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1

INTRODUCTION

The circulation of an aqueous slurry causes erosive damage to some materials of containment, corrosive damage to others, and erosive-corrosive damage to still others. In this section corrosion is defined as any of these types of damage caused by the presence or circulation of aqueous slurries.

Purely erosive damage occurs when the material being attacked does not react chemically with the aqueous medium. Figure 1 illustrates the erosive damage that resulted from the circulation of thorium oxide slurry through gold and platinum orifice sections at a velocity of about 90 fps.¹ It is found by examination of such specimens that the attack is confined to the regions of highest shear and that there is no attack of the surfaces against which there is no turbulent or shearing flow.

Purely corrosive damage occurs when the material being attacked is unaffected by the erosive forces but is corroded by the aqueous phase of the slurry. The appearance of a sintered aluminum oxide orifice plate after exposure to circulating thorium oxide slurry at 300° C for 330 hr and a flow velocity of 70 fps indicated that the attack on all surfaces of the plate was uniform, with no preference for the high-velocity regions; even the inner edges of the orifice opening remained sharp.² This supports the conclusion that the aqueous phase was corroding all surfaces at a uniform rate (perhaps by dissolving the matrix) and that the erosive forces were too weak to attack the very hard aluminum oxide.

The attack of primary concern in the development of reactors utilizing aqueous slurries is, however, erosive-corrosive in nature, because these

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reactors usually utilize normally active metals which are protected from continuing reaction by an oxide film. When hard slurry particles damage or remove this film, the metal subsequently (perhaps immediately) reacts with the aqueous phase, the corrosive reaction being inhibited in only those areas that are covered with a stable, tenacious film that remains intact. Figures 2 and 3 illustrate this type of attack.³

Three different slurries--uranium trioxide, uranium dioxide, and thorium dioxide--have been considered for possible utilization in aqueous reactors. The UO_3 and ThO_2 slurries have been tested extensively at ORNL; they have been circulated through loops at elevated temperatures for many tens of thousands of hours. Tests of UO_2 slurries in forced-circulation loops at room temperature have been carried out for periods of up to 1000 hr.

The present state of knowledge concerning the theory of the attack of circulating slurries on their container materials is considered to be adequate for the design of the main components of reactor systems to be tested as part of the AEC reactor development program. Ultimate successful development of full-scale power reactors will require quantitative information, however, particularly with respect to the effects of high-level and long-term irradiation and the performance of special items of equipment such as valves, bearings, and running joints.

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PLATE 955-1

Fig. 2. Erosive-Corrosive Attack on 8-1/2-in.-dia.
Type 347 Stainless Steel Impeller

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PLATE 955-3

Fig. 3. Erosive-Corrosive Attack on Running Joints of
Type 347 Stainless Steel Impeller

MECHANISM OF SLURRY ATTACK

The erosive-corrosive attack on an active metal covered by a protective film of oxide may be subdivided into three stages:

1. impingement of slurry particles upon the surface,
2. damage to the surface as a result of the impingement,
3. reaction of the underlying metal with the aqueous phase.

At a given instant all three stages may, of course, be occurring and be in any degree of completion at various points on the metal surface. In subsequent paragraphs, however, the three stages will be discussed in the order indicated above.

Stage 1. Impingement of the Particles on a Surface *

When a particle-laden fluid impinges on a body, the fluid will be deflected around the body, whereas the particles, by virtue of their greater inertia, will tend to be collected on the surface of the body. The basic principles of impingement can be presented in terms of so-called "target" efficiencies. Target efficiency represents the fraction of particles in the fluid volume swept by the body which will impinge on the body. Thus, for flow around a cylinder, as shown in Fig. 4, all particles that are initially carried in the fluid between streamlines A and B will be collected on the body, and the target efficiency will be (X/D_b) . It has been shown^{4,5,6,7,8} that the target efficiency should be a function of the dimensionless group, $\frac{V_o D_p^2 (\rho_s - \rho)}{18 \mu D_b}$, where

*The first paragraph of this section is largely paraphrased from the article on Impingement Separators in section 15, Mechanical Separations, of the Chemical Engineers' Handbook, 3rd Edition.

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Fig. 4. Impingement Separation of Slurry Particles

V_o = average velocity of the particle-laden fluid

D_b = representative dimension or diameter of body impinged upon

D_p = diameter of particle

ρ_s = particle density

ρ = fluid density

μ = fluid viscosity.

For simple shapes the relationship between the separation number (as the dimensionless group is sometimes called--other times called the particle parameter) and the target efficiency (sometimes called the collection factor) can be derived from classical hydrodynamics; for more complex shapes, experimental determinations are required. While the relationships given by the above investigators for various collecting-body shapes are somewhat conflicting, the recent values reported by Langmuir and Blodgett,⁷ Fig. 5, are believed to be reliable. Although these relationships are derived for conditions of potential (streamline) flow around the body, they should hold closely even if the flow around the body is turbulent, since conditions on the up-stream side of the body should approach those of potential flow in any case. It should also be noted that these relationships should apply whether the body moves through the fluid or whether the fluid moves around the body, as long as V_o is taken as the relative velocity between the body and the bulk of the fluid.

Situations which are of practical importance for reactor design but which are difficult to analyze include flow through the shrouded vanes of a pump impeller, around sharp bends or tee joints in piping systems, at the entrance to heat-exchanger tubes, and around unsuspected weld-metal

Fig. 5. Target Efficiencies for Various Collecting-Body Shapes

obstructions. Bailey⁹ has attempted to analyze the erosion due to particle impingement upon bends in circular conduits, and his report emphasizes the limitations surrounding his conclusions. He pointed out that the interference of one slurry particle with another can markedly reduce the magnitude of the attack and called attention to the importance of the physical phenomena taking place during the disintegration of the material at the point of erosion (Stage 2 attack).

