areas spread for some distance from the weld and might be responsible for accelerated loss of oxides. The surfaces that had been cleaned remained light-colored and more uniform, even after prolonged corrosion testing under dynamic conditions. One set of cleaned samples, autoclaved for 937 hr and followed by 1728 hr of dynamic testing at 500F, showed very little change in appearance after the first 100 hr.⁽³⁵⁾

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POST-WELD CLEANING		
NO CLEANING	10% HNO ₃	15% HNO ₃ , 2% HF
<p>A</p>	<p>D</p>

<p>B</p>	<p>E</p>

<p>C</p>	<p>F</p>
<p>A - CORROSION TESTED B - CONTROL. NOT TESTED. C - CONTROL. NOT TESTED.</p>	<p>D - DEGREASED BEFORE WELDING E - NO DEGREASING BEFORE WELDING F - GREASED BEFORE WELDING</p>	

FIG. 5

EFFECT OF CHEMICAL CLEANING ON CORROSION BEHAVIOR OF WELDED STAINLESS STEEL AISI TYPE 347 AUTOCLAVED FOR 184 HR IN 500F OXYGENATED WATER (5cc O₂ PER LITER OF WATER)

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b. Surface Finish

In general, the better the finish of a metal surface, the better is the corrosion resistance. A smoothly machined surface presents the most stable surface, corrosion-wise.

c. Passivation

A Type 347 stainless steel surface that has been thoroughly cleaned of cutting oils, welding scale, and possible dirt inclusions is quite passive. However, it has been found that the presence of various thicknesses of oxides, as around weld deposits, has a definite local depassivating effect; for instance, magnetic oxide scales cause the formation of red rust during corrosion tests. In all likelihood, the bare metal is anodic to the extremely insoluble Fe_3O_4 and tends to corrode at relatively low temperatures during the heating-up process. Similar effects have been experienced in boiler practice.⁽³⁶⁾ A specific passivating treatment with 20 to 25% nitric acid is often specified as a follow-up after hydrofluoric acid - nitric acid pickling of stainless steels; this practice is unnecessary if the surfaces are thoroughly rinsed after pickling. This specification arises from industrial pickling practice where sulfuric acid - hydrochloric acid mixtures are used to pickle heavily scaled steel. The salts formed in the bath tend to build up, so that when the metal is washed basic chlorides may be precipitated on its surface. Nitric acid removes these potential sources of corrosion. In the case of hydrofluoric acid pickling, basic halides do not form.⁽³¹⁾

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IV. FACTORS WHICH MAY AFFECT CORROSION PRODUCT DEPOSITION

One of the most important factors contributing to corrosion product deposition is energy throughput at the surface involved. Two examples are: (A) Heat Transfer, and (B) Irradiation.

A. Heat Transfer

In the case of the MIT heat transfer loop, it was noticed that the transport corrosion product deposited with sharp demarcation in the heat transfer region of the electrically-heated nickel tubes that lay between the 2 copper electrodes. The thickness of the coatings increased from the entrance to the exit end of the tubes and ended abruptly at the exit end.

The voltage drop along the heated tube was 10 to 19 v. and the possibility of an "electrophoretic" deposition exists since direct current was used. The temperature of the walls may be appreciably higher, by 100 to 200F, than the bulk water temperature. The heat throughput ranged from 0.5×10^6 Btu/(hr)(sq ft) to 3.5×10^6 Btu/(hr)(sq ft).

A similar phenomenon was noticed in the UCLA heat transfer loops. Here again deposition occurred sharply in the heat transfer region. Direct current was used, and rates of heat transfer ranged from about 250,000 Btu/(hr)(sq ft) up to several million Btu/(hr)(sq ft).

It has been noticed in power house practice that the deposition of magnetic oxides in high-pressure boilers takes place on the heat exchanger section that is directly in the heated zone, and that there is a sharp demarcation line between the oxides deposited in the heated zone and in the unheated zone. In particular it has been noted that, in a small experimental boiler, deposition will be heaviest at the points where there is a sharp change in heat flux.⁽³⁷⁾

B. Irradiation

1. Argonne Pilot Channel at Hanford

The information on transport corrosion products on the various materials irradiated in the Argonne Pilot Channel at Hanford is given in some detail in Appendix A. Briefly, the results indicate that more transport corrosion products deposit on materials in a radiation zone than out of radiation, the materials being otherwise exposed to identical conditions.

Tests in this loop have also shown that the following effects exist in the radiation zone:

a. There was no substantial difference in the amounts of loose deposit as a function of U^{235} concentrations in fuel plate cores.

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b. Zirconium and titanium were consistently covered with heavier loose deposits than were the stainless steels.

c. Water velocities, between 3 and 25 fps, had little effect on deposition per unit area, if the amount of suspended material was small.

d. Geometry had an effect, as downstream plates contained more deposit than did the leading plates in the same cartridge.

Since the major portion of the data relating to radioactivity from ionic exchange has been obtained from studies of the Argonne Pilot Channel at Hanford, this phenomenon is discussed here.

Three pumps from the loop exhibited contamination. Material from the impeller of one of the pumps (Serial Number 5-S-6-APE-8) was analyzed as follows: "Radioactivity identification by counting and absorption techniques showed the primary activity of a hard black tarnish scraped from the pump impeller to be two betas (0.25 mev) and a hard gamma background, which is in good agreement with the reported values for Fe^{59} (betas of 0.26 and 0.46 mev and two hard gammas)." This analysis indicates that Fe^{59} was formed in the reactor pressure tube by neutron activation of Fe^{58} , transported through the system and deposited, rather than formed, on the pump.⁽³⁸⁾ The radioactivity was firmly bound in the adherent thin oxide layer on the metal; neither mechanical scrubbing, hot 15% nitric acid, nor hot citric acid solution had any solvent effect on it.

A similar phenomenon was noticed on some plates in Cartridge ANL-140-E in which a number of zirconium and Type 347 stainless steel plates were exposed to 540F water outside the radiation zone in the Pilot Channel Loop at Hanford for 66 days.⁽³⁹⁾ The plates were counted for activity at Argonne after removal of loose products. The Type 347 plates showed an activity of 1500 mr/hr at a distance of 3 in., and the zirconium plates showed only 150 mr/hr. The major sources of the activity were identified as Fe^{59} , Zr^{95} , and Co^{60} . Some months later the remaining activity was identified as Co^{60} from decay curves. Similar observations have been made on the Westinghouse-Chalk River Experiment where out-of-pile piping had picked up activity.⁽⁴⁰⁾

Apparently a true ionic exchange mechanism between metal ions in solution and those on the metal surfaces is involved. In this connection, J. E. Kemme has recently described a procedure by which a substantial reduction was attained in the activity of the piping in the Pilot Channel.⁽⁴¹⁾ Water containing 800 ml oxygen per liter, STP, was circulated through the loop at 465F for several hours. The significant fact was that no loose material was recovered on a "Neva-Clog" filter in the line, but an appreciable solution of active oxides occurred. The fresh metal of the filter had an activity of 300 mr/hr after completion of the cleaning procedure. This

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indicates that the exchange of radioactive ions from the solution with the metal surface may occur at that time in the history of the surface when most corrosion activity is taking place.

2. Westinghouse-Chalk River Test No. 1

A pair of fuel plate subassemblies were tested for a total of 3350 hr in the Westinghouse loop at Chalk River.

One of the subassemblies was in the flux zone while the other was outside the flux zone. The subassembly outside the flux zone seemed to be relatively clean, showing only what has been described as "normal" oxide deposition.⁽⁴²⁾ The subassembly in the flux zone collected copious amounts of transport corrosion product. Handling caused some of the oxide to shake loose and lodge at one end of the assembly; the quantity of corrosion product has been estimated as 25 gm. No experience like this has been noted in the Hanford tests; the amounts of transport corrosion products collected have never been enough to shake loose with ease from the plates.

There are differences in the analyses of Hanford transport corrosion products and Westinghouse-Chalk River transport corrosion products; the results of spectrographic and chemical analyses are given in appendix C, Tables X and XI. The most notable difference is the extremely small amount of chromium determined in the latter. X-ray diffraction shows that the material from the Chalk River test is Fe_3O_4 . Table IV shows ratios of iron to chromium, nickel to chromium, and iron to nickel for transport corrosion collected in the Hanford experiments contrasted to like ratios for the Westinghouse-Chalk River experiment. The ratios were obtained from the results of the chemical analyses only. The iron to chromium ratios for the transport corrosion products from the Westinghouse-Chalk River Test No. 1 vary from 200 to 112, while the transport materials from the Hanford tests have iron to chromium ratios varying from 84 to 7.1. Material caught in a static spot in the Chalk River heater shows an iron to chromium ratio of 6.6; it probably represents the analysis of some corroded metal debris. These differences between the compositions of Hanford transport corrosion products and the materials caught on the Chalk River assemblies are difficult to explain.

