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June 1, 1953

FLOW LABORATORY INVESTIGATION OF "F-TYPE"
PITTING OF SLUGS AND TUBES.

INTRODUCTION

Between June and August, 1952, a series of leaking process tubes were discovered in F and D Piles. All of the tubes, six from F and two from D, were removed and found to be leaking from a pitting attack that progressed from inside the tube walls. During this same period a serious slug pitting attack, not previously observed, was found on many slugs discharged at F Pile. Subsequently five slugs from H Pile and one from B Pile showed a similar pitting attack.

In August, 1952, a program was initiated to determine the mechanism of this attack on slugs and tubes and what corrective measures could be taken.

Two hypotheses were presented that were considered the most likely explanations of the observed corrosion effects. The first mechanism presented involved the process of cavitation. Briefly this hypothesis proposed that an obstruction to the water flow in the slug-tube annulus, such as a cocked slug or partially plugged annulus, caused the formation of a low pressure area capable of sustaining water vapor formation. The water vapor thus formed moved into the undisturbed, or higher pressure, regions of the annulus with the subsequent collapse of the vapor bubbles. This mechanism weakened or destroyed the protective oxide coat on the slugs and tubes, producing a localized corrosion phenomenon. The second hypothesis proposed to explain the observed effects was the erosion-corrosion hypothesis. This hypothesis presented the mechanism of protective aluminum oxide coat removal by the abrasive action of small particles in the water with succeeding corrosion of the unprotected aluminum metal. To obtain corrosion on the tubes and all over the slugs, as was the case with pitted slugs at F Pile, areas of extreme turbulence were considered necessary. Therefore cocked slugs, other restrictions in the annulus and extreme turbulence at slug junctions were prime requisites for this mechanism to be valid under normal operating conditions of flow rate and temperature.

These two hypotheses were severely tested in flow laboratory equipment. This document is a report on the results of the laboratory experiments and the correlation of these data to in-pile corrosion data. The quantitative correlation of corrosion rate data obtained in flow laboratory mock-up tubes to data obtained in-pile has not been too successful⁽¹⁾ *. In this investigation, however, in-pile and flow laboratory corrosion data are compared only by visual observations. An assumption is made that corrosion mechanisms may be compared by the visual effects they produce.

(1) Goldsmith, S., "Increasing Power Levels at D Pile by Increasing Permissible Outlet Water Temperatures", HW-27803, May 1, 1953; p. 7.

* The discrepancy between in-pile and flow laboratory corrosion rate data may also be seen by comparing data presented by R. J. Shields in "Interim Report, Production Tests 105-9-P, 105-103-P, and 105-460-P, Corrosion of Slugs", HW-24134, April 18, 1952; to data presented by S. Goldsmith in "Laboratory Studies of 2-S Aluminum Corrosion at Elevated Temperatures", HW-24584, May 27, 1952.

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SUMMARY

Flow laboratory data show that erosion-corrosion in uninhibited process water was the cause of the serious "F-type" pitting of slugs and tubes observed in the summer of 1952. The data further show that cavitation or local boiling due to hot spots could not produce the observed in-pile corrosion effects. Correlation of flow laboratory data to in-pile data is presented to establish the above conclusions.

The addition of 2 ppm sodium dichromate to the various process waters will eliminate the danger of "F-type" slug and tube pitting.

CAVITATION OR LOCAL BOILING HYPOTHESIS

Upon observation of inside-out penetration of process tubes from F Pile the cavitation theory was immediately proposed as the mechanism of attack. The cavitation process is the formation of vapor bubbles or cavities at the metal-liquid interface, then the collapse of the vapor bubbles in a higher pressure region. Unstable vapor bubbles can be formed either by local low pressure areas caused by perturbations in the flow pattern or by local hot spots on the metal surface. The damage to the metal is produced by the high local pressures induced at the point of collapse of the vapor bubbles.

LABORATORY INVESTIGATIONS

Laboratory investigations of cavitation and local boiling which were carried out may be divided into three parts.

1. Glass Tube Studies
2. Local Boiling Studies
3. Laboratory Tube Mock-up Studies

1. Glass Tube Studies

A. Apparatus

Early in the cavitation investigations it was realized that visual observation and photographing of the cavitation process was necessary in order to obtain a true evaluation of the problem. A standard 41 mm ID pyrex glass tube was mounted in the high temperature mock-up facility at 105-D. High pressure radiator hose was used to connect the glass tube to process tubing in the mock-up. Aluminum ribs were fabricated and fastened to three aluminum dummy slugs to support the test slugs. Set screws, in holes drilled and tapped in the ribs, were used to support cocked slugs. A photograph of a section of the tube with the slug assembly in place is shown in Figure 1. In later cavitation studies a high pressure glass tube, with a process rib section cemented in the tube, was obtained from Mechanical Development Sub-Unit. This tube was mounted in the mock-up similarly to the low pressure pyrex tube.

B. Experimental Work

To obtain cavitation it was decided that some constriction to the water flow in the slug-tube annulus was necessary to develop low pressures capable of

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FIGURE 1
GLASS TUBE INSTALLED IN HIGH TEMPERATURE MOCK-UP
Note: slug at left center is cocked from near rib.

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producing bubble formation. Experimental work with venturi nozzles and ogival shaped obstructions⁽²⁾ indicated that bubble formation in a high velocity water stream was most easily obtained by a change in direction of water flow. Specifically, bubble formation occurred when the tangential velocity became great enough to overcome the static pressure holding the liquid to the metal surface. The most easily conceived method of diverging the stream from the slug surfaces was by means of a cocked slug. Therefore preliminary work on cavitation was carried on with cocked slugs in the glass tube set-up. Two cocked slugs were charged into the glass tube and exposed to preheated process water. No bubble formation was observed on the cocked slugs when exposed to water flow rates from 5 to 23 gallons per minute at temperatures from 35 to 95 C. Various flow geometries and position of slug cocking were tried with no positive results. High speed motion pictures were taken of the flow pattern around cocked slugs during these tests. The viewing of the films showed that the flow of water was around the cocked slugs not over them, as was believed to be the case. The blocking of the annulus by the cocked slugs forced the water to escape around and under the slugs; thereby reducing the velocity over the constriction to a value at which cavitation could not take place. A section of the film is presented in Figure 2, showing the flow of gas bubbles in the water stream around a cocked slug.