Although the mathematical analysis of practical situations is, indeed, very difficult, it appears possible to make use of the target-efficiency concept and the generalized separation number in such a way as to relate the extent of slurry attack to reactor design parameters under controlled conditions in which the impingement may be made more severe than is likely to be encountered in reactors. The relationships so obtained may then be used with confidence in the design of slurry reactor systems.

Stage 2. Damage to Surface

Whether an impinging particle will damage an inert-metal surface or an oxide film on top of an active-metal surface is a question which involves as many complications as the question of impingement itself. First of all there is the energy of the impinging impacts; this will be a function of how much energy remains in the particle after it fights its way along a compromise path between its original inertial path and the fluid flow lines. The relative hardness and elasticity of the surface film and the impinging particle will certainly be important; the mechanical properties of the metal underneath a very thin surface film may also be important. In that damage to the film may not occur unless the impinging particle hits hard enough to deform the underlying metal sufficiently to break the metal-film bond.

Finally, there is the question of the sharpness of the particle and thus the pressure involved at the impact. Bailey⁹ has discussed the observation of Rosenberg¹⁰ that sharp sand was about four times as erosive to steel (in an air blast) as larger, more rounded sand.

A jet-impingement test has been developed by McBride and Pattison for the laboratory, room-temperature, evaluation of the abrasive properties of slurries.¹¹ Typical apparatus for this test is shown in Fig. 6. The time for penetration of thin-metal shim stock by a jet of recirculated slurry is measured. In the absence of penetration within a reasonable time, a qualitative examination of the surface of the test plate permits an evaluation of the relative abrasiveness of the material. Good discrimination among slurries has been obtained with the use of this test. Table 1 illustrates the decided differences among different thorium oxides. Further indication of the sensitivity of the test is provided by tests with the more conventional standard abrasives, silicon carbide and aluminum oxide, as illustrated by the data in Table 2.

The conclusions drawn from these results were:

1. The penetration times showed deviations of less than 10% for a given combination of test conditions and metal thickness.
2. Halving the slurry concentration decreased the penetration rate by less than 20%.
3. Slurry homogeneity markedly affected the reproducibility of the test.
4. The abrasiveness of thorium oxide increased with increasing temperature of calcination. The increase in abrasiveness was attributed to the increase in gross particle size.

Damage to the metal surface is accompanied by corollary damage to the impinging particles, and it has been found that substantial degradation in

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Fig. 6. McBride-Pattison Jet-Impingement Test Apparatus

TABLE 1. ABRASIVE PROPERTIES OF THORIUM OXIDE SLURRIES

Abrasive Slurry Preparation	Thorium Concentration (g/liter)	Test-Plate Thickness (mils)	Duration of Test (min)	Results
Ames ThO ₂ - Seived through 325 mesh.	250-1000*	1.5**	22	Penetration
	250	1.5 (annealed)	32	Penetration
	250	1.5 (annealed)	41	Penetration
500° C ThO ₂ hydrolyzed for four days by treatment with water at 250° C.	250	1.5 (annealed)	60	No Penetration
900° C ThO ₂ , aged	250	5 (stainless steel)	2.2	Penetration
	250	10 (titanium)	3.9	Penetration
900° C ThO ₂ , fresh	250	5 (stainless steel)	3.3	Penetration
	250	10 (stainless steel)	6.6	Penetration
Th(OH) ₄ , hydrolyzed for more than 64 hr.	250	1.5 (annealed)***	150	No Effect
Th(OH) ₄ , hydrolyzed for 24 hr at 250° C.	250	1.5 (annealed)***	26	No Effect
ThO ₂ -ThF ₄ , F/Th = 0.5	250	1.5 (annealed)***	3	Penetration
ThO ₂ -ThF ₄ , F/Th = 0.9, hydrolyzed for 64 hr at 250° C with 500 psi CO ₂ .	217	1.5 (annealed)***	6	Penetration
UO ₃ ·H ₂ O, platelets	250-500*	1.5 **	85	No Effect

* Constant volume not maintained.

** Test-plate Knoop hardness number 213.

*** Test-plate Knoop hardness number 62.3.

TABLE 2. EXPERIMENTS FOR EVALUATION OF THE LABORATORY ABRASION TEST

Conditions: slurry volume, 20 ml
nitrogen pressure, 40 psi

Experiment No.*	Abrasive	Concentration (g/liter)	Stirring-Speed Setting	Test-Plate Thickness (mils)	Penetration Time (min)	Specific Penetration Rate (mils/min)
1 _a	800-mesh SiC	250 (Si)	70	5	2.0	2.5
1 _b	800-mesh SiC	250 (Si)	90	5	2.0	2.5
1 _c	800-mesh SiC	250 (Si)	90	25	10.5	2.4
2 _a	800-mesh SiC	125 (Si)	70	5	2.0	2.5
2 _b	800-mesh SiC	125 (Si)	100	5	2.6	1.92
2 _c	800-mesh SiC	125 (Si)	80	5	2.3	2.17
2 _d	800-mesh SiC	125 (Si)	80	25	12.3	2.04
3 _a	600-mesh Al ₂ O ₃	250 (Al)	90	5	2.4	2.08
3 _b	600-mesh Al ₂ O ₃	250 (Al)	70	5	2.0	2.5
3 _c	600-mesh Al ₂ O ₃	250 (Al)	90	25	14.3	1.75
4 _a	600-mesh Al ₂ O ₃	125 (Al)	90	5	3.0	1.66
4 _b	600-mesh Al ₂ O ₃	125 (Al)	90	5	2.9	1.72
4 _c	600-mesh Al ₂ O ₃	125 (Al)	90	25	17.6	1.43

*Subscripts a, b, c, d indicate that the abrasion tests were run consecutively using the same sample of slurry but a new test piece.

particle size is accomplished by recirculating slurries through regions of high impingement. Along with this, since the tendency for impingement and the damage from impingement are both reduced by reducing the particle size, is usually found a substantial decrease in slurry attack rate with time for recirculation studies. Such decreases were found in early Project studies of UO_2 and SiO_2 slurry attack¹² and have also been found in later Project studies of ThO_2 slurries, as discussed extensively in an analysis of the attack of circulating aqueous thorium oxide slurries on stainless steel systems.¹³ The replacement of degraded slurry with fresh slurry results in a return to the initial attack rates, with subsequent decline as the new batch of slurry is degraded. Thus the stage 2 attack is an attack on the circulating particles as well as an attack on the impingement surface. It has been proposed that precirculation of the slurry to degrade the larger particles can act as a protection against rapid attack in a more valuable system.