A brief recapitulation of the operational history of the Westinghouse River irradiation is found in the Appendix B; a comparison of the Mark I, Westinghouse-Chalk River, and Argonne-Hanford operating conditions is given in Table V.

The following differences in operation should be noted. The velocity in the Westinghouse-Chalk River Test No. 1 was 2.2 to 3.3 fps compared to about 10 fps in the actual Hanford operation. The Westinghouse loop was operated for only one week at Westinghouse at 500F without the ion exchanger, and for only 100 hr at Chalk River at full temperature in a dummy test with the ion exchanger prior to installation of the active fuel plate subassembly. During the dummy test the water was changed about 6 times. This

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Table IV

METAL RATIOS FOR TRANSPORT CORROSION PRODUCTS

	<u>Fe/Cr</u>	<u>Ni/Cr</u>	<u>Fe/Ni</u>
Type 347	3.7	0.5	7.3
ANL-140-2 A-1594 from fuel plates	45.0	3.2	14.1
ANL-140-2 A-1595 from SS plates	43.0	2.1	11.6
ANL-140-1; from fuel plates and SS plates	72.0	5.5	12.9
ANL-140-8; Subassembly A-3097	84.0	9.6	15.1
ANL-140-15; from SS and Zr plates A-3082	7.1	1.0	7.1
ANL-140-15; from fuel plates A-3089	12.7	3.1	4.1
ANL-140 material on screen A-3080	9.6	1.2	7.7
WAPD in-pile subassembly; Chalk River Test No. 1	112.0	16.5	6.7
WAPD out-of-pile control subassembly; Chalk River Test No. 1	200.0	49.0	4.1
WAPD filter backwash from ion exchanger bypass; Chalk River Test No. 1	145.0	17.6	8.2
WAPD loop heater, material caught in static spot; Chalk River Test No. 1	6.6	1.0	6.7
WAPD loop material ⁽²¹⁾ used by B & W	64.6	5.6	11.6
WAPD-D loop material ⁽⁸⁾ (Degassed Runs)	90.5 49.3	5.53 3.7	16.4 13.4
WAPD-D loop ⁽⁸⁾ (Hydrogenated Run)	19.3	2.15	9.0

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Table V

COMPARISON OF MARK I, WAPD-CHALK RIVER, AND
ARGONNE-HANFORD OPERATING CONDITIONS

	Mark I ⁽¹¹⁾	WAPD ⁽³⁾ Chalk River	Hanford ⁽¹¹⁾
Inlet Water Temperature, F	442	485, 490	540
Outlet Water Temperature, F	442-475	515, 510	540+
Temperature difference, plates to water, F	91 max.	11 at 17 gpm	15
Velocity in core, fps	3.6 (creep) 14.5 - 29 (normal)	2.2 - 3.2	10 - 15
Flow through core, gpm	2000 (creep) 8000-16000 (normal)	17-27	40
System volume, gal	3000	25	26
Flow through ion exchanger, gpm	10	0.1	0.2
Flow through filter, gpm	25 (net)	0.1	0.2
System pressure, psig	2050	1800	1550

means that a relatively fresh system was operated with deionization as compared to the Hanford loop, which operated for about a year prior to the operation of the ion exchanger. However, definite connection between the transport corrosion product deposition and the use of an ion exchanger cannot be proved as yet. Another point of difference is the higher neutron flux in the Chalk River test.

The individual and assembled parts of the Westinghouse-Chalk River loop were subjected to an elaborate cleaning procedure. The parts are reported to have been cleaned with a mixture of hydrofluoric acid and nitric acid and "passivated" with nitric acid prior to assembly. No screen or filter was placed in the main circulating stream at any time.

The authors have contended that chips and other dirt are a major cause of transport corrosion product deposition; however, these results would seem to indicate a pure corrosion pickup from a system that should be singularly passive because of vigorous cleaning. It is of course possible that some chips and other debris may have inadvertently entered the system during assembly.

3. University of Pittsburgh Cyclotron Work

The Westinghouse Atomic Power Division has set up a program for studying the mechanism of deposition of magnetite from water subjected to a deuteron beam from the Cyclotron at the University of Pittsburgh. The purpose was to find a more controllable method for studying the factors bearing on deposition in an irradiation field.

Two experiments were run in a static autoclave under the following conditions:⁽⁴³⁾

Time:	50 hr
Temperature:	600F
Deuterons:	1 to 3 mev
Beam current:	2.5 μ a
Targets:	1 plate of 97-1/2% Zr - 2.5% Sn Alloy

The gains in weight were:

On 97-1/2% Zr - 2.5% Sn	1 mg/cm ²
On Type 347	0.5 mg/cm ²

Examination showed a "deposit" only where the beam had struck. In the case of the zirconium, the gain in weight was due partly to an adherent layer of black ZrO₂ and partly to a layer of loosely deposited "iron oxide."

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In another experiment, a relatively large amount of magnetite from a Westinghouse loop was added to the water in the autoclave under similar conditions.⁽⁴⁰⁾ A very heavy deposit of magnetite formed on the irradiated areas; directly adjacent to these areas a layer of hematite mixed with magnetite was deposited.

Subsequently, a series of experiments were undertaken with a loop especially built to operate in conjunction with the cyclotron.

The conditions common to this group of experiments were:

Temperature:	500F
Pressure:	1500 psi
Irradiation time:	50 hr
Deuteron energy:	16 mev
Target:	0.009 in. thick Zr
Beam area:	About 1.9 cm ²

The specific conditions and results of the experiments were:

a. With non-degassed distilled water, there was obtained a preferential deposit on the target of 1.6 mg (per 1.9 cm²).⁽²⁹⁾

b. With non-degassed water maintained at pH 10 by LiOH additions, the change in weight of the target was 0.5 mg (per 1.9 cm²).

c. With non-degassed water, at pH 8.5 (from 7 to 9), there was a very slight deposit at one end of the target of transport corrosion product which contained more than 50% iron. The water was yellow, and contained 33 ppm Cr.⁽³⁰⁾

d. With water, partly degassed by boiling and maintained at pH 10 and flowing past some stainless steel chips, the deposit was again very slight and similar to that obtained in (c) above. It contained 12 to 24 ppm Cr in solution.

The conclusions to be drawn from this group of experiments are:

(1) The effect of irradiation as a prime precipitating factor for transport corrosion products is confirmed, since even in relatively rapidly flowing water the deposition was obtained on the target, and one can assume that the temperature of the target was not appreciably greater than that of the surrounding walls.

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(2) In water at pH 10, as contrasted to pH 7, less transport corrosion product precipitates in the irradiation zone, probably because of the "discharging" effect of the alkaline solution. This means that charges built up on the particles by the turbulent action of the water are discharged in solutions of relatively high conductivity; hence, the beam does not have a chance to discharge them preferentially in the vicinity of the target. Possibly all surfaces are given a high negative charge which prevents deposition in the beam.

(3) Considerable chromium is dissolved from the surface of the system by the alkaline water in the presence of air and ionizing radiations.

C. Water Velocity and Particle Size

The effects of water velocity and particle size are very much interrelated. Considerable evidence has been accumulated indicating that the fine particles collect in low flow areas and settle, while the larger particles remain fluidized in the rapidly flowing stream. Tests have also been run with test sections representing a series of velocities placed into the mainstream.

1. Settling of Fine Particles

Particles caught in a bypass filter in Westinghouse D loop were in the micron range with about one-half (44 to 52 %) of them 0 to 0.5 microns in size. (Appendix C. Table XI.)

Observations in the W pump loop at Westinghouse showed⁽⁶⁾ that the coarse material did not enter the low velocity pipe where the filter is located, but that most of the particles settled out in horizontal sections of the system after flow was stopped, indicating that during flow they were in active suspension. The transport corrosion product from Westinghouse-Chalk River Test showed that about 50 % of the particles were 3 microns or less in diameter (Appendix C, Table XII).

Such results have also been observed at Argonne in various loops where finely divided material and little else were caught in areas of low flow rate. For example, in Cartridge ANL-140-8⁽⁵⁵⁾ considerable deposition of transport corrosion product occurred on the outside of the cartridge, where flow rate was very low; the plates in the main flow showed relatively little deposition, judging by visual observation. In a number of cases where the pumps have failed electrically in the Hanford loop, examination has shown the presence of finely divided magnetite in the Inconel sleeve housing the rotor and even at the back end of the pump regions in which circulation is sluggish.