During the initial cocked slug tests small pieces of scale from a "fouled" heat exchanger were carried into the tube and became lodged at the cocked slugs. These pieces of calcium carbonate reduced the available water annulus enough to produce apparent bubble formation. It was then decided that some means of accurately determining the degree of constriction while uniformly reducing the annulus above the ribs was necessary. A "venturi" slug, that effectively reduced the annulus above the ribs to one-third the original area, was designed and fabricated. Upon exposure it was found that bubble formation was obtained at the venturi constriction at flow rates greater than 12 gpm and temperatures of 20 to 95 C. This bubble formation could be obtained only if the static pressure, as calculated at the constriction, was maintained at 25 psig or lower. The "venturi" slug was exposed to alum treated, dichromate-free Columbia River water at a flow rate of 16.5 gpm and 60 C temperature for four days. The calculated static pressure at the constriction was 17 psig. Figure 3 is a photograph of the slug after exposure. The slug was badly pitted just downstream of the constriction with the majority of the attack within one-quarter inch of the venturi bevel. A trail of small pits extending about one-half inch downstream of the constriction can be seen in the center of the attacked area. No pits were found on the "body" of the slug. During the four day exposure of the "venturi" slug a series of high speed motion pictures were taken of the bubble formation on the slug. It was found, upon viewing the films, that bubble formation did occur at the constriction and that bubble collapse probably took place within one-quarter inch downstream of the constriction. The point of collapse could not definitely be established as the highest speed film (2500 frames per second) did not effectively "stop" the motion of the bubbles. To further study the effect of uniform annulus constriction a series of "fins" were fabricated from one-sixteenth inch aluminum sheet. These fins were designed to simulate the constricted section of the "venturi" slug. When mounted on lead dummy slugs the fins provided an easy and inexpensive

(2) Knapp, R. T., and A. Hollander, "Laboratory Investigations of the Mechanism of Cavitation", Trans. ASME, Vol. 70, 1948; p. 419.

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FIG. 2

Consecutive frames (top to bottom, left to right) of high speed film showing flow around a cocked slug. The slug on the right is cocked on upstream end toward near side of tube. Downstream is to the right. Note path of gas bubble (arrows). Film speed - 920 frames per second.

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FIGURE 3
"VENTURI" SLUG AFTER EXPOSURE.
Note pits just downstream of bevel.

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way to uniformly reduce the available water annulus. The fins were fabricated in three sizes: 1.560, 1.530, and 1.500 inches effective constriction diameters. These fins provided a reduction of the annulus to one-third, one-half and two-thirds of the "normal" area respectively.

Two 1.500 inch "finned" slugs were exposed at the inlet and outlet ends of the glass tube at a series of flow conditions. No bubble formation was observed at flow rates from 5 to 28 gpm, temperatures from 20 to 95 C and static pressures at the constrictions from 10 to 40 psig. Tests in which 1.530 and 1.560 inch "finned" slugs were exposed to various water flow conditions yielded positive bubble formation results. The data from these experiments are presented in Table I. No correlation of static pressure, velocity and water vapor pressure with respect to the inception of cavitation at each condition of velocity and pressure could be obtained.

The data show that bubble formation can occur only at abnormal flow conditions as compared to present pile flow status. It was necessary, for instance, to replace the standard outlet pigtail with a low pressure-drop hose assembly in order to obtain pressures low enough to attain bubble formation at high flow rates. The calculated minimum rear nozzle pressure in a .240 zone tube is approximately 40 psig. At this static pressure on the last active charge the probability of producing cavitation due to flow irregularities is extremely remote. If the pressure in the rear of a tube were low enough to support bubble formation the constriction necessary to increase the velocity to cavitation values would be excessively large and a high panellit indication would in all probability be encountered.

2. Local Boiling Studies

In the local boiling studies carried out in this investigation two objectives were desired. First, to determine the corrosion effects of water vapor bubbles collapsing on aluminum metal surfaces and second, to define the conditions of local boiling with respect to metal temperatures and static pressure. The corrosion effects were determined in an induction heated unit. In this apparatus short sections of process tubes loaded with dummy slugs were exposed to various heat generation rates at a series of water temperatures and flow rates. Hot spots on the tubes and slugs were obtained by restricting the cooling water flow by cocked slugs. Local boiling was obtained at the points of contact between the cocked slugs and the tube walls. The test conditions of the experiments performed in the induction heater are presented in Table II.

TABLE II

INDUCTION HEATER LOCAL BOILING TESTS

Test Number	Water Flow Rate (gpm)	Type of Water	Water Temperature, C		Tube Pressure (psig)	Exposure Time (days)
			Inlet	Outlet		
I	8.0	Alum without dichromate	95.6	98.6	12	8
II	7.5	Alum with 2 ppm dichromate	92.9	96.0	15	8
III	12	Alum without dichromate	78.0	80.5	20	9

TABLE I

GLASS TUBE CAVITATION STUDIES WITH "FINNED" SLUGS

Flow Rate (gpm)	Water Temperature, C	Estimated Static Pressure at Constrictions, psig		Velocity at Constriction, fps	Water Vapor Formation	
		Upstream Fin	Downstream Fin		Upstream Fin	Downstream Fin
1.530 Inch "Finned" Slugs						
14.1	20 to 95	18	10	26.9	No	No
16.7	28	28	15	31.4	No	No
16.8	42	28	15	31.9	No	No
16.8	67	28	15	31.9	No	Yes
16.8	81	28	15	31.9	No	Yes
16.8	97	28	15	31.9	No	Yes
18.7	80	36	20	35.6	No	No
18.7	90	36	20	35.6	No	Yes
20.8	85	43	24	39.6	No	No
20.8	96	43	24	39.6	No	No
1.560 Inch "Finned" Slugs						
10.2	58	18	10	30.3	No	No
10.2	75	18	10	30.3	No	Yes
10.2	92	18	10	30.3	Yes	Yes
13.6	81	22	12	40.3	No	Yes
13.6	94	22	12	40.3	Yes	Yes
15.2	78	28	15	45.3	No	No
15.2	90	28	15	45.3	No	Yes
15.2	97	28	15	45.3	No	Yes
17.5	83	42	22	52.1	No	No
17.5	90	42	22	52.1	No	Slight
19.3	87	51	27	57.5	No	No
19.3	94	51	27	57.5	No	No
21.0	95	62	32	62.5	No	No

