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SUBJECT: HRP Dynamic Corrosion Studies (In-Pile Development) Summary of Runs HT-20, HT-21, and HT-22 (Thorium Oxide Slurry)

TO: H. C. Savage

FROM: D. W. Hubbard DWH

I. Summary

Runs HT-20, - 21, and - 22 were made to determine the handling characteristics of a slurry containing thoria calcined at 1600°C and to evaluate the operation of the 5-gpm slurry loop using this slurry. The low velocity core section was removed from the loop prior to these runs.

Thorium oxide slurry at ~ 500 g/kg H_2O was circulated for 192 hr during run HT-21 with no operating difficulty. Run HT-22 consisted of an additional 524 hr of trouble-free operation circulating thorium oxide slurry of the same concentration. After 524 hr, run HT-22, the pump became noisy; and on disassembly it was seen that considerable wear had occurred on the thrust surfaces of the front bearing and journal. This was presumably due to improper thrust balance of the pump.

It can be concluded that the 1600°C-calcined, 0.7 μ mean particle size, thoria slurry can be circulated in a 5-gpm loop, and that the improved operation of the 5-gpm loop is due to the improved handling properties of the slurry and elimination of the low velocity core section since previous attempts to circulate slurries of different character were unsuccessful.(1)

DISCLAIMER

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II. Loop Design

When it was decided to resume operation of the 5-gpm loop,[1] after it had been idle for nearly a year, some minor changes to the loop were made. Figure 1 shows schematically the present appearance of the loop.

Altering the pressurizer for full flow through a side inlet was one change made. The full stream of fluid now flows into the side of the pressurizer and out the bottom. All other lines on the pressurizer were removed except the vapor line to the condenser and the condensate return line.

The standard in-pile type of core body and sample holder was replaced by a hairpin bend of 3/8-in. schedule-40 stainless steel (type 347) pipe. A drain line was welded to the bottom of this bend so that the entire loop could be drained easily. In previous tests, liquid remained in some of the lower parts of the loop in spite of all efforts to remove it.

A venturi was installed in the main stream for measurement of flow. This is the only flow restrictor now in the system, all the orifice plates used previously having been removed.

The pump used was modified so that it is now equivalent to the standard 5-gpm in-pile double-bearing pump with aluminum oxide bearings and journal bushings. A 110-v, single-phase stator with class A windings was used.

III. Run HT-20

Run HT-20 was made to clean the loop, make flow measurements, and check the operation of all parts of the loop in preparation for slurry tests. A 3% trisodium phosphate solution was circulated in the loop for 4 hr at 100°C with 60 psi helium overpressure. The loop was then rinsed with distilled water and charged with a 5% nitric acid solution. After 22 hr of operation at 100°C and 60 psi helium pressure, a leak developed at the pump scroll gasket. This was repaired, and a second nitric acid run was made under the same conditions for 24 hr. Following the nitric acid treatment, the loop was again rinsed with distilled water and charged with distilled water. The flow was measured as 7.1 gpm at room temperature with an oxygen overpressure of 60 psi. The heaters, condensate system, and slurry addition system were checked for proper operation with the loop at ~ 250°C and ~ 600 psi total pressure (60 psi oxygen pressure added at room temperature). The total operating time for the distilled-water run was 8 hr. During this

time, the loop operated in a normal fashion except that the pump temperature was 85°C compared to a normal temperature of 60°C usually observed for this type of pump.

IV. Run HT-21

Run HT-21 was made to determine whether the loop would satisfactorily circulate thorium oxide slurry. The slurry used was the same as that used in 100A loop BS for runs BS-17[2] and BS-18. It is a mixture of thoria from batches LO-17 and TO-10-1000 recalcined to 1600°C and having a mean particle size of 0.7 μ. The run was started by charging the loop system with 1788 ml of distilled water. This included filling the pressurizer, the condensate tank, and the slurry addition tank. Enough oxygen was added to the loop to produce a pressure of 90 psi at room temperature. The flow through the loop was assumed to be the same as measured in run HT-20 -- 7.1 gpm. The loop temperature was brought to ~ 250°C, and the loop was operated for 51 hr while final operational checks were made. At this temperature, the total pressure was 670 psi.