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At first glance, these results seem to be in contradiction with the theory utilized in hydraulic separation. However, they can be explained as follows:

The velocity of the water is sufficient to keep all the particles in active suspension. The fine particles, hence, are sustained by forces much greater than would be required to keep them in suspension in quiescent liquid. Therefore, particles of the micron range (0.2 to 5 microns) in dynamic systems will behave like particles of the colloidal range (0.005 to 0.2 microns) in quiescent systems. They will follow the water as it diffuses into regions of small flow. As the velocity decreases, the particles will settle out. The larger particles, because of their greater inertia (proportional to their area or d^2), will be swept past small openings, such as instrument ports, bypasses, etc. and will not have a chance to settle out.

2. Velocity Tests

A rough test was made to determine the critical velocity at which fine particles would deposit, as well as their particle sizes. A long, tapered section inserted into Argonne Corrosion Loop No. 2 showed deposition in a region calculated to have a fluid velocity of less than 10 to 12 fps. It should be stressed that these velocity limits are very approximate. Electron micrographs of the deposited particles indicate that most of the particles are definitely less than 0.5 micron in diameter and mainly of the order of 0.1 micron.

Tests were made to determine the effect of velocity on the deposition of transport corrosion product in the Large Scale Component Test Facility, without heat transfer or radiation.^(45, 46) The maximum deposition in the velocity sections took place at 18.5 fps rather than at the lowest velocity of 5.0 fps as was expected (see Figure 6). The amount of material deposited per unit area on the 5.0 fps and the 33 fps sections was about equal. However, the fact that the inserted synthetic transport corrosion product oxidized in the system may have influenced its deposition, since 2 competing processes may have been occurring. In one, at high turbulence, the high frequency of collisions with the walls and other particles may have helped to "coagulate" and deposit the particles, while in the other the higher velocity of the water may have had a scrubbing action on the walls. Hence, the intermediate velocity region rather than the lowest or highest velocity regions provided optimum conditions for deposition. The behavior of this material was certainly atypical.

In order to determine the effect of velocity on the deposition of transport corrosion products under pile irradiation, 2 cartridges, ANL-140-20 and 20G, were inserted in the Hanford Loop. The test plates were of Type 347 stainless steel and of arc melted crystal bar zirconium. Most of the plates were corrosion tested under dynamic conditions for 176 hr at 500 F before insertion into the test cartridges. The placement of the plates and the relative velocities are shown in Figures 7 and 8.

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FIG. 6
DEPOSITION OF IRON OXIDE ON VARIABLE
VELOCITY SECTIONS PER UNIT AREA

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FIG. 7
POSITION OF TEST PLATES IN VARIABLE
VELOCITY CARTRIDGE ANL-140-20

RE-7-12663-A

NUMBERS ENCLOSED IN PARENTHESES INDICATE
WEIGHT (mg/cm²) OF LOOSE DEPOSIT

FIG. 8
POSITION OF TEST PLATES IN VARIABLE
VELOCITY CARTRIDGE ANL-140-20G

Cartridge ANL-140-20 was in the flux zone while cartridge ANL-140-20G was outside the flux zone. Table VI shows the amounts of deposited transport corrosion product found on each plate. The transport corrosion product is determined by subtracting the weight of the specimen after vigorous brushing with acetone from the weight determined after removal from the cartridge. From this test it can be concluded:

- a. That in a clean system where the amount of suspended material is small, water velocity between 3 and 25 fps has little effect on the deposition per unit area. The Hanford loop has been gradually cleaned up (see Table II).
- b. That more material settles on surfaces in the radiation zone than in the external circuit.

D. Charge on Particles

In general, metallic oxide particles approaching colloidal sizes are positively charged. If so, they would discharge on system walls, which tend to be negative. In time, positively charged colloidal particles in water will coagulate merely by being forced rapidly through a narrow space in a metal tube.⁽⁴⁷⁾ The combination of mechanical action and the negative charge set up on the system walls by the liquid streaming past them is sufficient to cause coagulation. Positively charged particles, such as ferric oxide sols, will coagulate in an irradiation field of beta particles if the solution has been "sensitized" by the addition of a salt.⁽⁴⁸⁾ However, the need for sensitization is a matter of intensity of energy input; in a strong irradiation field positive particles should be discharged very readily. Ceric oxide sols are said to coagulate readily under similar conditions without sensitization. Negatively charged particles are more stable to mechanical agitation and also to radiation. Work has been done in imposing negative charges on hydrous oxide particles, thereby stabilizing them.⁽²⁷⁾ However, such methods probably cannot be applied when deionization is being practiced.

Data on the sign of the charge on Fe_3O_4 particles are scarce; however, W. S. Fagan⁽⁴⁹⁾ has recently summarized a series of experiments with both stainless steel and zirconium plates which would indicate that the charge on particles of impure Fe_3O_4 is negative. When parallel plates were oppositely charged with a potential of 1.3 volts between them, with the assembly being in the main circulating system of a loop operating at 450F, appreciable deposition took place on the positive plate. The negative plates were bright and clean, as were the series of stainless steel and zirconium plates that were electrically "floating." Wet chemical analysis of the deposit showed 58.2% copper and 5.4% iron. In another experiment, a simulated fuel plate subassembly was made negative, while 2 short assemblies were positively charged relative to the subassembly. Due to mechanical difficulties a rather short run was made, hence conclusive evaluation could not be made. The positive plates showed a slight deposit; the main section

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Table VI

EFFECT OF VELOCITY ON DEPOSITION ON TEST PLATES
IN THE ARGONNE PILOT CHANNEL AT HANFORD

Test Conditions

Media: Water containing ~ 0.2 ml O_2 /liter

Temp: 540F for 1360 hr

300F for 240 hr

Resistivity: 100,000 ohm-cm

No Deionization

<u>ANL No.</u>	<u>Material</u>	<u>Water Velocity, fps</u>	<u>Weight of Deposit, mg</u>	<u>Weight of Deposit per Unit Area, mg/cm²</u>	<u>Rate of Deposition, mg/(cm²)(mo)</u>
<u>Internal Cartridge (ANL-140-20)</u>					
3251	Zr	3	+4.4	+0.038	0.016
3255	Zr	3	5.5	0.047	0.020
3256	Zr	3	3.9	0.034	0.015
3248	Type 347	3	3.0	0.026	0.011
3252	Zr	12	3.2	0.028	0.012
3258	Zr	12	3.4	0.028	0.012
3260	Zr	12	3.4	0.029	0.012
3262	Zr	12	2.3	0.020	0.009
3245	Type 347	12	1.1	0.009	0.004
3247	Type 347	12	0.1	---	---
3246	Type 347	12	1.6	0.014	0.006
3261	Zr	25	3.1	0.027	0.012
3259	Zr	25	3.7	0.032	0.014
3249	Zr	25	6.3	0.054	0.023
<u>External Cartridge (ANL-140-20G)</u>					
3271	Zr	3	1.2	0.010	0.004
3268	Zr	3	1.7	0.015	0.006
3276	Zr	3	0.4	0.004	0.002
3264	Type 347	3	0.2	---	---
3275	Zr	12	0.4	0.004	0.002
3277	Zr	12	1.3	0.011	0.005
3278	Zr	12	0.7	0.006	0.003
3280	Zr	12	0.2	---	---
3266	Type 347	12	0.2	---	---
3265	Type 347	12	0.6	0.005	0.002
3263	Type 347	12	0.5	---	---
3267	Zr	25	1.0	0.009	0.004
3269	Zr	25	0.6	0.005	0.002
3279	Zr	25	0.8	0.007	0.003

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plates were clean. Dummy sections in each of these tests remained clean. Appreciable copper and lead were found in all deposits. The systems used had heavy-metal contamination which was difficult to remove. The particles deposited in these runs evidently had a negative charge.

E. Surface Material Composition

Two types of plates were exposed in the Hanford loop in 2 different cartridges: in cartridge ANL-140-2 for 118 days and in cartridge ANL-140-1 for 200 days; these were enriched uranium fuel plates with zirconium cladding and Type 347 stainless steel plates, in random placement. Some of the fuel plates had natural uranium cores and others had uranium cores with varying degrees of enrichment. The ratio of transport corrosion product collected per unit area on the fuel plates to that on the stainless steel plates was 3.5:1. This difference seems to be a surface phenomenon apparently due to differences of adherence of corrosion products to the oxide on zirconium and the oxide on stainless steel, since no change of deposition vs. degree of enrichment was detected. There seems to be little evidence of increased deposition due to longer exposure of cartridge ANL-140-1. Figure 9 shows views of fuel plates from cartridge ANL-140-1 after irradiation.

A fuel plate subassembly, with stainless steel box section riveted at the 2 ends, was tested as cartridge ANL-140-8. After 3 months at Hanford, only very slight corrosion product deposition occurred on the stainless steel, but the outer surfaces of the zirconium immediately adjacent showed deposition. Furthermore, the parts of the zirconium case that were not corrosion resistant seemed to have picked up an exceptionally thick layer of the magnetic oxide (Figure 10).