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As the type of corrosion attack under investigation in these experiments is very rapid the exposure times of the tests were maintained between 8 and 10 days. The results of exposing aluminum metal to local boiling show that no excessive corrosion of the metal takes place due to the "hammering" of collapsing vapor bubbles. Figures 4, 5, and 6 show the tube sections exposed in Tests I, II, and III respectively. The tube sections are shown because the majority of the heat generation took place in the tube and because the flow pattern and scale effects can best be seen on the tube walls. All of the slugs exposed in these tests showed little or no attack due to the local boiling. The tube shown in Figure 4 was exposed to local boiling over the entire "active" section. The water leaving the tube was partially in the vapor state. The vapor collected on the top of the tube and "swept" the film from the tube wall at the downstream end. A heavy calcium carbonate scale formed over the upper one-third of the tube in the active section. Part of the scale was removed during the tube slitting operation. No excessive corrosion of the tube metal was found at any place in the tube section. The tube section shown in Figure 5 was exposed to local boiling at the contact point between a cocked slug and the tube wall. The area of boiling is shown by the "arrow-shaped" calcium carbonate deposit to the left of center of the top tube section. The film on the tube wall upstream of the scale deposit was partially removed during discharge of the cocked slug. An indication that the vapor bubbles collapsed is shown by the absence of a film removal trail on the downstream end of the top tube section. The bright spots in the rib section are spots where the film has been removed for metal surface examination. No excessive corrosion of the tube surface was found in any section of the tube. A similar calcium carbonate deposit formed by boiling at a cocked slug can be seen in Figure 6. The absence of a film removal trail indicates vapor bubble collapse in the annulus. The metal at the hot spot was in good condition. The slug junction mark to the right of center in Figure 6 was caused by the water turbulence induced by a "finned" slug located at this point during the test.

The tests to define the conditions of local boiling with respect to metal surface temperature and static water pressure were run in the Heat Transfer Sub-Unit resistance heater apparatus. Standard thin-walled 2-S aluminum cans were placed on slip-fit inserts that were in turn connected to the bus bars of the high current d-c generator. One inch sections of the can walls were uniformly machined from the inside to reduce the wall thickness. These reduced metal sections effectively increased the resistivity of the cans, thus producing a higher local heat generation or hot spot when current was passed through the cans. The cans and inserts were mounted in a glass tube and water at the desired temperature and flow rate was fed to the tube. The inside can temperatures were measured with thermocouples placed on a movable probe. The inception of boiling was determined by slowly increasing the heat generation in the can until the formation of vapor bubbles was observed. All of the data presented in Table III were obtained with a film-free, unobstructed can surface. A thin calcium carbonate scale deposit was noticed on a can surface after three hours exposure.

A plot of can surface temperature at start of boiling versus absolute static pressure is given in Figure 7. The plot of saturated water temperature versus absolute pressure is also presented. The can surface temperature at start of boiling exceeded the saturated water temperature from 13 to 16 C over the range of temperatures and pressures covered in the experiment. No relationship of temperature difference between can surface temperature and saturated water temperature to absolute pressure could be determined.

FIGURE 4
TUBE SECTION EXPOSED TO LOCALIZED BOILING
Note heavy scale and water vapor trail on upper half of tube.
Half of rib section removed for examination.

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FIGURE 5
TUBE EXPOSED TO LOCAL BOILING AT A COCKED SLUG POSITION

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FIGURE 6
TUBE SECTION EXPOSED TO LOCAL BOILING
Note slug junction mark caused by "Finned" slug.

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TABLE III

LOCAL BOILING AS A FUNCTION OF METAL SURFACE
TEMPERATURE AND STATIC WATER PRESSURE

Water Flow Rate (gpm)	Water Temperature, C		Static Water Pressure (psia)	Observations (Boiling)	Metal Surface Temperature, C	Heat Generation (KW/ft.)
	Inlet	Outlet				
18.0	90.5	97.2	22.8	Start	126	84.4
18.0	90.0	98.9	22.8	Yes	133	113
18.0	87.8	97.8	35.0	Start	142	135
13.0	88.3	97.2	22.7	Start	127	62.3
13.0	86.7	96.1	22.7	Yes	130	69.2
13.0	83.8	96.7	42.8	Start	149	117
13.0	87.8	100	62.8	Start	161	144

Boiling observed in this experiment was the small, individual bubble type as differentiated from over-all or "sheet" boiling. The more severe over-all boiling would be encountered at higher heat generation rates and higher can surface temperatures.

The primary aim of these local boiling studies was to determine the probability of local boiling on slug surfaces under normal pile operating conditions. The data show that local boiling on the body of a slug is very improbable at present power levels and flow rates. To have slug surface boiling on the most downstream active metal surfaces in a normal .240 zone tube a metal surface temperature of about 151 C is required. As the downstream metal is in a low heat flux region this surface temperature would be practically impossible to attain. The surface temperature necessary to produce boiling in the highest heat flux region of a normal .240 zone tube would be approximately 180 - 190 C. In this region, just downstream of center, the bulk water temperature is relatively low and can surface temperatures of 180 - 190 C would be very difficult to reach under normal operating conditions.

3. Laboratory Tube Mock-up Studies

In conjunction with the glass tube studies on cavitation a series of laboratory tube mock-up studies were carried out. In these studies, flow configurations under observation in the glass tube were duplicated in the high temperature mock-up and exposed under various flow rates and temperatures for extended periods of time. The tests, in general, involved the exposure of coked slugs and "finned" slugs to determine the effect of cavitation on slug and tube corrosion. Conditions and results of the individual tests are presented in Table IV.

With the exception of the stressed tube tests no concentrated corrosion of slugs or tubes was observed. As the glass tube tests revealed the difficulty of obtaining cavitation in a process tube set up, it is possible that cavitation was not produced in the laboratory mock-up tests despite the effort to control the exposure conditions at cavitation levels. Whether cavitation was obtained or not these tests yielded positive results to the effect that cavitation is not the cause of the localized pitting of slugs and tubes that has been noticed during the past year.