After 41 hr of operation, the loop was charged with approximately 690 g of thorium oxide contained in a dense slurry. This dense slurry was placed in the slurry addition tank and pumped into the loop. The volume of the slurry in the addition tank was replaced by an equal volume of condensate from the loop, so that the fluid volume in the loop remained essentially the same before and after the addition. No difficulty was noted during the addition of the slurry, and inspection of the addition tank after the loading had been completed showed that all the thorium oxide has been transferred to the loop. For some reason, the pump temperature decreased markedly, from 85°C to 60°C, after the slurry addition.

A considerable amount of instrument trouble during the run made it necessary to shut down for adjustments. Two such shutdowns were made, one of 43 hr duration after 144 hr of slurry operation and another of 65 hr duration after 174 hr of slurry operation. When the loop was restarted after each shutdown, the slurry resuspended easily.

Twice during the run, at hour 164 and hour 188, the pump power demand decreased from 1020 watts to 900 watts, indicating that the density of the slurry had been reduced or that the flow had decreased. Samples taken immediately following these decreases in pump power demand showed that the

thorium concentration in the circulating stream had decreased to ~ 200 g Th per kg water. The average thorium concentration for the run was ~ 500 g Th per kg water. This condition was corrected by adding 20 ml of water to the loop and adding oxygen to increase the total pressure from 600 to 690 psi. Evidently, the pressure on the suction side of the pump had decreased below the vapor pressure of the fluid and allowed vapor to form in the pump, causing the flow to decrease.

At times, it was difficult to produce enough condensate to keep a steady purge stream flowing to the pump bearings. This difficulty may have been due to having too little liquid in the system, blocking the condenser with noncondensable gases, or having an insufficient temperature differential in the condenser system. When the pump bearing purge was cut off for periods of 3 hr, no adverse effects were noticed in the operation of the pump.

The loop was operated for 192 hr while circulating thorium oxide slurry in run HT-21. Two general conclusions may be drawn form this run. The 1600°C-calcined thorium oxide slurry used can be added, circulated, and resuspended after a shutdown, without difficulty. With a small-volume loop such as this, the operation with thoria slurry is very sensitive to changes in fluid volume, temperature, or pressure. From Table 2, it is seen that the thorium concentration of samples taken during the run deviated considerably from the calculated concentration of the circulating stream. Poor sampling techniques may be the cause of this difficulty.

V. Run HT-22

No additional changes were made to the loop after the previous run, HT-21. The purpose of run HT-22 was to continue evaluation of the handling properties of the 1600°C-calcined slurry. The slurry used was the same as that used in run HT-21

The loop was filled with distilled water and enough oxygen was added to give a pressure of 60 psi (room temperature). After the water had been heated to 250°C and the system had reached equilibrium, 700 g of ThO_2 was placed in the addition tank and added to the loop. Operation was normal while the slurry was being circulated. When the system pressure was low, the power demand would decrease, indicating that the flow had been reduced

and some of the thoria had settled in the lines. This was probably caused by pump cavitation. This situation could always be corrected by adding oxygen and/or water to bring the system pressure to about 100 psi over the vapor pressure of water at operating temperature.

During this run, the pump bearings were purged with a stream of condensate at the rate of 20 ml/min, a flow rate which was easily maintained throughout the run. When 523 hr of operation with slurry had elapsed, the pump temperature rose suddenly to 90°C -- it had been operating at 55°C. Also at this time, a scraping or dragging noise was heard in the pump. The run was terminated and the pump was disassembled. It was noted that 0.0130 in. of material had been worn from both the front-bearing thrust surface and the front-rotor-journal thrust surface. The bearing and journal bushings (aluminum oxide) were rather badly cracked and scarred, making it difficult to obtain accurate measurements. It was evident that the thrust-surface wear had allowed the rotor to move forward far enough to enable the front-journal lock ring to strike the bearing spacer and produce the scraping sound when the pump was operating. The inside surfaces of both bearings were worn approximately 0.005 in.; the journal bushings (on the rotor) showed no wear.