A variety of structural materials irradiated in cartridge ANL-140-13 included among others: A-nickel, K-monel, Inconel, Type 440A stainless steel, Westinghouse Grade I crystal bar zirconium, 3 sets of zirconium-tin alloys, and titanium. After 3 months of irradiation there was a sharp difference in the appearance of the zirconium-containing metals and of titanium from that of the other metals. The heterogeneous group was mostly bright and clean, while the zirconium-titanium group was covered thickly with transport corrosion product. Figure 11 shows a number of uncleaned specimens, including stainless steels, Stellite, Grade I crystal bar zirconium and titanium. It would seem that the group had a preferential pickup effect. However, this may be due to the greater corrodibility or "solubility" of the other materials, as compared to the zirconium group of metals, when operating with the ion exchanger, which maintains the water resistivity in the range of 1,000,000 to 2,000,000 ohm-cm.

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ZIRCONIUM CLAD, NATURAL URANIUM FUEL PLATE

BEFORE CLEANING

AFTER CLEANING

ZIRCONIUM CLAD, ENRICHED URANIUM FUEL PLATE

FIG. 9
POST-IRRADIATION APPEARANCE OF FUEL PLATES IRRADIATED FOR
200 DAYS IN FLUX ZONE IN ARGONNE PILOT CHANNEL AT HANFORD.
CARTRIDGE ANL-140-1

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SECTION (a) BEFORE CLEANING

SECTION (a) AFTER CLEANING

FIG. 10
POST-IRRADIATION APPEARANCE OF SMALL MARK I TYPE
SUBASSEMBLY AT VARIOUS STAGES OF EXAMINATION,
CARTRIDGE ANL-140-8

ARMCO 17-7-PH

TYPE 347

STELLITE 6

WAPD GRADE 1 CRYSTAL
BAR ZIRCONIUM

TITANIUM

FIG. 11
POST-IRRADIATION APPEARANCE BEFORE CLEANING OF STAINLESS
STEEL, STELLITE, ZIRCONIUM AND TITANIUM SAMPLES IRRADIATED
FOR 103 DAYS IN FLUX ZONE IN ARGONNE PILOT CHANNEL AT HANFORD.
CARTRIDGE ANL-140-13

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F. Water Conditions

1. Deionization

a. Babcock and Wilcox Tests

Perhaps the most notable effect of deionization has been on deposition observed in the Babcock and Wilcox Heat Transfer Loops, originally designed to study the effect of "scale" oxide on heat transfer in zirconium tubes.

The loops are described in detail by the Babcock and Wilcox Research and Development Department.^(50, 51) The essential parts of the loops are a heat transfer tube made of zirconium, with the dimensions 0.175 in. ID, 0.210 in. OD, and 12 in. length which is heated by water flowing in a tube annularly placed around it. Velocities of the water both inside and outside the tubes (counter-current) are regulated by bypasses around the test section. There are "ballast" sections of either zirconium or stainless steel designed to simulate the surface area ratio 1:3 of Zr to stainless steel in the STR. These loops were designed to measure relative changes in heat transfer rates rather than actual heat transfer coefficients.

Two tests, totaling 1350 hr on one set of 2 tubes and 2880 hr on a second set of 2 tubes, were run. An ion exchanger was used on one loop but not on the other. The heat flux density was 500,000 Btu/(hr)(sq ft). The heat flux density was 500,000 Btu/(hr)(sq ft). The temperatures of the water were 455F at the inlet and 475F at the outlet. The oxygen content was maintained at an average between 2 and 5 ml O₂ per liter of water (STP). No significant trends were noted in pressure drop or heat transfer rate even though magnetite-type deposits with thicknesses of the order of 0.001 to 0.003 in. were found on the tubes on sectioning. The water from the loop with no ion exchanger showed considerable amounts of suspended ZrO₂. The water from the loop with ion exchanger showed no ZrO₂ suspension, even though the zirconium sheet ballast, which was the source of the zirconium oxide, was equally corroded in both loops. The ion exchanger apparently acted as a filter.

Results are reported on 95 days of operation under conditions simulating those in tests at Chalk River.^(17, 20, 21) Actual conditions were: velocity, 2.2 fps; heat flux, 35,000 Btu/(hr)(sq ft); oxygen content, 0.02 to 0.08 ml O₂ per liter of water; temperatures, 440 to 475F; deionization and no deionization. "Corrosion product," obtained from WAPD, was added twice to each loop in 0.5 gm amounts during this period. No detectable change in heat transfer rate occurred in either system, and both had about the same coefficient.

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Results of 76 days of test on a set of zirconium tubes are reported.⁽⁵¹⁾ In this case conditions were: flow rate, 30 fps; average temperature, 460F; heat flux, 500,000 Btu/(hr)(sq ft). The oxygen concentration was 100 to 200 ml O₂ per liter of water with rather wide fluctuations above these levels. Type 347 stainless steel powder was added in one gram amounts to both systems. Where the ion exchanger was being used, there was no change in either pressure drop or heat transfer coefficient. However, where no ion exchanger was used there was an immediate decrease in heat transfer coefficient, and the coefficient leveled off in 10 days at about 60% of the original value. When the ion exchanger was turned on, bypassing 2% of the main flow, there was an immediate rise in heat transfer rate, and the rate leveled off at about the original value in 15 days.

In another system under similar conditions where the ion exchanger was used immediately, the heat transfer coefficient remained high until the ion exchanger was shut off, and then the heat transfer immediately decreased. Turning the ion exchanger on and off resulted in similar cycling up and down of the heat transfer coefficient. No increases in pressure drop were noted under these conditions.

This ability of a deionizer to "clean up" heat transfer surfaces is unexpected and can hardly be explained on the basis of an ionic pickup by the ion exchanger. One may postulate that the stainless steel powder oxidizes slowly and continuously, giving rise to fine corrosion product which settles on the heat transfer surface, reaching a certain equilibrium thickness which manages to maintain itself against continual scrubbing action of unoxidized metal powder in the water. If the ion exchanger is turned on, the freshly produced oxide, because of its very small particle size, is apparently caught by the ion exchanger. This is in accord with previous observations indicating that fine particles tend to migrate from the main stream into bypasses. Any deposited material is scrubbed off the heat transfer surface by the suspended metal powder and similarly removed by the ion exchanger. This mechanism seems plausible because of the change of heat transfer coefficient with operation or shutting-off of the ion exchanger. The greater mass of the metal particles permit them to be suspended in the main stream, while the smaller Fe₃O₄ particles go into the slower stream entering the ion exchanger.

The fact that the low velocity system discussed previously did not show any decrease in heat transfer coefficient may be explained by one of the following:

(1) The formed "corrosion product" inserted into the system does not deposit in the same fashion as the freshly formed oxide.

(2) The low rate of heat transfer, 35,000 Btu/(hr)(sq ft) as compared to 500,000 Btu/(hr)(sq ft), does not cause the material to deposit preferentially.

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(3) The low oxygen content of the system water, 0.02 to 0.08 ml O₂ per liter of water at 2 fps as compared to 100 to 400 ml O₂ per liter of water at 30 fps, has some bearing on deposition.

Whatever the correct explanation, the phenomenon needs further investigation. Examination of the ion exchanger in the high-velocity system should show whether the iron is adsorbed in ionic form or in the form of magnetite; that is, whether the ion exchanger is acting as a filter or is performing its true function.

b. MIT and UCLA Heat Transfer Tests

It was found during the course of heat transfer studies at MIT that the formation of silicate-containing deposits on the heat exchanger surfaces was eliminated when a monobed ion exchanger was installed on a bypass in the system. However, regeneration analysis of the resin indicated very little change in the capacity of the resin. No attempt has been made to determine the amount of undissolved solids in the bed, although it is probable that the bed acted more as a mechanical filter than as an ion exchanger. About one quarter of the total flow goes through the ion exchanger. Since the Hayward-Tyler pump used in the system was responsible for the origin of much of the asbestos material which deposited on the heat exchanger surfaces, it is possible that its replacement by a Westinghouse pump helped eliminate the difficulty.

The ion exchange resin bed at the UCLA Heat Transfer Project did not give conclusive results in preventing deposition of particles.⁽¹⁾ The direction of flow through the ion exchange column was upward instead of downward; hence the column was not operating at maximum efficiency and also could not act as an efficient mechanical filter.

2. pH

In the UCLA heat transfer work it was found that raising the pH to 10 prevented deposition of transported products on the heat transfer surfaces. However, the conditions were not entirely controlled, and there was some question as to the reasons for this effect. Considerable asbestos material was present in the loop and the alkali may have eliminated this.