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TABLE IV

LABORATORY TUBE MOCK-UP STUDIES OF CAVITATION

<u>Type of Water</u>	<u>Test Pieces</u>	<u>Exposure Conditions</u>	<u>Observations</u>
Alum without dichromate	Cocked Slugs Stressed Tubes 1. Unstressed 2. Stressed, Compression 3. Stressed, Torsion	1. 12 days at 20 gpm and 90 C 2. 12 days at 20 gpm and 90 C 3. 9 days at 15 gpm and 90 C	1. Slight attack on cocked slugs. No tube attack. 2. Heavy pitting on front lead dummy and tube adjacent to dummy. One cocked slug and two normally charged slugs had similar pitting on upstream bevels. Pitting caused by calcium carbonate particles in water from a "fouled" heat exchanger. 3. Two cocked slugs showed heavy pitting just downstream of upstream bevel. Water contained scale from "fouled" heat exchanger. No tube attack.
Alum with 2 ppm dichromate	Cocked Slugs	15 days at 20 gpm and 95 C	Very mild attack on cocked slugs.
Alum without dichromate	Cocked Slugs	10 days at 20 gpm and 95 C	Mild attack on cocked slugs.
Alum without dichromate	1.530 Inch "Finned" Slugs 1.560 Inch "Finned" Slugs	16 days at 20 gpm and 95 C 8 days at 20 gpm and 95 C	1.530 inch "finned" slugs showed slight attack on fins and on the bodies of the slugs. No attack on slugs downstream of the fins. 1.560 inch "finned" slugs showed attack on fins and pits along rib marks on the bodies of the slugs. No attack on slugs downstream of the fins.
Alum with 2 ppm dichromate	1.520 Inch "Finned" Slugs 1.560 Inch "Finned" Slugs	14 days at 20 gpm and 95 C 10 days at 20 gpm and 95 C	1.530 inch "finned" slugs had slight attack at rib marks. No attack on fins or on slugs downstream of fins. 1.560 inch "finned" slugs showed slight attack at rib marks on the bodies of the slugs. No attack on fins or on slugs just downstream of fins.

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The excessive corrosion observed in the stressed tube tests was traced to the presence of calcium carbonate scale in the water. This scale, from a fouled heat exchanger, produced an erosion-corrosion effect on the slugs and tubes. A complete description of these test pieces is presented in the erosion-corrosion section of this document.

EROSION-CORROSION HYPOTHESIS

At the outset of the program to determine the cause of the leaking tubes and "F-type" pitted slugs the erosion-corrosion theory was thought less applicable than the cavitation theory. However, just previous to and immediately following the start of this program three separate occurrences in flow laboratory mock-up tests enhanced the erosion-corrosion theory as an explanation of the observed facts.

1. On June 18, 1952, a tube exposed in the 105-D Flow Laboratory to raw river water at 95 C and 20 gpm developed a leak after 12 days operation. An attempt was made to seal the leak with hose clamps and the tube was run for an additional four days. After 16 days exposure the tube was removed and slit open for examination. It was found that the leak had occurred adjacent to a rib and at a spot corresponding to a slug junction. The tube was also pitted on the opposite rib one slug length upstream of the tube perforation. Figures 8 and 9 are photographs of the tube rib section showing the hole and pitted rib. An indication of the flow pattern and slug positions can be obtained by an examination of the upper tube section shown in Figure 10. The light spot just downstream of the lead dummy position is where the first aluminum dummy slug touched the tube wall and the light spot at the next downstream slug junction is the point at which the next aluminum dummy touched the tube wall. If the slug orientation is visualized it will be noted that the first aluminum dummy was cocked from the left rib (looking downstream in the tube) at the front of the slug and the next downstream aluminum dummy was cocked from the right rib at the front of the slug. It can then be seen that the distance between the left rib and the first dummy slug was at a maximum at the front of the slug and decreased uniformly to the downstream end of the slug, where the slug touched the rib. The decreasing annulus between the slug and tube rib apparently forced the water flow outward against the tube and the outward direction and velocity reached a maximum at the point where the rib was pitted as shown in Figure 9. A similar reconstruction can be made for the next downstream slug, the right rib and the hole shown in Figure 8. It is difficult to conceive that the force exerted by the water alone impinging on the tube was great enough to produce the observed concentrated attack. However, the mechanism of erosion-corrosion seems plausible if the fact is considered that raw river water contained an average turbidity of 12 ppm over the exposure period.
2. On July 14, 1952, a slug discharged from the test to determine the effect of velocity on 2-S slug jacket corrosion showed an extremely heavy pitting attack on the upstream end of the slug. This slug was the first upstream slug in a tube through which ferric sulfate treated, dichromate-free water flowed at 20 gpm and 95 C for 12 days. A photograph of the slug is presented in Figure 11. At the completion of the velocity test the tube was removed and examined. The tube was found to be pitted at the point corresponding to the position of the pitted area on the slug. Figure 12 is a photograph of the pitted section of the tube. During discharge of the slug and tube, pea-sized particles of calcium carbonate scale were found in the tube. Later, examination of the heat exchanger and connecting

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FIGURE 8
TUBE SECTION SHOWING HOLE NEAR RIB
Note: The hole was probably increased in
size due to the efforts to seal the leak.

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FIGURE 9
PITTED RIB SECTION LOCATED ONE SLUG LENGTH
UPSTREAM OF SECTION SHOWN IN FIGURE 8

Eight Inch Lead Dummy Position

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No Attack
On Ribs

Pitted Rib
Figure 9

Hole
Figure 8

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FIGURE 10
TOP HALF OF TUBE SECTION SHOWN IN FIGURES 8 AND 9

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FIGURE 11
SLUG FROM THE EFFECT OF VELOCITY TEST.
Note penetration to Al Si bond in badly corroded areas.

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FIGURE 12
TUBE SECTION OPPOSITE PITTED AREA OF SLUG SHOWN IN FIGURE 11.

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piping revealed large deposits of scale in the process water system. The attack on the slug and tube was believed to be caused by the erosive action of scale in the water impinging on the slug and tube. The attack was probably localized by a high velocity segment in the annulus produced by an accumulation of scale around the front of the slug.