Stage 3. Corrosion Subsequent to Film Damage

Stage 3 occurs only when an active metal is attacked via the destruction of a normally protective film. In the absence of such a film the rate of this stage corresponds to the "bare-metal corrosion rate" of the metal in contact with the particular aqueous solution. For stainless steel, titanium, zirconium, and most other metals or alloys of interest as possible reactor materials, this rate is extremely high at the temperatures considered for reactor operation.

As discussed in the section on Corrosion by Aqueous Reactor Fuel Solutions, changes in the nature of the aqueous solution in contact with bare metal can profoundly alter the rate of bare-metal attack and also the total amount of metal consumed before the reaction is stifled by the reformation of a protective film. This "stifling factor" will certainly be important in determining the extent of corrosion following erosive slurry damage to a protective oxide film.

ATTACK BY URANIUM DIOXIDE AND QUARTZ SLURRIES

Studies of the attack of slurries carried out at room temperature in the early days of the Manhattan Project made use of small-scale circulation loops and a spinning-cylinder system.¹² Aqueous slurries of both UO_2 and quartz were tested. A brief description of the apparatus, summary of the experimental results, and discussion of the results may be found in the analysis of attack by circulating thorium oxide slurries already mentioned.¹³ The conclusions from the early experiments with UO_2 and quartz were:

1. Over a range of velocities from 20 to 40 fps the variation in attack was proportional to the second or third power of the velocity.*
2. Erosion rates varied markedly with the size of the slurry particle.
3. Erosion rates increased with slurry concentration.
4. The ratio of erosion rates for an 18/8 stainless steel vs aluminum was almost constant (for various conditions of velocity, etc.) and was independent of slurry concentration.

Further analysis of the data indicated that the attack of both slurries decreased, following very closely a parabolic relation with time; this was true for elapsed time after the addition of fresh slurry no matter how many additions were made, and it suggested to Hiskey that the decrease of attack rate with time is not caused by a change at the metal surface but by changes in the slurry. In this connection it was reported that a considerable degradation in particle size of the UO_2 was observed; after 60 hr about 90%

* No direct experimental support for this conclusion could be found in Hiskey's review of the subject,¹¹ nor in the original papers which were available, but the conclusion appears very reasonable in view of the relationship between velocity and impingement as indicated by the target efficiency and the results of ORNL slurry corrosion tests.

of the particles had been reduced to below $2\text{ }\mu$ in diameter (whereas the slurry initially had been screened to give 43 to $74\text{ }\mu$ particles). The parabolic relationship between attack rate and time appears reasonable from the point of view of the kinetics of particle degradation; if a first-order kinetic relationship holds, then the exponential display of attack vs time resembles closely a parabola for intermediate lengths of time. The presence of successive exponentials for various particle sizes would heighten the parabolic nature of the observed results.

Slurries of UO_2 have been considered for use as reactor fuels at elevated temperatures.¹⁴ Plans have been indicated for studying the erosion and corrosion of the reactor circuit under irradiation, but results have not yet become available.

ATTACK BY URANIUM TRIOXIDE SLURRIES

Consideration of uranium trioxide slurries as potential reactor fuels has led to study of their corrosive properties when circulated through reactor components. It has been reported that the attack of stainless steel components by circulating uranium trioxide slurries at 250°C is essentially nil, even on pump impellers with a tip velocity of 120 fps.¹⁵ This conclusion was supported by studies carried out in the mechanical systems shown in Figs. 7 and 8, in which the slurries were circulated under a variety of conditions for thousands of hours.¹⁶ The absence of attack on stainless steel components was established by chemical analysis of slurry samples withdrawn after operation and by visual inspection of the pump impeller, orifice-type flow restrictor, and piping system after each test. The maximum flow velocity was obtained in the impeller; that through the orifice plate was ~ 83 fps, and that through the piping was ~ 20 fps.

The lack of attack by circulating uranium trioxide slurries under conditions in which impingement certainly occurred is attributed to the relative softness of the uranium trioxide particles and thus to their inability to damage the oxide film on the stainless steel.

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Fig. 7. Schematic Flow Sheet for S Loop

Fig. 8. Schematic Flow Sheet for T Loop

ATTACK BY THORIUM OXIDE SLURRIES

Analysis of Circulating-Loop Results

Many thousands of hours of circulating thorium oxide slurries at temperatures of 250 to 300° C in stainless steel test loops have provided information which has been interpreted in terms of the effects of several variables upon the attack by the slurry.¹³ Schematic flowsheets for two of the test loops used in these studies were already presented as Figs. 7 and 8. Details of the tests may be found in the ORNL Homogeneous Reactor Project Quarterly Progress Reports.¹⁶ The tests covered a range of conditions including thorium concentration (200 to 1000 g per kilogram of H₂O), temperature (250 to 300° C), linear fluid flow velocity in pipes (20 to 25 fps), and local velocities in regions of excessive turbulence (up to 120 fps). A total system pressure of ~2000 psi was used in most experiments. Figures 9 and 10 illustrate the undamaged condition of the loop piping after 2000 hr of slurry circulation testing.