More recently Westinghouse has found that, in the University of Pittsburgh Cyclotron loop, at pH 10 there was substantial reduction of the precipitation of black oxide on the surfaces of the radiation beam. The effect may be due either to putting a high negative charge on all surfaces, causing repulsion, or to the existence of sufficiently high water conductivity to result in the elimination of charges on particles and on system walls, thus avoiding preferential deposition on surfaces in the radiation field.

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V. METHODS OF PREVENTING TRANSPORT CORROSION PRODUCT BUILDUP

A. Cleaning the System

Fabrication should be as clean as possible. However, on the basis of experience with loops discussed in Section III-A, it is clear that the need for initial cleanup in a loop, during the run-in period, cannot be overemphasized. This is best accomplished by the use of filters and ion exchangers.

Chemical cleaning of components, such as degreasing and etching with hydrofluoric-nitric acid solutions, help in removing corrosion-promoting residues. However, in systems with field welds, such cleaning of assembled systems is usually not feasible.

B. Reduction of Corrosion Rate

1. The Use of Ionic Inhibitors in Solution

Work at Argonne has shown that a number of agents present in water, in low concentrations, seem to reduce appreciably the corrosion rate of Type 347 stainless steel and some other metals.^(39,40) Silicon dioxide, vanadium pentoxide, zinc oxide and others seem to be promising, as shown by short-time static tests at 500F in oxygenated water. The use of such agents may result in reducing the formation of loose transportable oxide. If any such agents are considered for use, the nuclear cross-section of the ions, their effective concentration, their tendency to precipitate on heated surfaces by dehydration, and their tendency to promote crevice corrosion effects on bearing surfaces, as well as other factors, will have to be evaluated critically.

2. Operation with Hydrogen in the Water

Work at Babcock and Wilcox, as discussed before, indicates that hydrogen at concentrations above about 50 ml H₂ per liter of water (STP) reduced the corrosion rates of most metals used in the STR. This appears to be a most promising method of reducing the amount of transport corrosion product formed by steady-state corrosion.

C. Methods for Preventing Deposition

1. Filters

Micrometallic filters have been rather satisfactory in the removal of the fine magnetite particles from the water. Scrap zirconium packed into a section with relatively low flow rate may act as a trickling filter which provides surfaces on which the transport corrosion product can coagulate.

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2. Low-Velocity Sections

Low-velocity sections act as settling chambers for fluid-borne particles. Although the efficiency of such sections has not been quantitatively evaluated, they do have the advantages of low pressure drop and easy cleaning.

3. Magnets

Both permanent magnets and electromagnets have shown ability to catch ⁽¹⁶⁾ transport corrosion product even in active flow areas. Furthermore, cold-worked stainless steels become magnetic enough so that chips may also be caught. For example, an Alnico V permanent magnet canned in Type 347 stainless steel was installed for several runs in the main flow of the Argonne Pilot Channel at Oak Ridge. Each time substantial amounts of solids were collected.

4. Ion Exchangers

The effectiveness of ion exchangers has sometimes been overshadowed by the fact that they were also acting as filters. They contribute to reducing the deposition problem in the following ways:

- a. Reducing soluble impurities which might become radioactive and create a radiation hazard.
- b. Removing impurities which might retard the recombination of oxygen and hydrogen.
- c. Minimizing corrosion by maintaining proper water conductivity.
- d. Retaining charged fine particles by electrostatic forces on ion exchange resins.⁽⁵³⁾

5. Charged Plates

Electrically charged plates of suitable sign have been shown to have some effectiveness in catching fine particles in a flowing stream.⁽⁴⁸⁾

APPENDIX A

RECAPITULATION OF TESTS IN ARGONNE PILOT CHANNEL AT HANFORD

A description of the Pilot Channel Loop at Hanford is presented in another report.⁽¹¹⁾ A variety of materials were tested during various periods of time, referred to as test runs. The materials tested were mounted in stainless steel holders called cartridges. Cartridges designated by numbers were positioned in the reactor flux; duplicate cartridges were simultaneously exposed under the same water conditions, but outside the flux zone, as controls. Control cartridges were designated by capital letters.

Particular emphasis has been placed on cartridges that contained materials fabricated into plate form. Test data on these materials have been condensed in Table VII. Discussion of these plate cartridges follows:

1. Cartridge ANL-140-2: This cartridge contained 16 fuel plates and 12 Type 347 stainless steel plates. The cores of the fuel plates contained varying percentages of U^{235} alloyed with zirconium; the cores were clad with zirconium. Crystal bar zirconium was used for the core and clad.

Post-irradiation examination of the cartridge showed a thin film of black powder on its surface. This powder was not firmly attached and rubbed off easily in handling the cartridge with the manipulators. The cartridge surface under the film of powder had a dark lustrous appearance resembling the oxide film frequently found on stainless steel which has been exposed to high-temperature water. The stainless steel and the fuel plates were covered with a very black, dull powder of appreciable thickness. Some of the powder, found to be substantially Fe_3O_4 , fell off during handling.

There was no substantial difference in the amount of loose deposit on plates of different concentrations of U^{235} in the cores of the plates. In fact, fuel plate A-561, with a U^{238} core, picked up more deposit (1.96 mg/cm^2) than the average for the 2-in. length enriched fuel plates (1.04 mg/cm^2). The zirconium clad fuel plates had more loose deposit per unit area than the stainless steel plates.

Type of Plate	Length of Plate, In.	Average Deposit mg/cm^2
Fuel	6	2.10
Fuel	2	1.04
Stainless Steel	6	0.24
Stainless Steel	2	0.49

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The relative positions of the plates in the cartridge are shown in Figure 12. No definite statement can be made on the effect of the geometry of placement on transport corrosion deposition from a study of this figure.

2. Cartridge ANL-140-A: This cartridge contained one fuel plate, A-568, similar to those in Cartridge ANL-140-2. The core of this control plate contained natural uranium. On post-test examination, the surface of the cartridge was a shiny blue-black. There were no indications of transport corrosion product deposition. The control plate lost one milligram as a result of cleaning.⁽³⁸⁾ It may be seen that no appreciable transport corrosion product deposition took place on this control material compared to similar plates in Cartridge ANL-140-2 exposed to reactor flux.

3. Cartridge ANL-140-1: This cartridge contained 24 fuel plates and 12 Type 347 stainless steel plates. The fuel plates were similar to those in Cartridge ANL-140-2.

On post-irradiation examination, the cartridge was found covered with a black deposit. The plates were carefully removed from the cartridge and placed in a plastic container in such a way as to preserve their identity with respect to their position in the cartridge.

The same observations as were made on cartridge ANL-140-2 in regard to the deposit on the test plates can be made for this cartridge. The concentration of U^{235} in the cores of the fuel plates did not have any marked effect on the amount of loose deposit. The fuel plates had more loose deposit per unit area than the stainless steel plates. Figure 13 shows the appearance of 2 plates before and after cleaning. The stainless steel plates show a considerably thinner deposit of transported product than do the zirconium plates. The thickness of the deposited material on the uncleaned zirconium plate can be judged by observing the area at the lower left of the uncleaned zirconium plate where the deposit had flaked off. As a quantitative indication of the relative weights of deposited material, 531 mg of deposit were removed from the zirconium plate whereas only 68 mg were removed from the stainless steel plate.

The relative position of the plates in the cartridge are shown in Figure 14. As observed for Cartridge ANL-140-2, no definite statement can be made on the effect of geometry on transport corrosion deposition from a study of this figure. No definite conclusion can be made as to the effect of time on the amount of loose deposit. Cartridge ANL-140-1 was exposed to irradiation at 540F for a longer period of time (200 days) than Cartridge ANL-140-2 (118 days); the former has more loose deposit as shown by the weight changes. However, the plates in Cartridge ANL-140-2 were scuffed in handling, and an appreciable amount of deposit was lost before weights were taken; therefore, the amount of loose deposits may have been approximately the same for the 2 cartridges. From this it appears that the limiting thickness of loose deposit may well have been attained for the 2 cartridges for these particular test conditions.