3. A pitted tube and four severely pitted slugs from tests to determine the effects of cocked slugs on tube and slug corrosion further substantiated the erosion-corrosion theory. In these tests three tubes, containing identical cocked and normally charged slug loading patterns, were exposed to dichromate-free, aluminum sulfate treated water at 95 C for 9 to 12 days. Two tubes at flow rates of 20 and 15 gpm were fed preheated water from a "fouled" heat exchanger. The other tube was fed water at 20 gpm from a clean heat exchanger. The tube and all of the slugs exposed for 12 days to water from the clean heat exchanger showed no excessive corrosion. The most upstream slug, an eight inch lead dummy, in the 20 gpm tube fed from the "fouled" heat exchanger for 12 days was severely pitted on the upstream end. Photographs of the lead dummy and the tube section at the position of the lead dummy slug are presented in Figures 13 and 14. The tube was pitted in a manner similar to the tube section shown in Figure 12. The mechanism of this attack is believed to be identical to that hypothesized for the slug and tube presented above. One normally charged aluminum dummy slug from the tube in which the pitted lead slug was exposed showed a heavy pitting attack just behind the upstream bevel, as seen in Figure 15. As far as could be determined, this slug was never cocked in the tube. However, it was in an extremely turbulent section of the tube; being one slug length downstream of a cocked slug. The tube was not pitted adjacent to this slug. Two pair of cocked slugs in this tube exhibited no excessive corrosion and they did not produce any noticeable corrosion of the tube at their exposure positions. Two cocked slugs from the 15 gpm tube, also fed from the "fouled" heat exchanger, showed a very heavy pitting attack on their surfaces. Figures 16, 17 and 18 are photographs of these slugs which were exposed to alum water at 95 C for 9 days. The slug shown in Figures 16 and 17 was located just upstream of the slug shown in Figure 18. Pitting on the leading bevel of the upstream cocked piece had penetrated almost through the can and complete penetration of the can wall had occurred on the upstream end of the downstream cocked slug. The tear-drop shaped pits, with apexes pointing upstream, shown in Figure 18, resemble very markedly the "F-type" pits observed on in-pile exposed slugs (see Figure 46). All of the concentrated pitting attack observed in these tests was undoubtedly caused by the presence of calcium carbonate scale in the water.

The evidence from these three incidents strongly supported the erosion-corrosion hypothesis. This evidence, coupled with the fact that the cavitation program was not yielding positive results, initiated a re-orientation in the program to determine the causes of "F-type" pitting of slugs and tubes.

LABORATORY MOCK-UP INVESTIGATIONS

Upon obtaining the evidence supporting the erosion-corrosion theory presented above, a review of the 100 areas process water cotton plug analyses was made. A plot of cotton plug solids in ppm for January to October 1952, is presented in Figure 19. It will be noted that 100-F experienced three periods of high turbidity, two before and one after the elimination of sodium dichromate as a process water inhibitor. As the

FLOW →

FIGURE 13
LEAD DUMMY SLUG EXPOSED IN FLOW LABORATORY TUBE.
Note triangular - shaped pits similar to pits observed on in-pile
exposed slugs. Apexes of triangles always point upstream.

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FIGURE 14
TUBE SECTION OPPOSITE LEAD DUMMY SHOWN IN FIGURE 13.

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FIGURE 15
ALUMINUM DUMMY SLUG EXPOSED IN SAME
TUBE AS SLUG SHOWN IN FIGURE 13.

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FLOW →

FIGURE 16
SLUG EXPOSED TO WATER FROM A "FOULED"
HEAT EXCHANGER.

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FLOW →

FIGURE 17
OPPOSITE SIDE OF SLUG SHOWN IN FIGURE 16.

FLOW →

FIGURE 18
ALUMINUM DUMMY SLUG FROM SAME TUBE AS
SLUG SHOWN IN FIGURE 16.
Note triangular-shaped pits similar to "F-Type"
pits observed on in-pile exposed slugs.

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FIGURE 19
COTTON PLUG SOLIDS FOR 1952

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first tube leaks and pitted slugs at F Pile were encountered in June 1952, the elimination of sodium dichromate was suspected to be the key to the observed corrosion phenomena. The elimination of dichromate, a peak turbidity of 0.10 ppm following the elimination of dichromate and the slightly high average turbidity at 100-F indicates the possibility that the corrosion problems were initiated by an erosion-corrosion process in uninhibited process water.

To study the effects of turbidity on slug and tube corrosion in various types of water a series of tests were performed. The experimental conditions of these tests are presented in Table V.

TABLE V

TURBIDITY TESTS

Test Number	Type of Water	Diatomaceous Earth (ppm by weight)*	Water Flow Rate (gpm)	Temperature, C	Exposure Time (Days)
1	Alum with 2 ppm dichromate	9.2	23	87	20.2
2	Same as above	0.9	23	87	15.0
3	Alum without dichromate	8.4	15	90	17.3
4	Same as above	1.1	22	88	6.0
5	Ferrifloc without dichromate	9.4	17.5	90	17.3
6	Same as above	1.1	22	90	25.3
7	Same as above	0.1	22	90	10.1
8	Alum with 2 ppm dichromate	0	20	95	27.0
9	Alum without dichromate	0	20	95	22.0

* Average over total operating period.

These tests were run in flow laboratory mock-ups at 105-D, 105-F, and 190-H. Data were obtained on standard (1.610 ID) process tubes and four-inch aluminum canned dummy slugs. Preheated water was fed to the experimental tubes at 105-D and 190-H. A steam jacketed process tube was used at 105-F to supply water at the desired temperature. Diatomaceous earth slurry was fed to the mock-up tubes by means of jet eductors located on the inlet pigtails. The slurry was metered through small (0 to 200 ml/min) rotameters. Two cocked slugs, one 1.530 inch "finned" slug and a series of normally charged slugs were exposed in each of the tests. The relative positions of the special and normally charged pieces were held constant throughout the tests. At the completion of each of the tests the slugs and tubes were removed and examined for excessive corrosion.