Samples of slurry were withdrawn from the loops at periodic intervals and analyzed for corrosion products. The principal corrosion product found was iron; analysis showed that essentially all the iron followed the solid phase (ThO₂) and that no substantial amount remained in the liquid phase. Since the amount of thorium being circulated often varied during a test, the data for slurry attack may conveniently be represented as changes in the Fe:Th ratio with time. The changes in pH and in the Cr:Th ratio are also frequently of interest. A plot of data obtained during a run is presented as Fig. 11. It will be noted that the pH and the Cr:Th ratio varied in erratic fashion but the Fe:Th ratio increased rapidly at the beginning of the

Fig. 9. Section of Defilmed Stainless Steel Piping from Loop T

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Fig. 10. Enlarged Section of T-Loop Piping Showing Welds and Grinding-Tool Marks

Fig. 11. Changes in Slurry Properties During Test Run S-49

run and then increased much more slowly as the run progressed; two additions of fresh thorium oxide were followed by short periods of rapid increase in this ratio, again followed by longer periods of much slower increase as the attack rate decreased. This was the longest run of the reported series; others were stopped shortly after the attack rate began to level off.

Arithmetic plots of the changes in the Fe:Th ratio with time for 39 circulation loop tests are presented in Figs. 12-20. The operating conditions for these tests are shown in Table 3. Tests of less than 100 hr in duration were omitted from the tabulation.

The conclusions pertaining to attack rate by thorium oxide slurries that were drawn from these tests are:

1. The principal attack on stainless steel occurred at regions of high velocity and/or high shear.
2. The presence of oxygen decreased the attack rate.
3. Increasing the oxide calcination temperature in the range of 650-950° C increased the initial attack rate.
4. The tendency for a given slurry to attack stainless steel was diminished if the slurry was pretreated by pumping in a circulation loop at elevated temperature.
5. Apparently the attack on stainless steel was less severe at loop temperatures of 300° C than at 250° C.
6. It appeared desirable to keep the sulfate concentration below 2000 ppm in runs at 300° C in order to prevent high rates of attack resulting from the low pH and high acidity due to the hydrolysis of the sulfate.

In an effort to provide an objective interpretation of the attack-rate data, the quantities listed in Table 4 were considered. It was realized that

Fig. 12. Arithmetic Plot of Slurry Attack During Test Runs S-41, S-43, S-44, and S-47

Fig. 13. Arithmetic Plot of Slurry Attack During Test Runs S-49, S-53, S-54, and S-58

Fig. 14. Arithmetic Plot of Slurry Attack During Test Runs S-51, S-52, and S-55

Fig. 15. Arithmetic Plot of Slurry Attack During Test Runs S-59, S-64, S-65, and S-66

Fig. 16. Arithmetic Plot of Slurry Attack During Test Runs S-67, S-68, S-69, and S-70

Fig. 17. Arithmetic Plot of Slurry Attack During
Test Runs S-71, S-40, S-45, and S-46

Fig. 18. Arithmetic Plot of Slurry Attack During Test Runs T-20, T-37, T-39, and T-43

Fig. 19. Arithmetic Plot of Slurry Attack During Test Runs T-46, T-47, T-48, and T-51

Fig. 20. Arithmetic Plot of Slurry Attack During
Test Runs T-53, T-54, and T-59

TABLE 3. OPERATING CONDITIONS

Run No.	Duration of Test (hr)	Loop Temp. (°C)	Thorium Oxide			pH ^a	SO ₄ ⁼ Conc. (ppm) ^b	O ₂ Conc. (ppm)	Reason for Shutdown
			Source	Firing Temp. (°C)	Av. Conc. (g/kg H ₂ O)				
S-40	729	250	Ames	650	1060	5.2	NA ^c	None	NR ^d
S-41	231	250	Th(OH) ₄	-	170	2.4	NA	None	Leak around thermal spacer
S-43	183	250	Ames	650	886	5.3	8100 ^e	None	Leaks and front bearing worn
S-44	143	250	Formate	650	370	5.0	6300 ^e	None	NR
S-45	140	250	Formate	650	400	6.4	NA	None	NR
S-46	118	250	Ames	650	400	6.7	NA	None	NR
S-47	143	250	Formate	650	376	5.4	6500 ^e	None	NR
S-49	1459	250	Formate	650	400	4.4	6800 ^e	400	NR
S-51	328	250	Formate	650	722	4.4	NA	500	NR
S-52	189	250	Lindsay No. 7	950	1070	8.7	NA	40	Bearing wear
S-53	307	250	XBI	650	534	5.0	4770	500	NR
S-54	318	250	Lindsay No. 12	650	500	5.9	3400	400	NR
S-55	309	250	Lindsay No. 13	650	1200	5.9	5600	300	Lost O ₂ at ~240 hr
S-58	234	250	D-12	650	378	5.2	1300	500	NR
S-59	335	250	D-12	650	486	5.6	1300	500	Teflon spacers disintegrated; therefore, F ⁻ in system
S-64	375	250	D-15	650	443	5.8	400	500	NR
S-65	216	250	D-26	800	611	8.3	<100	500	Sample valve gave way
S-66	187	250	D-26	800	516	6.2	560	500	NR
S-67	308	300	D-26	800	623	4.9	1600	500	NR
S-68	284	275	D-26	800	560	5.2	1450	500	NR
S-69A	288	300	D-26	800	894	6.0	600	500	NR
S-70	310	250	D-31	800	1080	4.7	3000	500	NR
S-71	274	275	D-31	800	955	5.6	2700	500	Bearing failure

^aThe pH represents an average value taken after the attack rate leveled off, or in those cases where the attack did not level off it represents the value from the last sample.

^bppm based on slurry.

^cNot available.

^dNo reason given for shutdown.

^eEstimated from analysis of starting material.