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NUMBERS ENCLOSED IN PARENTHESES INDICATE WEIGHT (mg/cm²) OF LOOSE DEPOSIT

"A" NOS. REFER TO FUEL PLATES

* TYPE 347 STAINLESS STEEL PLATES

BLANK SPACES CONTAINED STAINLESS STEEL PLATES WHICH WERE NOT WEIGHTED

FIG. 12
POSITION OF PLATES VS DEPOSITION.
CARTRIDGE ANL-140-2

RE-7-12666-A

BEFORE CLEANING

AFTER CLEANING

ZIRCONIUM CLAD, ENRICHED URANIUM PLATE

BEFORE CLEANING

AFTER CLEANING

STAINLESS STEEL AISI TYPE 304

FIG. 13
POST-IRRADIATION APPEARANCE OF ZIRCONIUM-ENRICHED URANIUM
AND STAINLESS STEEL AISI TYPE 304 PLATES IRRADIATED FOR
200 DAYS IN ARGONNE PILOT CHANNEL AT HANFORD.
CARTRIDGE ANL-140-1

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F.W.KLEIMOLA:F.S.,3-22-54

NUMBERS ENCLOSED IN PARENTHESES INDICATE WEIGHT (mg/cm²) OF LOOSE DEPOSIT

*A# NOS. REFER TO FUEL PLATES

* TYPE 347 STAINLESS STEEL PLATES

FIG. 14
POSITION OF PLATES VS DEPOSITION.
CARTRIDGE ANL-140-1

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4. Cartridge ANL-140-C: This cartridge contained one plate with a core containing 10% natural uranium 90% zirconium alloy clad with zirconium-tin alloy, and three 95% zirconium - 5% tin alloy plates. The plates in Cartridge ANL-140-C were controls for the plates in Cartridges ANL-140-9 and ANL-140-11.

Post-examination of the stainless steel cartridge and plates revealed clean surfaces. There was no visual evidence of a deposit on the plates, although weight changes indicate some loose deposit was present. These plates showed no evidence of corrosion.

5. Cartridge ANL-140-9: This cartridge contained four 95% zirconium - 5% tin alloy plates and 2 zirconium clad, thorium core plates.

Post-irradiation examination showed a thin dull film of black deposit on the stainless steel surfaces of the cartridge. This deposit was loosely adherent and tended to spall off during handling. The test plates had a rather heavy but loose coating of black deposit. Part of the deposit on these plates had areas with a gray tint. The plates were cleaned by brushing and by rubbing with tissues wet with acetone. The appearance of the plates after cleaning was substantially the same as before irradiation. Figure 15 illustrates the effect of irradiation on the deposition of transported products on 95% zirconium - 5% tin alloy plates. The upper plate, taken from Cartridge ANL-140-9, shows heavy deposition of transported corrosion product. The lower plate, taken from Cartridge ANL-140-C, was subjected to comparable conditions but outside the irradiation zone; it shows the relative cleanliness of plates outside the irradiation zone.

6. Cartridge ANL-140-11: This cartridge contained 2 plates with 95% zirconium - 5% tin clad over 10% U^{235} - 90% zirconium cores, and two 95% zirconium - 5% tin alloy plates. During irradiation cartridges ANL-140-11 and ANL-140-9 were joined together; water flowed through Cartridge ANL-140-11 and then through Cartridge ANL-140-9.

After irradiation both Cartridge ANL-140-11 and the test plates in it were covered with a film of loosely adherent black powder. Identification marks on the test plates were covered. After cleaning, the plates looked very much as they did before irradiation. Comparison of the amounts of loose deposit reveals that the plates from Cartridge ANL-140-11 picked up less deposit than the plates in Cartridge ANL-140-9.

7. Cartridge ANL-140-15: This cartridge contained 12 zirconium - tin alloy plates, 2 enriched uranium fuel plates, and 4 Type 347 stainless steel plates.

Post-irradiation examination of the cartridge surfaces showed a thin black deposit. The relative position of the plates in the cartridge, the direction of water flow, and the amounts of deposit removed are shown in Figure 16. It may be seen that the zirconium-tin alloy plates at the down

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IN FLUX ZONE
CARTRIDGE ANL-140-9

EXTERNAL TO FLUX ZONE
CARTRIDGE ANL-140-C

FIG. 15
COMPARISON BETWEEN DEPOSITION ON ZIRCONIUM - 5 PER CENT TIN
ALLOY SAMPLES IRRADIATED FOR 153 DAYS IN FLUX ZONE AND
EXTERNAL TO FLUX ZONE IN ARGONNE PILOT CHANNEL AT HANFORD

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* = STAINLESS STEEL AISI TYPE 347 PLATES
 ** = ENRICHED URANIUM FUEL PLATES
 BALANCE = ZIRCONIUM-TIN ALLOY PLATES
 NUMBERS ENCLOSED IN PARENTHESES INDICATE WEIGHT (MG/CM²) OF LOOSE DEPOSIT

FIG. 16
 POSITION OF PLATES VS DEPOSITION.
 CARTRIDGE ANL-140-15

NR-G-2900-A

stream end of the cartridge had about twice as much deposit as the leading plates. The 2 fuel plates and the 4 stainless steel plates had deposits approximating that found on the trailing zirconium-tin alloy plates. The amounts of loose deposit removed from the various types of plates exposed in this cartridge were several orders of magnitude less than for the plates in cartridges previously discussed.

The 2 fuel plates, A-2501 and A-2502, had surface patterns that were distinctly different from the heavy black deposit normally found on fuel plates. As may be seen in Figure 17, the plates looked the same on both back and front surfaces; the cores of the plates were outlined by different shades of gray. Another more pronounced pattern was caused by a concentration of black specks. It is assumed that the various patterns found on these fuel plates were caused by local boiling at the end of the test run. This may take place when water recirculation is stopped with residual gamma heating still effective.

The various grades of zirconium used on test plates all showed good corrosion resistance while exposed to irradiation, judging from the adherent black oxide coating found on the plate surfaces. The zirconium-tin alloy plates in Cartridge ANL-140-15 were fabricated from induction-melted Bureau of Mines Grade A-B sponge zirconium. These plates were not considered corrosion resistant in preirradiation corrosion tests; yet, following irradiation, no difference could be noted in the appearance of these plates compared to Grade I crystal bar zirconium plate material.

8. Cartridge ANL-140-E: This cartridge contained 5 zirconium-tin alloy plates, one normal uranium fuel plate, and 3 Type 347 stainless steel plates.

Post-test examination of the cartridge showed a very thin loose surface layer. All test plates also had very little loose transport corrosion product on their surfaces. The stainless steel plates had more loose deposit on their surfaces than did the zirconium-tin alloy plates and the fuel plate. However, the stainless steel plates tested in Cartridge ANL-140-15 in flux showed the presence of slightly greater deposit than the similar plates in this control Cartridge ANL-140-E.

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FIG. 17
POST-IRRADIATION APPEARANCE BEFORE CLEANING OF ZIRCONIUM -
5 PER CENT TIN ALLOY CORE FUEL PLATES IRRADIATED FOR 66 DAYS
AT 540 F IN FLUX ZONE IN ARGONNE PILOT CHANNEL AT HANFORD

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Table VII
(Cont'd.)

		Weight of Plate, gm			
		After Irradiation			
ANL No. A-	Approx. Wt. U ²³⁵ In Core, g.	Measurements, In.	Before Cleaning	After Cleaning	Wt. Change, mg
CARTRIDGE 140-2		Zr Coated Fuel Plates			
502	2.90	6 x 1.5 x .125	123.2734	122.8734	400.0
506	4.12	6 x 1.5 x .095	94.7781	94.4381	340.0
508	1.73	6 x 1.5 x .095	97.0183	96.8479	170.4
509	3.41	6 x 1.5 x .095	98.4299	97.8808	549.0
512	2.27	2 x 1.5 x .125	41.8486	41.7721	76.5
515	2.27	2 x 1.5 x .125	41.7942	41.7068	87.4
527	2.11	6 x 1.5 x .060	58.8856	58.7976	88.0
528	0.59	2 x 1.5 x .125	40.8724	40.8076	64.8
532	1.21	2 x 1.5 x .124	40.9112	40.8663	44.9
534	0.59	2 x 1.5 x .124	40.3769	40.3372	39.7
541	0.71	2 x 1.5 x .094	30.7402	30.7140	26.2
543	0.35	2 x 1.5 x .094	20.5553	20.5342	21.1
549	0.22	2 x 1.5 x .094	30.4641	30.4232	40.9
551	1.06	2 x 1.5 x .094	31.0381	31.0010	37.1
559	0.26	2 x 1.5 x .061	20.3687	20.3212	47.5
561	---*	2 x 1.5 x .093	30.0159	30.0122	75.7
		Stainless Steel AISI Type 347			
		6 x 1.5 x .094	110.9512	110.9134	37.8
		6 x 1.5 x .094	110.2745	110.2564	18.1
		2 x 1.5 x .125	36.9403	36.9238	16.5
		2 x 1.5 x .125	48.9115	48.8898	21.7
CARTRIDGE 140-A		Zr Coated Fuel Plates			
568	---*	2 x 1.5 x .042	30.0825	20.0815	1.0
		Weight of Plate, gm			
		After Irradiation			
		Before Cleaning	After Cleaning		
ANL No. A-	Composition, % Sn in Zr	Measurements, In.	Wt. Change, mg		
CARTRIDGE 140-9					
2075	5	6 x 1.5 x .082	79.7747	79.5602	214.5
2076	5	6 x 1.5 x .084	81.5508	81.2359	314.9
2077	5	6 x 1.5 x .077	73.8992	73.6576	241.6
2078	5	6 x 1.5 x .077	73.9575	73.7111	246.4
2079	(b)	6.5 x 1.5 x .470	704.8230	704.7040	119.0
2080	(b)	6.5 x 1.5 x .470	705.6675	705.4155	252.0
CARTRIDGE 140-11					
2081	(c)	6.1 x 1.25x .080	62.9959	62.8775	118.4
2082	(c)	6.2 x 1.25x .088	73.5373	73.3919	145.4
2083	3	6 x 1.25x .056	44.7635	44.5584	205.1
2084	3	6 x 1.25x .056	45.0329	44.8621	170.8

*Core: Zr: 3% U^{235} .