RESULTS

A general outline of the results of the turbidity tests is given in Table VI. As the examination and reporting of evidence of in-pile slug and tube corrosion are treated as separate investigations, the results of the turbidity tests on slug corrosion and tube corrosion will be discussed individually.

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TABLE VI

RESULTS OF TURBIDITY TESTS

Test Number	Type of Water	Turbidity (ppm)	Observations
1	Alum with 2 ppm dichromate	9.2	Slugs and tube covered with very tenacious, smooth, dark brown film. No excessive corrosion of tube or slugs. "Finned" slug and tube at fin position in good condition.
2	Alum with 2 ppm dichromate	0.9	Slugs and tube covered with smooth, dark brown film. No excessive corrosion of tube or slugs. Fin in good condition.
3	Alum without dichromate	8.4	Slugs have very rough surfaces. Autoclave coating broken in many places. Fin chewed up. Tube pitted on ribs at fin position, and pitted on wall above fin and at cocked slug positions. Little or no film on slugs and tube.
4	Alum without dichromate	1.1	Slugs pitted slightly. Exposure time too short for adequate appraisal of this set-up. Very light film on slugs and tube.
5	Ferrifloc without dichromate	9.4	Slugs badly pitted on upstream bevels. Autoclave coating completely removed from slugs in very turbulent regions. Fin chewed up. Both ribs deeply pitted at finned slug position. Upper part of tube pitted to depth of 1/3 the tube wall thickness at fin junction. Rib pitted at the point the downstream end of a cocked slug touched the rib. Tube also pitted on a rib and between ribs at junction of normally charged slugs. Little or no film on tube and slugs.
6	Ferrifloc without dichromate	1.1	Slugs and tube covered with light, easily removed film. Film on slugs was broken with corrosion of slug surfaces at points film was removed. Autoclave film broken in many places. Tube pitted on ribs at fin junction. Fin chewed up.
7	Ferrifloc without dichromate	0.1	Slugs and tube covered with very light film. Few small pits on slug surfaces. Tube showed signs of attack on ribs at fin junction. Fin chewed up.
8	Alum with 2 ppm dichromate	0	Slugs and tube covered with light greenish-brown film. No noticeable corrosion of slugs or tube.
9	Alum without dichromate	0	Slugs and tube covered with light brown film. No noticeable corrosion of slugs or tube.

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1. Tube Corrosion

In general the results of the turbidity tests on tube corrosion duplicate the in-pile observations. Figure 20 is a photograph of the loaded sections of four of the tubes from the effects of turbidity tests. The tube from Test 1 had a thin, very tenacious film over the normal flow regions. The two bright spots shown at the upstream and downstream ends of the top half of the tube are points where cocked slugs touched the tube wall. The position in the tube at which the "finned" slug was exposed is shown by the "junction" mark at the center of the photograph. Little or no corrosion occurred at any point in this tube. Film on the tube walls in Tests 5, 6, and 7 varied from no film in Test 5 to a small amount of loosely adhering film in Test 7. The order of severity of attack on these three tubes, all exposed to Ferrifloc treated, dichromate-free water, was in direct proportion to the turbidity concentration of the water. The tube from Test 5 was very badly pitted on the ribs and above the ribs at the "finned" slug position. Figure 21 is a close-up of the tube section and slugs at this position. Pitting of one rib can be seen three slug junctions upstream of the fin position. This pitting occurred at the downstream end of a cocked slug that was cocked from the rib on which the pitting attack took place. A pitting attack on the opposite rib and between the ribs appeared at the next downstream slug junction. Apparently the attack at this point took place at a "normal" slug junction. A photograph showing a close-up of the pitting at these two slug junction positions can be seen in Figure 22.

The appearance of slug junction marks on the tubes from the uninhibited water tests indicate that corrosion is taking place at these very turbulent regions. The relatively smooth film surface on the tubes from the inhibited water tests indicate that dichromate tends to "heal" the film even in very turbulent regions, thereby protecting the tube metal from the erosive action of the turbidity in the water.

If a comparison is made of flow laboratory and in-pile tube corrosion data a very strong case for erosion-corrosion is evident. Figures 23 and 24 are photographs of an in-pile tube exposed at 105-F over the period of high turbidity and non-inhibited water during the summer of 1952. Note the similarity of these pitted ribs to those shown in Figure 22. Note in each case that the attack took place at a slug junction position. The pitted areas shown in Figures 25 and 26 were on and between the ribs of in-pile exposed tubes. Note the similarity of this attack to that shown on and between the ribs in Figures 8, 9, 21, and 22. The pitted slug junction marks shown in Figure 27 resemble very markedly the attack at the "finned" slug position seen in Figure 21. (The use of a "finned" slug to produce extreme turbulence and rapid corrosion can be justified by the reduction in testing time necessary to duplicate long time in-pile tube exposure.) The likeness of the attack noted on laboratory exposed tubes to that observed on in-pile exposed tubes leaves no doubt that the mechanism of attack is the same for both types of exposure; namely, erosion-corrosion.

2. Slug Corrosion

The data from the tests to determine the effects of turbidity on slug corrosion confirm that the process of erosion-corrosion was the mechanism of the corrosion attack observed on in-pile exposed slugs. Figure 28 is a photograph of a representative slug from each of the turbidity tests. It should be noted that every one of the slugs exposed to uninhibited water shows breaks in the hydrous oxide film and/or pits

TEST NO. 7

TEST NO. 6

TEST NO. 5

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TEST NO. 1

FIGURE 20
TUBES FROM EFFECTS OF TURBIDITY TESTS.
Note: Downstream is to the left.

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FIGURE 21
TUBE SECTION AND SLUGS AT "FINNED" SLUG
JUNCTION FROM TEST 5.

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FIGURE 22
PITTED TUBE RIB SECTION FROM TEST 5.

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← FLOW

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FIGURE 23

INSIDE VIEW OF A SECTION OF TUBE 3684-F,
REMOVED FROM THE PILE ABOUT 8-8-52, SHOWING
PITTING ON RIB AT A SLUG JUNCTION.

← FLOW

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FIGURE 24

ANOTHER SECTION OF TUBE 3684-F, SHOWING
A DIFFERENT AREA OF PITTING ATTACK.