TABLE 3. OPERATING CONDITIONS
(CONTINUED)

Run No.	Duration of Test (hr)	Loop Temp. (°C)	Thorium Oxide			pH ^a	SO ₄ ⁼ Conc. (ppm) ^b	O ₂ Conc. (ppm)	Reason for Shutdown
			Source	Firing Temp. (°C)	Av. Conc. (g/kg H ₂ O)				
T-20	784	250	Ames	650	280	5.6	2700 ^e	None	Bearing wear
T-35	141	250	Lindsay No. 3	950	480	8.8	100 ^e	None	NR
T-37	140	250	Lindsay No. 1	950	420	7.6	100 ^e	None	NR
T-38	618	250	Lindsay No. 3	950	473	6.4	1800	1000	Impeller rubbing against front weld pads
T-39	188	250	Lindsay No. 3	950	240	5.7	100 ^e	1000	NR
T-43	181	250	Lindsay No. 3	950	230	5.2	100 ^e	500	Sample valve locked open
T-45	581	250	Lindsay No. 7	950	266	5.2	520	700	NR
T-46	139	250	Lindsay No. 12	650	290	9.2	200	600	Pump kept shutting off because of overload
T-47	257	250	From S-53	650	220	5.3	2200	900	NR
T-48	298	250	Lindsay No. 11	650	600	5.7	<400	900	NR
T-49	401	250	H6B	650	480	9.0	3700	100	NR
T-51	136	250	D-15	650	500	8.1	<400	900	NR
T-53	167	250	D-26	800	400	8.1	<400	900	NR
T-54	308	250	From T-52	650	380	7.0	<400	900	Pump wattmeter running too high
T-56	277	250	D-26	800	930	6.1	None	900	Impeller rubbed front weld pads
T-59	237	250	NA	800	800	5.6	1500	900	NR

^aThe pH represents an average value taken after the attack rate leveled off, or in those cases where the attack did not level off it represents the value from the last sample.

^bppm based on slurry.

^cNot available.

^dNo reason given for shutdown.

^eEstimated from analysis of starting material.

TABLE 4. SLURRY ATTACK DATA

Run No.	Y_1	Y_2	X_1	X_2	X_3	X_4	X_5	X_6
S-43	18	47	53	89	65	81	0	0
S-44	16	35	50	37	65	63	0	13
S-47	23	46	54	38	65	65	0	13
S-49	2	22	44	40	65	68	4	8
S-53	8	24	50	53	65	48	5	13
S-54	12	34	59	50	65	34	4	13
S-58	16	36	52	38	65	13	5	13
S-59	14	35	56	49	65	13	5	13
S-64	5	36	58	44	65	4	5	13
S-65	20	42	83	61	80	1	5	13
S-66	19	44	62	52	80	6	5	13
S-67	17	42	49	62	80	16	5	8
S-68	17	41	52	56	80	15	5	8
S-69A	16	42	60	89	80	6	5	0
S-70	15	37	47	108	80	30	5	0
S-71	10	38	56	96	80	27	5	0
T-20	15	41	56	28	65	27	0	10
T-37	15	39	76	42	95	1	0	8
T-39	16	39	57	24	95	1	10	13
T-43	14	43	52	23	95	1	5	13
T-46	17	40	92	29	65	2	6	13
T-47	1	13	53	22	65	22	9	13
T-48	12	31	57	60	65	35	9	8
T-51	15	39	81	50	65	4	9	8
T-53	16	37	81	40	80	4	9	8
T-54	12	24	70	38	65	4	9	8
T-59	15	41	56	80	80	15	9	0

$$Y_1 = [\text{Log}(10)(\text{linear attack rate})(\text{av ThO}_2 \text{ conc.})] \times 10.$$

$$Y_2 = [\text{Log}(\text{parabolic attack rate})(\text{av ThO}_2 \text{ conc.})^2] \times 10.$$

$$X_1 = (\text{pH})(10).$$

$$X_2 = (\text{ThO}_2 \text{ concentration}), \text{ g/10 kg.}$$

$$X_3 = (\text{ThO}_2 \text{ calcination temperature}), ^\circ\text{C}/10.$$

$$X_4 = (\text{Sulfate concentration}), \text{ ppm}/100.$$

$$X_5 = (\text{Oxygen concentration}), \text{ ppm}/1000.$$

$$X_6 = [\text{Impeller diameter, in.} - 7] \times 10.$$

the results could have only limited validity because factors now known to be important in attack had not been controlled during the experiments. For example, the attack rate is known to be strongly dependent on the flow velocity and upon the particle-size distribution, but only average attack rates for the over-all system were measured and particle-size data for the tests were only fragmentary. In the tests the volumetric flow rate was essentially constant with varying combinations of impeller diameter and orifice-plate diameter. The effects of these combinations upon bulk fluid flow rate, velocity in loop piping, maximum venturi velocity, and orifice velocity are shown in Table 5. Because the arithmetic plots of attack versus time contain pronounced curvature at the beginning of runs, and because many of the tests were terminated shortly after the rate had decreased sharply but before a long-term attack rate had become well established, it was believed that a useful representation of more of the data might be had by consideration of a plot of the square of the attack against time, a device frequently useful in corrosion analysis. Although this method of presentation implies a specific mechanism, when a straight-line relation is found it uses all the data in determining the attack, and the slope is independent of the duration of the test. Parabolic attack plots for the data from S loop are presented as Figs. 21 through 26.

It should be emphasized that both the linear and parabolic attack rates are purely relative and that no absolute rates can be determined from these tests. The rates do represent the gross attack on S and T loops at the particular conditions under which they were run, with the further qualification that they probably represent loss of material from high-velocity and high-shear regions such as the pump impellers, impeller hubs, volutes,

TABLE 5. EFFECT OF IMPELLER DIAMETER AND ORIFICE
DIAMETER ON VELOCITY IN S AND T LOOPS

Impeller Diameter, in.	8-1/4	7-3/4	7	7
Bulk fluid flow rate, gpm	118	110	100	
Loop velocity, fps	22.8	21.2	19.2	
Maximum venturi velocity, fps	54.8	51.1	46.4	
Orifice diameter, in.	3/4			1
Orifice velocity, fps	83	80	71	41

Fig. 21. Parabolic Plot of Slurry Attack During
Test Runs S-41, S-43, S-47, and S-49

Fig. 22. Parabolic Plot of Slurry Attack During Test Runs S-40, S-45, and S-44

Fig. 23. Parabolic Plot of Slurry Attack During Test Runs S-51, S-52, S-55, and S-46

Fig. 24. Parabolic Plot of Slurry Attack During Test Runs S-53, S-54, S-58, and S-59

Fig. 25. Parabolic Plot of Slurry Attack During Test Runs S-64, S-65, S-66, and S-67

Fig. 26. Parabolic Plot of Slurry Attack During Test Runs S-68, S-69, S-70, and S-71

etc., as well as the orifice plates.