The sampling technique was not changed during this run; and, as in run HT-21, the thorium concentration of the samples deviates considerably from the calculated values.

VI. Summary of Operations

Run HT-20

(a) Purpose: Clean, measure flow, and check operation of redesigned "HT" loop.

Charge No. 1: 1200 ml 3% TSP

Gas pressure: Helium, 60 psi at 25°C

Temperature: 90°C

Time: 4 hours

(b) Charge No. 2: 1200 ml, 5% HNO_3

Gas pressure: 60 psi helium at 25°C

Temperature: 95°C

Time: 46 hours

(c) Charge No. 3: 1200 ml H_2O
Gas pressure: O_2, 60 psi at 25°C
Temperature: 250°C
Time: 8 hours
Flow rate: 7.1 gpm
Operation: Loop functioned normally

Run HT-21

Purpose: Operate the loop using 1600°C-calcined thorium oxide slurry.
Charge: 1200 ml H_2O (690 g ThO_2 were added during the period from hr 51-97)
Gas pressure: O_2, 90 psi at 25°C
Temperature: 250°C
Pressure: 670 psi
Slurry concentration: 500 g ThO_2/kg H_2O
Time: 243 hr (192 hr with ThO_2 slurry)
Flow rate: 7.1 gpm
Operation: Loop operated satisfactorily.

Run HT-22

Purpose: Continue to evaluate the handling properties of 1600°C-calcined thoria slurry in the 5-gpm slurry loop.
Charge: 1200 ml H_2O (at hour 23, 700 g ThO_2 were added)
Gas pressure: 60 psi O_2 at 25°C
Temperature: 250°C
Pressure: 610 psi
Slurry concentration: 550 g Th/kg H_2O
Time: 547 hours (544 hr with ThO_2 slurry)
Flow Rate: 7.1 gpm
Bearing purge rate: 10 ml/min
Pump temperature: 55°C
Operation: No difficulty was experiences until the final hours of the run. The front thrust surfaces of the bearings and journals were worn so that the rotor moved forward and began to rub on the bearing spacer. This caused the pump temperature to rise, and the run was terminated.

APPENDIX

VOLUME OF LOOP = 1400 cc
VOLUME OF ADDITION SYSTEM = 485 cc (ADDITION TANK = 300 cc)
VOLUME OF CONDENSATE SYSTEM = 200 cc