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FIGURE 25
SECTION OF PROCESS TUBE 1692-D, REMOVED
FROM THE PILE ON 8-26-52.
The hole was located 8-1/2 feet
from the rear Van Stone flange.

FIGURE 26
SECTION OF PROCESS TUBE 3175-D, REMOVED
FROM THE PILE ON 8-8-52.
The hole, located 8 feet from the rear Van Stone
flange, is in the bottom of the pit between the ribs.

FIGURE 27
INSIDE VIEW OF TUBE 3184-D, REMOVED FROM THE PILE ON 1-22-53.
Note the pitting around the tube wall at slug junctions.

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FIGURE 28
REPRESENTATIVE SLUGS FROM THE TURBIDITY TESTS.
Note absence of attack on slugs exposed to water containing
2 ppm dichromate (Tests 1 and 2).

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into the slug jackets, while the slugs exposed to water containing 2 ppm sodium dichromate (Tests 1 and 2) show no breaks in the film or pits on the slug surfaces. From the results of the turbidity tests the mechanism of slug corrosion for both laboratory turbidity tests and "F-type" pitting observed on in-pile exposed slugs can be deduced as follows:

1. Removal of the hydrous oxide film from the slug surfaces by the erosive action of particles in water.
2. Removal of the protective autoclave coating at the areas devoid of film by erosion-corrosion.
3. Subsequent corrosion of the aluminum can at the "weakened" areas, aided by the erosive action of turbidity in the water.

This mechanism is proposed only for uninhibited water, as dichromate aids the formation of a tenacious, tough hydrous oxide film and also acts as an oxidizing agent to "heal" any breaks in the autoclave coating or metal surface. The autoclave coating is the aluminum oxide formed on the slug surfaces during testing in steam autoclaves.

Figure 29 is a photograph of five slugs that were exposed to dichromate-free, Ferrifloc treated water containing 9.4 ppm diatomaceous earth at a flow rate of 17.5 gpm and temperature of 90 C for 17.3 days (Test 5). These test conditions were so severe that complete removal of the autoclave coating over most of the surfaces of four of the slugs shown in Figure 29 was accomplished. (The center slug is an unautoclaved lead dummy.) The slugs shown in Figure 30 were exposed in Test 3 (alum water without dichromate, 8.4 ppm turbidity, 90 C, 15 gpm, 17.3 days). The conditions of this test were not severe enough to produce complete removal of the autoclave coating. The surfaces of these slugs are very rough and many breaks in the autoclave coatings can be seen. An examination of the slugs from Test 6 (Ferrifloc water without dichromate, 1.1 ppm turbidity, 90 C, 22 gpm, 25.3 days) shows that the areas of film removal and pitting attack (not as severe as that noted for the two tests presented above) was similar to the attack that was observed on in-pile slugs exposed for a much longer time. A close examination of the surfaces of the slugs from the turbidity tests shows the relative areas and degrees of attack from the different tests. It also affords a means of comparison with longer exposure in-pile slug corrosion. Figures 31 through 40 are photographs of the slug surfaces from the various turbidity tests. Figures 31 and 32 are photographs of slug surfaces exposed in Test 5. Figure 31 shows an area where partial removal of the autoclave film is evident, while Figure 32 shows an area completely devoid of autoclave coating. The arrow-shaped pit seen in Figure 32 resembles the "F-type" pitting noted on in-pile slugs. The position of this pit on the slug can be seen near the upstream end of the slug on the right in Figure 29. Photographs of slug surfaces exposed in Test 3 (alum water without dichromate, 8.4 ppm turbidity, 15 gpm) are shown in Figures 33, 34, and 35. The extremely rough and heavily pitted surfaces of these slugs can be seen. The areas containing many small pits were quite prevalent on these slugs indicating that the test conditions were too severe to obtain discrete large pits. The slug surfaces shown in Figures 36, 37 and 38 are photographs of slugs from Test 6 (Ferrifloc water without dichromate, 1.1 ppm turbidity, 22 gpm). These slug surfaces exhibited areas of discrete pitting attack similar to that observed on in-pile slugs. Figures 39 and 40 are photographs of slug surfaces exposed in Test 1 and 2 (alum water with 2 ppm dichromate, 9.2 and 0.9 ppm turbidity, 23 gpm). These two photographs were taken of the areas at which the most severe pitting occurred. The extremely good condition of the slug surfaces should be

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FIGURE 29
FIVE SLUGS FROM TURBIDITY TEST 5.
Note absence of the autoclave coating on the four outside slugs.

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FIGURE 30
SLUGS FROM TURBIDITY TEST 3.
Note rough surfaces and breaks in autoclave coating.

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FIGURE 31
SURFACE OF SLUG FROM TEST 5 SHOWING PARTIAL
REMOVAL OF AUTOCLAVE COATING.
Black line is a pencil mark. 5.1X

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FIGURE 32
SLUG SURFACE (TEST 5) SHOWING COMPLETE
REMOVAL OF AUTOCLAVE COATING.
Note "F-Type" pit in surface. 5.1X

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FIGURE 33

SLUG SURFACE EXPOSED TO DICHROMATE-FREE
ALUM WATER AT 8.4 ppm TURBIDITY (TEST 3).
Note many breaks in autoclave coating and few large pits.

5.1X

RESTRICTED

FIGURE 34

SLUG SURFACE EXPOSED IN SAME TEST AS
SURFACE SHOWN IN FIGURE 33.

Note dark areas where autoclave coating is re-
moved and pitting of aluminum has occurred.