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Table VII
(Cont'd.)

ANL No. A-	Composition, % Sn in Zr	Measurements, In.	Weight of Plate, gm		Wt. Change, mg
			After Irradiation		
			Before Cleaning	After Cleaning	
<u>CARTRIDGE 140-C</u>					
2085	5	6 x 1.5 x .082	81.3206	81.3157	4.9
2086	5	6 x 1.5 x .055	53.1731	53.1711	2.0
2087	(d)	6 x 1.25x .055	70.9917	70.9896	2.1
2088	5	6 x 1.25x .056	44.9462	44.9417	4.5
<u>CARTRIDGE 140-15</u>			<u>Zirconium-Tin Alloy</u>		
2266	2.59	6 x 1.5 x .087	84.0226	84.0147	7.9
2267	4.15	6 x 1.5 x .087	85.2345	85.2194	15.1
2269	5.03	6 x 1.5 x .087	84.0518	84.0432	8.6
2270	5.05	6 x 1.5 x .087	84.8388	84.8265	12.3
2272	7.15	6 x 1.5 x .087	85.2622	85.2566	5.6
2273	6.63	6 x 1.5 x .087	85.2554	85.2418	13.6
2274	6.38	6 x 1.5 x .087	84.8570	84.8506	6.4
2276	8.40	6 x 1.5 x .087	86.0346	86.0238	10.8
2277	8.47	6 x 1.5 x .087	86.1659	86.1604	5.5
2278	7.55	6 x 1.5 x .087	85.8615	85.8520	9.5
2279	11.00	6 x 1.5 x .087	87.0684	87.0625	5.9
2281	10.40	6 x 1.5 x .087	85.3034	85.2882	15.2
			<u>Enriched Uranium Fuel Plates</u>		
2501	1.55	3 x 1.5 x .060	30.3380	30.3314	6.6
2502	1.51	3 x 1.5 x .060	30.1940	30.1892	4.8
			<u>Stainless Steel AISI Type 347</u>		
15-1		6 x 1.5 x .060	69.1229	69.1106	12.5
15-2		3 x 1.5 x .060	34.1275	34.1204	7.2
15-3		6 x 1.5 x .060	68.5235	68.5123	11.3
15-4		3 x 1.5 x .060	34.2070	34.2015	5.5
<u>CARTRIDGE 140-E</u>			<u>Zirconium-Tin Alloy</u>		
2265	2.02	6 x 1.5 x .087	83.5930	83.5905	2.5
2268	5.03	6 x 1.5 x .060	58.6101	58.6062	3.9
2271	6.69	6 x 1.5 x .087	85.1641	85.1623	1.8
2275	8.40	6 x 1.5 x .087	85.2974	85.2967	0.7
2280	10.40	6 x 1.5 x .087	86.3313	86.3313	---
			<u>Stainless Steel AISI Type 347</u>		
E-1		3 x 1.5 x .060	34.4786	34.4701	8.5
E-2		6 x 1.5 x .087	102.5518	102.5459	6.0
E-3		6 x 1.5 x .060	102.8162	102.8107	5.5
			<u>Natural Uranium Fuel Plate</u>		
A-2503	(h)	3 x 1.5 x .060	31.1143	31.1139	0.4

(a) Induction melted sponge zirconium (A-B Grade) used in fabrication of plates.

(b) A-2079 and A-2080: Thorium core clad with zirconium.

(c) A-2081 and A-2082: 10% U^{235} - 90% zirconium core clad with 5% tin - 95% zirconium.

(d) A-2087: Natural uranium - zirconium core clad with 5% tin - 95% zirconium.

(f) Approximate weight of U^{235} in core of plate, clad with 5% tin - 95% zirconium.

(h) Core, 60% natural uranium, clad with 5% tin - 95% zirconium.

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APPENDIX BOPERATIONAL HISTORY OF WESTINGHOUSE-CHALK RIVER TEST NO. 1

Pertinent details regarding the operating characteristics of the Westinghouse-Chalk River Test No. 1 are quoted:⁽³⁾

"The pressure differential across the in-pile subassembly increased steadily from 3 in. of water, as predicted by calculation, at the start of the experiment, to 8-1/2 in. after 750 hr of operation. Loop flow was increased at this time to 28 gpm, the maximum obtainable with this system, for one hour, and after resumption of 17 gpm operation the pressure drop had fallen to 6 in. It increased again to 7-1/2 in. of water after 215 more hours and 2-1/2 hr 'blast' at 28 gpm lowered it to 6 in. Thereafter, the pressure drop increased over a period of 315 hr to 10-1/2 in. and another 28 gpm 'blast,' this time for four hours, dropped it to 5 in. of water. A further 120 hr of operation raised the pressure drop to 7 in. at which time the loop flow was increased to 21-1/2 gpm for 72 hr and then further raised to 27 gpm, (3.2 fps through the subassembly), which flow rate was maintained for the remainder of the experiment. The subassembly differential increased as the square of the flow rate, as expected, and 27 gpm operation commenced with a differential of 23 in. of water. By the end of the experiment it had risen to 45 in. A reduction in flow to 17 gpm at this time produced a pressure drop of 11 in.

"The experiment terminated on March 1, 1952 after 2728 hr of irradiation at an average pile power of 29.6 megawatts. The total time for the subassemblies at loop operating conditions of temperature, pressure and flow, including operation before and after the irradiation period was about 3550 hr."

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APPENDIX C
SUPPLEMENTARY TABULAR DATA

Table VIII

OVER-ALL FILM HEAT TRANSFER COEFFICIENTS FOR TRANSPORT CORROSION PRODUCTS
OF VARYING Fe_3O_4 CONTENT

Thickness of Deposit, Mils	Fe_3O_4 in Deposit, % By Volume	k_e	h_f	U_o
0.05	38	0.66	157,900	9,400
0.10			78,960	8,900
0.15*			52,640	8,410
0.50			15,790	6,100
1.00			7,896	4,410
0.05	25	0.56	134,400	9,300
0.10			67,200	8,700
0.23*			29,220	7,450
0.50			13,440	5,730
1.00			6,720	4,020
0.05	10	0.44	105,600	9,130
0.10			52,800	8,410
0.50			10,560	5,140
0.57*			9,260	4,800
1.00			5,280	3,450

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*Calculated on the basis of $0.05 \text{ mg}/(\text{cm}^2)(\text{mo})$ corrosion rate

$$k_e = (1-F) k_{\text{H}_2\text{O}} + F k_{\text{Fe}_3\text{O}_4}$$

where:

k_e is the thermal conductivity of the deposit in $\text{Btu}/(\text{hr})(\text{sq ft})(\text{F}/\text{ft})$

F is the volume fraction of Fe_3O_4 in the deposit

$k_{\text{H}_2\text{O}} = 0.356 \text{ Btu}/(\text{hr})(\text{sq ft})(\text{F}/\text{ft})$

$k_{\text{Fe}_3\text{O}_4} = 1.15 \text{ Btu}/(\text{hr})(\text{sq ft})(\text{F}/\text{ft})$

$$h_f = 12,000 k_e/t$$

where:

h_f is the "scale" coefficient of heat transfer in $\text{Btu}/(\text{hr})(\text{sq ft})(\text{F})$

t is the thickness of the deposit in mils

$$U_o = \frac{1}{\frac{1}{h_w} + \frac{1}{h_f}}$$

where:

U_o is the over-all coefficient of heat transfer in $\text{Btu}/(\text{hr})(\text{sq ft})(\text{F})$

h_w is $10,000 \text{ Btu}/(\text{hr})(\text{sq ft})(\text{F})$, the film coefficient for Mark I