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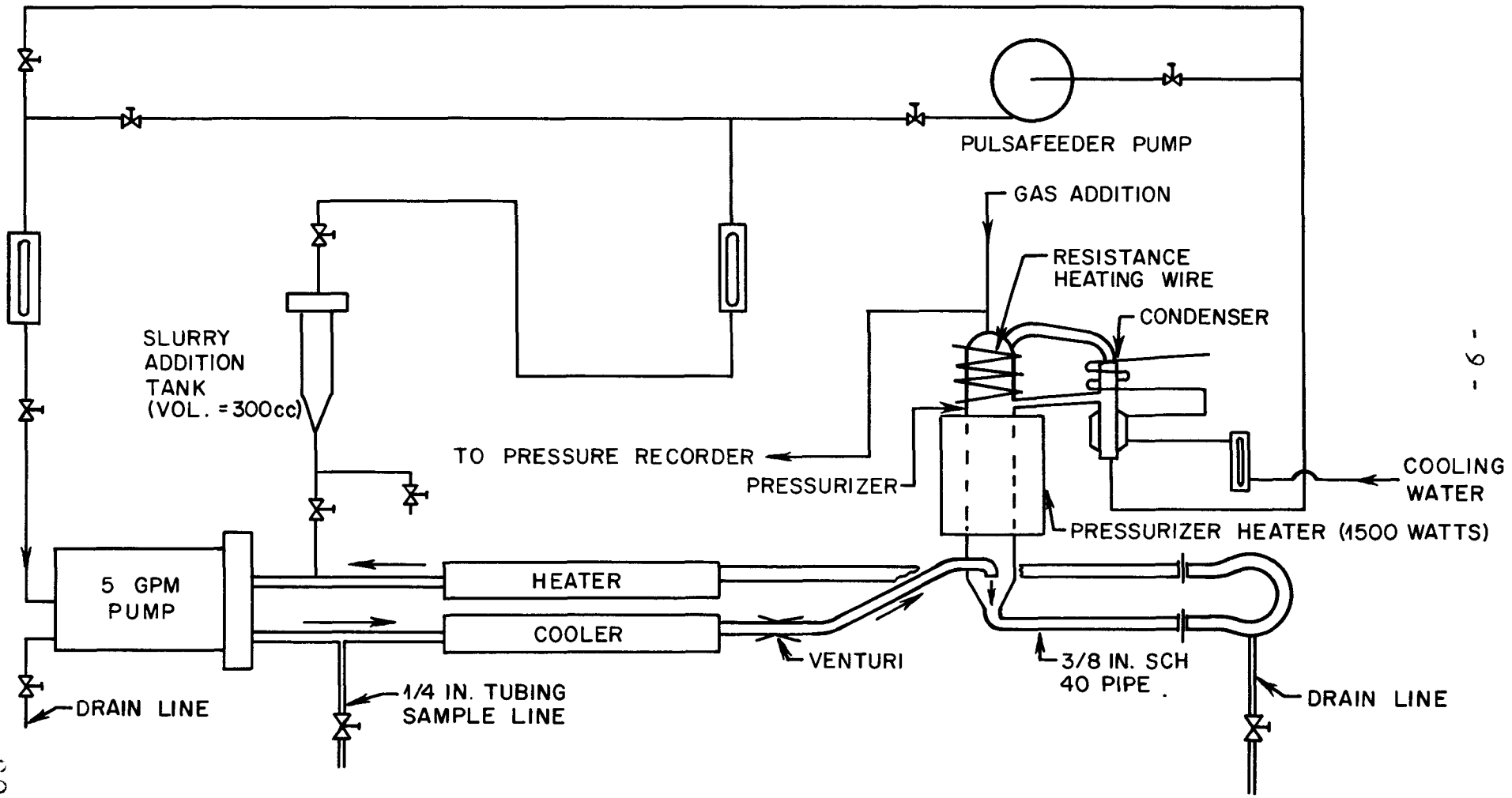

Fig. 1. 5 GPM Slurry Development Loop (Runs HT-20, HT-21, HT-22)