5.1X

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FIGURE 35

SLUG SURFACE EXPOSED IN TURBIDITY TEST 3.
Note extremely rough surface and many pits in
autoclave coating. 5.1X

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FIGURE 36

SLUG SURFACE EXPOSED TO DICHROMATE-FREE
FERRIFLOC WATER CONTAINING 1.1ppm TURBIDITY (TEST NO. 6).
Note many discrete areas of pitting on surface. 5.1X

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FIGURE 37
SLUG SURFACE EXPOSED IN TURBIDITY TEST 6.
Note shape of three consecutive pits at left center. 5.1X

FIGURE 38
SLUG SURFACE EXPOSED IN TURBIDITY TEST 6.
Note line of deep pits just above a rib mark. 5.1X

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FIGURE 39
SLUG SURFACE EXPOSED TO ALUM WATER CONTAINING
2 ppm SODIUM DICHROMATE AND 9.2 ppm TURBIDITY (TEST 1).
Note extremely good condition of surface. 5.1X

FIGURE 40
SLUG SURFACE EXPOSED TO ALUM WATER CONTAINING 2 ppm
SODIUM DICHROMATE AND 0.9 ppm TURBIDITY (TEST 2).
Note smooth, relatively unattacked surface. 5.1X

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noted. A more convincing display of the effectiveness of dichromate as an inhibitor can be seen in Figure 41. This figure is a photograph of six of the "finned" slugs from the various turbidity tests. Note the condition of the fins on each of the slugs. All of the fins exposed to uninhibited water are badly chewed up, but the fins from the tests where dichromate was present in the water show no attack whatsoever.

A correlation of flow laboratory turbidity test data with in-pile slug pitting data can be obtained by viewing a few examples of in-pile slug pitting. Figure 42 is a view of a pitted slug discharged from tube 3684-F in July 1952. An observation made of this slug at time of examination was that the attack noted on the slug surface was the initial stages of the more severe "F-type" pitting(3). Note the breaks in the autoclave coating and the small pits similar to those shown in Figures 30 and 33 through 38. The slug shown in Figure 43 was discharged from the same tube as the slug shown in Figure 42. The pitting attack noticeable over most of the surface resembles a more advanced condition of the attack seen in Figures 34 through 37. The pitting attack in a line just below an apparent rib contact area seen in Figure 43 resembles very markedly the attack shown in Figure 38. The slug shown in Figure 44 was discharged from tube 3879-F in August 1952. The severe attack covering a relatively large area is similar to that observed on the slugs from Test 3 (Figures 30, 33, 34, and 35). Note the absence of attack on rib marks. The one slug from B Pile that exhibited "F-type" pitting is shown in Figure 45. Note the similarity of the attack seen on this slug to the attack observed on slugs from flow laboratory experiments (Figures 16, 18, and 29). Another pitted slug that resembles flow laboratory exposed pieces is shown in Figure 46. This slug was discharged from tube 1479-F in October 1952. The concentration of the attack near the end of the slug indicates the mechanism of corrosion was the same as that of the slugs shown in Figures 16 and 18.

The similarity of in-pile exposed pitted slugs to flow laboratory turbidity test slugs definitely indicates that the "F-type" pitting found on in-pile charges during 1952 was caused by the erosive action of turbidity in uninhibited water.

DISCUSSION OF RESULTS AND RECOMMENDATIONS

The results of the laboratory tests to determine the causes of leaking tubes and severely pitted slugs observed on in-pile exposed pieces during the summer of 1952, can be summarized as follows:

1. The extremely serious corrosion of in-pile slugs and tubes during 1952 was not caused by cavitation or local boiling due to hot spots.
2. Erosion-corrosion in uninhibited water resulted in the observed corrosion of in-pile slugs and tubes.
3. The addition of an inhibitor, 2 ppm sodium dichromate, to the water effectively eliminates tube and slug corrosion that is induced by the presence in process water of turbidity up to a concentration of 10 ppm.

The experiments on cavitation and local boiling on a cylindrical slug surface indicated that the possibility of either of these processes occurring in-pile is extremely remote. The fact that in-pile exposed slugs corroded more severely from erosion-corrosion than flow laboratory slugs can be accounted for by the fact that the in-pile

(3) Rohrbacher, R. A., "Technical Activities Report, Irradiated Materials Examination, September 1952", HW-25682, October 10, 1952; p. 28.

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FIGURE 41
"FINNED" SLUGS FROM THE TURBIDITY TESTS.
Note good condition of fins exposed to water
containing 2 ppm dichromate. (Tests 1 and 2).

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FIGURE 42
SLUG FROM TUBE 3684-F, DISCHARGED ON 7-31-52.
Note beginning of pitting attack and general roughness of surface.

FIGURE 43
SLUG FROM TUBE 3684-F, DISCHARGED ON 7-31-52
Note absence of attack at possible rib contact areas.

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FIGURE 44

SLUG FROM TUBE 3879-F, DISCHARGED ON 8-30-52.

Note rib mark adjacent to badly corroded area.

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FIGURE 45

SLUG FROM TUBE 0962-B, DISCHARGED ON 9-9-52.

Note concentration of attack near bevel.

neg 052179-33

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FIGURE 46

SLUG FROM TUBE 1479-F, DISCHARGED ON 10-19-52.

Note concentration of attack on upstream end and characteristic "Tear Drop" shape of pits.

neg 052205-37

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slugs were exposed to erosion-corrosion for a much longer time. Recent data have indicated that the corrosion of 2-S aluminum in process water is substantially increased in the presence of high pile flux⁽⁴⁾ which would account for the more severe corrosion observed on in-pile charges. The effectiveness of 2 ppm sodium dichromate as an aluminum corrosion inhibitor, demonstrated in the effects of turbidity tests, further substantiated previous data on this subject⁽⁵⁾. An investigation of the feasibility of relaxing the process water turbidity limits is urged, provided a suitable inhibitor is present in the water at all times.

The fact that the excessive corrosion of in-pile tubes and slugs observed during the past summer occurred during a period when uninhibited water was being fed to the piles and the fact that no corrosion of this type occurred when water containing 2 ppm sodium dichromate was fed to the piles indicates that dichromate inhibitor will eliminate this corrosion problem. It should be realized that a number of tubes and slugs, now in the piles, were probably damaged by erosion-corrosion during the summer of 1952. These charges may fail, due to their weakened condition, even with dichromate present in the water.

On the basis of flow laboratory data obtained on leaking tubes and pitted slugs and the correlation of these data to in-pile corrosion data it is recommended that the use of 2 ppm sodium dichromate as an aluminum corrosion inhibitor in process water be continued until data for lowering the dichromate concentration or the use of another inhibitor are obtained.

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- (4) Goldsmith, S., "Interim Report Number 1, PT-105-510-E, Determination of Pile Irradiation Effect on Corrosion of 2-S Aluminum", HW-27531, June 1, 1953.
- (5) Kidder, G. P., "CMX Final Report", 7-4444, July 31, 1946; p. 7.