The data and the definition of the units for 27 tests were presented in Table 4. Seven S-loop tests, (S-40, 41, 45, 46, 51, 52, 55) which were characterized by quite linear attack rates on the arithmetic plots, were excluded from the parabolic relationship analysis. The attack rates from both linear and parabolic plots are given; the thorium concentration was removed from the rates by multiplying by the average concentration and the average concentration squared, respectively.

The mean values of the parabolic attack rates found with S and T loops were 37.6 and 35.2, respectively, and the corresponding ranges were 22-47 and 13-43. These results indicate that there was no significant difference between the two loops, and so their combined data were used in the correlation.

It was then assumed that the $\log_{10}$ of the attack rate might, to a first approximation be related linearly to five different variables: (1) the pH, (2) the ThO_2 concentration, (3) the ThO_2 calcination temperature, (4) the sulfate concentration, and (5) the oxygen concentration. Thus the attack rate could be expressed by a regression equation of the form

$$y = b_{y.12345} + b_{y1.2345} X_1 + b_{y2.1345} X_2 + b_{y3.1245} X_3 + b_{y4.1235} X_4 + b_{y5.1234} X_5 \quad (1)$$

where X_1, X_2, X_3, X_4, X_5 are variables

y is $\log_{10}$ (attack rate)

$b_{y.12345}$ is the mean value of y minus the contribution to y of the variables X_1, X_2, X_3, X_4 and X_5 at their means.

$b_{y1.2345}$ measures the rate of increasing y with increasing X_1 ; X_2, X_3, X_4 and X_5 being constant.

$b_{y2.1345}$ measures the rate of increasing y with increasing X_2 ; X_1, X_3, X_4 and X_5 being constant.

etc.

The results of the analysis are given in Tables 6, 7, and 8. The total correlation was better when based on the parabolic attack rate than when based on the linear attack rate, but this may have been due to the selection of data. Analysis based on both sets of attack rate data indicated that the effect of oxygen had the highest significance, the effect of the sulfate ion concentration had very low significance, and that pH, thorium concentration, and calcination temperature were of intermediate significance. The attack rate is decreased as the oxygen concentration is increased; it is increased as the calcination temperature, thorium concentration, and pH are increased. The significance of the effects due to oxygen, thorium concentration, and calcination temperature was greater when the analysis was based on the parabolic attack rate data.

The amount of unexplained residual variance in the analysis was sufficiently large that an attempt was made to search for its source. The impeller diameter was tested as a possible source, together with the known most significant effects--thorium concentration and oxygen concentration--in a separate analysis; the regression coefficient for the effect of changes in the impeller diameter was, however, small and of very low significance. Further data concerning impeller weight loss, orifice weight loss, and the results of a material balance on the system are shown in Table 9. The difference between the ratio of impeller weight loss to orifice weight loss for systems in which both components were of stainless steel and that for systems in which both were of titanium was tested and found to be without statistical significance, i.e., mean of 18.6 and standard deviation of 2.0 for stainless steel vs. mean of 19.5 and standard deviation of 5.4 for titanium. It has subsequently been found that particle size is a very important determiner of slurry corrosion, and it seems

TABLE 6. RANGE OF VARIABLES IN CORRELATION

	Variable	Min.	Mean	Max.
$\{\text{Log [10 x linear attack rate]}\} \times 10$	y_1	1	13.9	23
$\{\text{Log [parabolic attack rate]}\} \times 10$	y_2	13	36.6	47
pH x 10	x_1	44	59.9	92
Th conc., g/10 kg	x_2	22	51.8	108
Calcination temp., °C/10	x_3	65	73.3	95
$\text{SO}_4^{=}$ conc., ppm/100	x_4	1	22.4	81
O_2 conc., ppm/1000	x_5	0	5.1	10

TABLE 7. RESULTS OF STATISTICAL ANALYSIS

Coefficient*	Linear Attack Rate		Parabolic Attack Rate	
	Coefficient	Significance Level**	Coefficient	Significance Level**
$b_{y.12345}$	-5.38		12.78	
$b_{y1.2345}$	0.17 ± 0.18	0.082	0.16 ± 0.25	0.228
$b_{y2.1345}$	0.04 ± 0.08	0.361	0.10 ± 0.13	0.083
$b_{y3.1245}$	0.14 ± 0.22	0.219	0.24 ± 0.30	0.127
$b_{y4.1235}$	0.005 ± 0.13	>0.50	-0.06 ± 0.18	0.50
$b_{y5.1234}$	-0.62 ± 0.71	0.097	-1.38 ± 0.97	0.01
$b_{y2.1345} + b_{y4.1235}$		70.20		-
$b_{y1.2345} + b_{y4.1235}$		-		0.155
Total correlation		0.10		0.02

*See Eq. 1, page 46.

**It is a matter of judgment what probability level is taken as being significant. For many purposes the 0.05 level is accepted, but it must be realized that if a long series of possible effects were tested, none of which actually existed, in 1 of every 20 tests it would be incorrectly asserted that the effect did exist. If a higher significance level, say 0.01 is chosen, then the frequency with which this type of error is made is reduced, but the frequency with which effects which really exist are ignored is increased. A practical compromise is to regard effects at about the 0.05 level as well worth following up. For presentation of final results and conclusions, a higher level is preferred. This can be obtained, if the effect really exists, by making more observations. It must also be recognized that a significance level of 0.05 or less does not prove the lack of true correlation but merely shows that the particular data considered remain uncorrelated.