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Table IX

SPECTROGRAPHIC ANALYSES OF TRANSPORT CORROSION PRODUCT FROM VARIOUS LOOPS

Element	MIT Tube 2, %	MIT Tube 1, %	ANL Small Heat Throughput Loop		ANL Large Heat Throughput Loop %	ANL Corrosion Loop No. 3 Surge Tank Heater, %	ANL Corrosion Loop No. 3 Surge Tank Heater, %	UCLA Heat Transfer Tubes	
			on SS, %	on Zr, %				No. 23	No. 18
Ag	---	---	---	3.0	---	---	---	---	---
Al	0.5	2.0	>>1.0	6.0	9.8	0.2	2.0	2.0	2.0
Ba	0.1	0.1	1.0	0.1	---	---	---	---	---
Ca	2.0	5.0	0.5	0.4	---	0.6	0.8	1.0	2.0
Co	0.4	0.2	---	---	---	---	---	---	---
Cr	0.04	0.1	>1.0	0.6	7.3	5.0	3.0	1.0	0.6
Cu	2.0	1.0	>>1.0	4.0	1.0	---	---	---	---
Fe	15.0	2.0	>1.0	10.0	34.5	6.0	15.0	10.0	6.0
Mg	4.0	10.0	>1.0	10.0	---	0.6	50.0	60.0	60.0
Mn	0.5	1.0	---	---	---	1.0	2.0	0.3	2.0
Ni	25.0	18.0	>1.0	5.0	---	8.0	2.0	1.0	1.0
Si	2.0	4.0	>1.0	10.0	4.4	2.0	6.0	6.0	2.0

Table X

CHEMICAL AND SPECTROGRAPHIC ANALYSES OF TRANSPORT CORROSION PRODUCT
FROM ARGONNE PILOT CHANNEL AT HANFORD

	Dummy Run		Test Run No. 1						Test Run No. 2			
	Pile Tube Vantuyl 10-2-50 Spec.	Zirconium Cladding Pieces A-2113 10-2-50 Spec.	Pile Tube A-1599 3-7-51 Spec.	140-2 Fuel A-1594 3-7-51 Spec.	140-2 Fuel A-1594 3-7-51 Chem.	140-2 SS A-1595 3-7-51 Spec.	140-2 SS A-1595 3-7-51 Chem.	140-6 Structural A-1762 3-7-51 Spec.	140 Material on Screen A-3080 6-11-51 Spec.	140 Material on Screen A-3080 6-11-51 Chem.	140-1 Fuel and SS A-3083 6-11-51 Spec.	140-1 Fuel and SS A-3083 6-11-51 Chem.
Al	0.2	---	0.83	0.3	---	2.0	---	2.0	0.5	---	1.0	---
Bi	0.5	---	0.03	---	---	---	---	0.01	---	---	---	---
Ca	2.0	2.0	0.83	0.3	---	2.0	---	0.6	1.0	---	0.2	---
Cb	0.1	0.1	0.83	0.2	---	0.06	---	0.4	2.0	---	0.1	---
Cr	10.0	0.2	2.35	1.0	0.83	1.0	0.86	6.0	2.0	4.12	0.3	0.79
Fe	70.0	20.0	16.67	20.0	37.60	20.0	20.00	50.0	50.0	39.70	4.0	56.70
Mg	0.2	0.1	8.33	0.1	---	0.1	---	0.2	3.0	---	0.3	---
Mn	1.0	0.2	1.67	0.4	---	0.2	---	0.8	1.0	---	0.5	---
Ni	5.0	1.0	8.33	2.0	2.7	2.0	1.72	7.0	7.0	5.18	4.0	4.38
Pb	0.5	1.0	0.16	0.2	---	0.2	---	10.0	0.7	---	0.1	---
Si	1.0	0.2	16.67	0.8	0.95	0.8	1.95	1.0	---	1.2	4.2	---
Sn	1.0	---	0.08	---	---	---	---	0.2	---	---	---	---
Ti	2.0	0.007	0.32	0.4	---	0.08	---	0.1	0.4	---	0.1	---
Zr	0.2	---	0.03	20.0	4.3	4.0	0.60	0.4	0.2	---	0.4	---

Table X
(Cont'd.)

	Test Run No. 3				Test Run No. 4			
	140-8 Subassembly A-3078 11-19-51 Spec.	140-8 Subassembly A-3079 11-19-51 Spec.	140-8 Subassembly A-3079 11-19-51 Chem.	140-13 Subassembly A-3081 11-19-51 Spec.	140-15 SS & Zr A-3082 3-2-52 Spec.	140-15 SS & Zr A-3082 3-2-52 Chem.	140-15 Fuel A-3089 3-2-52 Spec.	140-15 Fuel A-3089 3-2-52 Chem.
Al	0.4	2.0	---	1.0	0.6	---	1.0	---
Bi	---	---	---	---	---	---	---	---
Ca	0.1	0.4	---	0.1	0.3	---	2.0	---
Cb	---	0.1	---	0.1	0.7	---	0.1	---
Cr	0.2	0.4	0.22	0.4	2.0	6.29	0.8	1.2
Fe	15.0	30.0	18.5	60.0	50.0	44.8	25.0	15.2
Mg	0.2	0.1	---	0.1	0.2	---	0.5	---
Mn	0.1	0.2	---	0.3	0.4	---	0.2	---
Ni	1.0	4.0	2.11	10.0	6.0	6.29	4.0	3.73
Pt	0.1	1.0	---	0.2	0.8	---	1.0	---
Si	8.0	1.0	---	6.0	3.0	---	8.0	---
Sn	---	---	---	---	---	---	---	---
Ti	0.004	0.5	---	0.03	0.1	---	0.02	---
Zr	50.0	2.0	---	0.4	4.0	---	3.0	---

Table XI

CHEMICAL AND SPECTROGRAPHIC ANALYSES OF TRANSPORT CORROSION PRODUCT
FROM WESTINGHOUSE-CHALK RIVER TEST⁽³⁾

	In-Pile Subassembly*		Out-of-Pile Control Subassembly		Filter Backwash Ion Exchanger Bypass		Loop Heater Material Caught in Static Spot		Scale 3 Chips Spec.
	Spec.	Chem.	Spec.	Chem.	Spec.	Chem.	Spec.	Chem.	
Al	0.07	---	0.15	---	0.7	---	0.08	---	0.005
Cb	< 0.1	---	< 0.01	---	0.75	---	0.25	---	< 0.1
Co	0.35	---	0.40	---	0.10	---	0.10	---	< 0.1
Cu	0.03	---	0.10	---	3.0	---	4.0	---	0.24
Cr	0.60	0.54	0.16	0.28	4.0	0.17	7.5	7.11	32.4
Fe	74.0	60.50	46.0	59.90	52.0	24.60	70.2	47.40	< 1.0
Mg	4.0	---	1.0	---	2.0	---	2.0	---	3.0
Mn	0.5	1.35	0.25	0.36	3.0	0.94	1.2	1.35	26.7
Mo	2.5	---	0.05	---	0.07	---	1.5	---	< 0.01
Ni	16.0	8.96	12.0	13.70	3.0	2.99	6.0	7.06	0.03
Sn	0.15	---	0.7	---	2.8	---	0.10	---	3.0
Ti	0.015	---	0.03	---	1.0	---	0.15	---	7.1
V	0.004	---	0.004	---	0.01	---	0.03	---	< 0.09

*Analysis of HCl soluble portion.

Identification	Soluble in HCl, %	Residue after Ignition, %
Out-of-pile Control Subassembly	100.00	53.8
Filter backwash Ion exchanger Bypass	78.27	50.1
Loop Heater Material Caught in Static Spot	76.00	91.2
In-pile Subassembly	100.00	100.00

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Table XII

PARTICLE SIZE ANALYSES OF TRANSPORT CORROSION PRODUCT
FROM WESTINGHOUSE TESTS⁽³⁾

1. Chalk River

Size Range, microns	%		Cumulative %	
	Filter Backwash	In-Pile Subassembly	Filter Backwash	In-Pile Subassembly
Under 0.5	3	3	3	3
0.5 - 1	5	7	8	10
1 - 1.5	9	10	17	20
1.5 - 2	11	11	28	31
2 - 2.5	11	11	39	42
2.5 - 3	13	11	52	53
3 - 3.5	7	3	59	56
3.5 - 4	5	7	64	63
4 - 5	12	12	76	75
Over 5	24	25	100	100
Mean diameter, microns	3.0		2.9	

2. D Loop (Micrometallic Filters)^{(8)*}

Under 0.5	44	52	44	52
0.5 - 1	31	34	75	86
1 - 1.5	18	10	93	96
1.5 - 2	5	3	98	99
2 - 2.5	2	1	100	100
2.5 - 3	< 0.05	< 0.05	100	100

*These distributions are log normal.

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