TABLE 8. COMPARISON OF VALUES OF ATTACK RATE CALCULATED FROM
STATISTICAL RESULTS WITH THOSE EXPERIMENTALLY MEASURED

Run No.	Linear Attack Rate		Parabolic Attack Rate	
	$Y_1(\text{exp})$	$Y_1(\text{calc})$	$Y_2(\text{exp})$	$Y_2(\text{calc})$
S-43	18	16.53	47	40.84
S-44	16	13.91	35	36.07
S-47	23	14.64	46	36.68
S-49	2	10.54	22	29.63
S-53	8	11.34	24	31.77
S-54	12	13.31	34	35.11
S-58	16	10.92	36	32.68
S-59	14	12.03	35	34.45
S-64	5	12.13	36	34.80
S-65	20	19.11	42	44.23
S-66	19	15.21	44	39.71
S-67	17	13.43	42	38.10
S-68	17	13.71	41	28.01
S-69A	16	16.30	42	43.24
S-70	15	14.95	37	41.71
S-71	10	16.00	38	42.05
T-20	15	14.40	41	38.29
T-37	15	22.36	39	51.62
T-39	16	12.21	39	33.01
T-43	14	14.43	43	39.01
T-46	17	16.71	40	37.30
T-47	1	8.03	13	25.11
T-48	12	10.25	31	28.89
T-51	15	13.79	39	33.51
T-53	16	15.48	37	36.04
T-54	12	11.45	24	30.54
T-59	15	12.83	41	35.62

TABLE 9. MATERIAL BALANCE ON S AND T LOOP RUNS

Run No.	Impeller		Orifice Wt. Loss (g)	Total Fe on ThO ₂ (g)	Impeller Wt. Loss Orifice Wt. Loss	Impeller Wt. Loss x 0.74 Total Fe on ThO ₂
	Material	Wt. Loss (g)				
S-40	SS	38	0.81	85	47	0.33
S-41	SS	7	0.25	33	28	0.16
S-43	SS	52	0.26	40	200	0.96
S-44	SS	1	0.24	7.0	4.2	0.10
S-45	SS	7	0.18	9.3	39	0.52
S-46	SS	8	0.08	13	100	0.46
S-47	SS	1	0.08	12	13	0.10
S-49	SS	2	+0.06	8.7	-	0.17
S-51	SS	+2	0.04	7.6	-	-
S-52	SS	17	0.70	67	24	0.18
S-53	Ti	0.5	0.02 ^b	2	25	-
S-54	Ti	2	0.07 ^b	11	28	-
S-55	SS	17	0.37	35	46	0.36
S-58	Ti	4	0.12	23	33	-
S-59	Ti	3	0.24 ^c	12	13	-
S-64	Ti	3	0.88 ^c	10	3.4	-
S-65	Ti	13	0.92 ^c	23	14	-
S-66	SS	15	(a) ^c	26	-	0.43
S-67	SS	18	1.06 ^d	16	17	0.83
S-68	SS	8	(a) ^d	17	-	0.35
S-69A	SS	19	1.48	25	13	0.55
S-70	SS	19	0.06	15	-	-
S-71	SS		+0.86	48	-	-
T-20	SS	46	0.47	162	100	0.21
T-35	SS	23	0.84	26	28	0.65
T-37	SS	5	0.24	9.0	21	0.41
T-38	SS	21	0.37	20	57	0.78
T-39	SS	10	0.30	14	33	1.03
T-43	SS	14	0.55	7.1	25	1.48
T-45	SS	17	0.71	36	24	0.35
T-46	SS	4	0.28	13	14	0.23
T-47	SS	~0	0.25	2.4	-	-
T-48	SS	4	0.16 ^b	6.1	25	0.48
T-49	SS	15	2.28	22	6.6	0.50
T-51	SS	6	0.30	12	20	0.37
T-53	SS	(a)	(a)	14	-	-
T-54	SS	(a)	1.27	12	-	-
T-56	SS	(a)	(a)	27	-	-
T-59	SS	(a)	(a)	-	-	-

a - not recorded.

b - Ti orifice plate.

c - quartered orifice plate; weight loss corresponds to that for full size orifice plate of same material as impeller.

d - Pt orifice plate.

likely that failure to appreciate this effect was responsible for most of the unexplained variance in the analysis of the S and T loop tests. Since it has also been subsequently demonstrated that the degradation of large particles is most rapid at the beginning of tests with fresh slurry it also seems likely that the parabolic relationship between attack rate and time which was detected may have been due to the degradation of larger particles into smaller particles. This is consistent with the previously presented analysis of impingement as a cause of corrosion.

Detailed Study of Factors Affecting Slurry Corrosion

The Reactor Materials Research Section of the Homogeneous Reactor Project has undertaken a study involving the use of 100-gpm circulating loops,¹⁷ such as that illustrated in Fig. 27, and the toroid apparatus developed for the study of corrosion in aqueous uranyl sulfate solutions,¹⁸ illustrated in Fig. 28.

Correlation of the results of circulating-loop studies of slurry attack on type 347 stainless steel pins at 300° C indicated a dependence on the square of the velocity and the first power of the concentration.¹⁹ The data for other materials appeared to be generally consistent with this behavior, and the correlation has been used as a basis for comparing various materials by adjusting the observed rates under a variety of conditions to a common basis (20 fps and 1000 g of thorium per kilogram of water). Table 10 indicates the comparative corrosion rates of various materials after adjustment. The corrosion rates of stainless steels exhibited a moderate temperature effect, increasing by a factor of 1.7 on going from 250 to 300° C. Platinum exhibited a similar temperature effect and a stronger dependence on concentration and velocity. Gold, platinum, and the Stellites appeared to be sensitive to

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Fig. 27. Schematic Diagram - Slurry Circulation Loop CS

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Fig. 28. Toroid Corrosion Test Apparatus

TABLE 10. COMPARATIVE CORROSION RATES OF VARIOUS MATERIALS
BY CIRCULATING THORIUM OXIDE SLURRY* AT 300° C

Material	Adjusted Corrosion Pin Attack Rate (mpy)
Platinum	2
Gold	2
Zircaloy-2	3
Titanium and alloys	4
Type 347 and other stainless steel	7
Nickel	12
Inconels	24
Stellites	14-40
Aluminum oxide	280
Graphitar; $\text{ThO}_2 + 0.5\% \text{CaO}$	Consumed

*Adjusted to 20 fps and 1000 g. of thorium per kilogram of H_2O .

particular batches of oxide, and the Stellites appeared to be undergoing attack by water alone as well as by the flowing slurry. Table 11²⁰ presents comparative corrosion rates at 300° C for various metals exposed to circulating slurry. The influences of velocity, particle size distribution, and slurry preparation are seen to be important. The rapid degradation of particles greater than 3 microns in diameter to smaller particles is clearly shown.

Applying the analysis of impingement presented previously to the case of toroids containing cylindrical pins suggested that, under the conditions of the tests, particles below 3 μ in diameter would not strike the surface of the test pins.¹³ The relevant portion of data obtained in a study of the effects of particle-size distribution and calcination upon slurry corrosion are presented as Table 12.²¹ As can be seen, upon reducing the weight fraction of particles greater than 3 μ in diameter from $\sim 40\%$ to 4-15%, the attack rate was reduced by factors of 6 to 10. At the same time it appears that, for slurries having similar particle-size distributions, the higher calcination temperatures were associated with higher corrosion rates. This may be attributed to the less frangible nature of the more highly calcined products, which are consequently more highly sintered. The agreement between the expected and the observed relationship of particle size to slurry corrosion encourages the further application of theoretical investigations to the determination of the form of the functional relationship between the various slurry parameters and the attack rate.

Further studies of slurry corrosion have been carried out in loops and in toroids,^{22, 23, 24} and the present conclusions concerning slurry corrosion can be summarized:

1. Degradation of thorium oxide particles is generally observed upon circulation.

TABLE 11 COMPARATIVE CORROSION RATES AT 300°C

Run CS-25 (ThO ₂ Batch LO-2)				Run CS-27 (ThO ₂ Batch LO-2)			Run CS-28-29 (ThO ₂ Batch LO-2)			Run CS-31 (ThO ₂ Batch LO-14)		
Corrosion Rate, mpy												
	11 fps	21 fps	41 fps	10 fps	22 fps	41 fps	13 fps	22 fps	43 fps	13 fps	25 fps	41 fps
Austenitic SS	2-4	5-9	18-23	1	2-4	6-9	0.5	0.5	2-4	1-2	4-6	10-12
Gold	0.4	0.4	4	0.1	0.1	0.6	0.03	0.08	0.90	0.1	1	2
Titanium and alloys	1	3-5	7-11	0.2	2	6	0.1	1.0	4-5	2	4	3-6
Zircaloy-2	0.1	1	6	0.1	0.1	2	<0.1	<0.1	1	<0.3	4	13
Mild steel	150	165	205	11	13	24	21	21	21	40	42	48
Average loop corrosion rate, mpy												
Duration of test, hr												
Average circulating concentration (g of Th per kg of H ₂ O)												
pH of slurry												
Additive												
Average Sedimentation Particle Size, wt %												
Approximate Circulation Time (hr)	> 3 μ	1-3 μ	< 1 μ	> 3 μ	1-3 μ	< 1 μ	> 3 μ	1-3 μ	< 1 μ	Sedimentation particle size not available; microscopic examination revealed formation of spheroid agglomerates of approximately 6- to 20-μ diameter formed during circulation		
0	48	27	25	59	26	15	3	32	65			
5	8	25	67									
20	7	20	73	7	26	67						
30	5	21	74	7	25	69	7	28	65			
50	2	30	67	7	17	76	2	22	76			
150				8	36	56	7	42	51			
200				5	26	69						
300				3	32	65	2	21	77			

TABLE 12. EFFECT OF PARTICLE SIZE ON ATTACK IN TOROID TESTS

ThO ₂ Treatment	Particle Size (wt. %)				Av. Pin Corrosion Rate (mdd)			Type 347 SS Toroid Corrosion Rate (mdd)
	0-1 μ	1-3 μ	3-10 μ	>10 μ	Type 347 SS	Ti-75A	Zr-2	
D-17 as received	23	36	26	15	65	5.4	6.7	3.8
D-17-1000 (D-17 calcined 6 hr at 1000° C)	20	40	28	12	177,161	30	7.0	31
CS-4 (D-17 circulated in 100A loop CS)	87	9	2	2	10,10.5	4.6	1.7	1.6
CS-4-1000 (CS-4 calcined 6 hr at 1000° C)	70	15	8	7	16,18	15	1.8	3.2

Slurry concentration: 1000 g of Th per kilogram of H₂O.
 Relative velocity: 26 fps
 Temperature: 247-255° C
 Oxygen pressure: 90 psi (at 250° C)
 Duration of test: 208 hr

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2. The conditions of circulation and of oxide preparation (velocity, turbulence, calcination temperature) determine the extent of particle degradation.

3. Particle size distribution affects attack. Materials show best resistance to attack when the thorium oxide particles are small.

4. High calcination temperatures and small sized particles (even with high temperature) lead to relatively low attack.

5. With oxides of small particle size (originally or finally) attack depends on run duration, velocity, concentration, temperature, atmosphere, and additives.

6. Fairly good agreement in results between toroids and loops is noted, after making proportional correction for reduced contact time due to slug flow through the toroids.

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19. ORNL 2057, page 89.

20. ORNL 2222, page 85.

21. ORNL 1943, page 147.

22. ORNL 2148, Homogeneous Reactor Project Quarterly Progress Report, July 31, 1956, p. 86 ff.

23. ORNL 2272, Homogeneous Reactor Project Quarterly Progress Report, January 31, 1957, p. 90 ff.

24. ORNL CF-56-7-51, HRP Dynamic Slurry Corrosion Studies, July 31, 1956, p. 26-39.

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