TABLE I

Chemical Analysis of Samples Taken During Run HT-20

Fluid	Loop Temperature (°C)	Gas	Operation Time (hours)	pH	Analysis (mg/gram of sample) Fe (mg/g)	Ni (mg/g)	Cr (mg/g)	Cu (mg/g)	Al (mg/g)	Ti (mg/g)	Th (mg/g)	Cl^- (mg/g)	$PO_4^=$ (mg/g)
TSP (3%)	-	↑	0	11.0	$< 1x10^{-3}$	$2x10^{-3}$	$< 1x10^{-3}$	$< 1x10^{-3}$	-	$2x10^{-3}$	-	$8x10^{-3}$	13.4
TSP (3%)	90		2.2	12.0	< 0.005	< 0.005	< 0.005	< 0.005	-	< 0.02	0.17	< 0.01	17.4
TSP (3%)	40		4.5	11.9	< 0.005	< 0.005	< 0.005	< 0.005	-	< 0.02	< 0.02	< 0.01	17.6
HNO_3(5%)	-		0	0.4	$< 1x10^{-3}$	$2x10^{-3}$	$< 1x10^{-3}$	$< 1x10^{-3}$	$2x10^{-3}$	$23x10^{-3}$	-	$< 2x10^{-3}$	-
HNO_3(5%)	100	Helium	0.6	0.4	0.055	0.011	0.019	< 0.005	< 0.02	< 0.02	0.09	< 0.01	< 0.02
HNO_3(5%)	90		18.1	0.6	0.089	0.022	< 0.005	0.059	< 0.02	< 0.02	0.03	< 0.01	< 0.02
HNO_3(5%)	-		19.8	0.6	0.094	0.023	0.006	0.063	< 0.02	< 0.02	0.05	< 0.01	< 0.02
HNO_3(5%)	-		0	0.2	$< 1x10^{-3}$	$< 1x10^{-3}$	$< 1x10^{-3}$	$< 1x10^{-3}$	$1x10^{-3}$	$1x10^{-3}$	-	$< 1x10^{-3}$	-
HNO_3(5%)	94		1.9	1.3	$12x10^{-3}$	$2x10^{-3}$	$1x10^{-3}$	$1x10^{-3}$	$6x10^{-3}$	$2x10^{-3}$	$< 2x10^{-3}$	$< 2x10^{-3}$	-
HNO_3(5%)	94		19.3	1.1	$31x10^{-3}$	$4x10^{-3}$	$3x10^{-3}$	$3x10^{-3}$	$12x10^{-3}$	$2x10^{-3}$	$9x10^{-3}$	$< 2x10^{-3}$	-
HNO_3(5%)	100	↓	25.3	1.0	$37x10^{-3}$	$5x10^{-3}$	$4x10^{-3}$	$3x10^{-3}$	$11x10^{-3}$	$2x10^{-3}$	$14x10^{-3}$	$< 2x10^{-3}$	-

TABLE 2

Chemical Analysis of Samples Taken During Run HT-21

Fluid	Loop Temp. (°C)	Gas	Operation[1] Time (hours)	Analysis (mg/g of sample) Fe	Al	Density (g/ml)	pH	Th[2] (g Th/kg H_2O)	Load Concentration Calculated (g Th/kg H_2O)
ThO_2-H_2O slurry	250	O_2	71.5	0.73	0.15	1.18	6.6	210[3]	[3]
"	250	"	97.4	1.80	0.33	1.59	6.7	590	500
"	250	"	163.9	1.10	0.33	1.20	6.2	220[4]	"
"	250	"	187.8	0.54	0.06	1.17	6.2	190[4]	"
"	250	"	194.1	1.30	0.16	1.49	6.6	550	"
"	254	"	198.6	2.33	0.26	1.94	6.4	1020	"
"	-	"	222.5	2.05	0.28	1.65	6.4	710	"
"	250	"	230.5	1.76	0.46	1.45	6.4	490	"
"	250	"	237.9	2.23	0.37	1.66	5.8	590	"
"	42	"	243.2	1.55	0.24	1.34	5.9	350	"
"	55	"	244.6	1.30	0.18	1.32	5.9	340	"

(1) 700 g ThO_2 added during the period between hrs 51-97.

(2) Calculated from the density

(3) Sample taken while adding slurry

(4) Samples taken during period when there was evidence of pump cavitation.

TABLE 3

Chemical Analysis of Samples Taken During Run HT-22

Fluid	Loop Temp. (°C)	Pump Power (watts)	Gas	Operating Time Hours*	Load Concentration Calculated g/kg H_2O	Analysis (mg/g of sample)					
						Th (g/kg of H_2O)	Ni	Cr	Fe	Al	pH
↑	250	955	↑	118	550	368	-	-	1.49	0.31	6.5
Thorium	250	1050		139		768	-	-	2.31	0.40	6.0
Oxide	250	996		143		710	-	-	2.08	0.36	5.7
Slurry	245	1020		186		762	-	-	2.41	0.35	6.2
↓	250	1080	Oxygen	209	↓	800	-	-	2.21	0.35	5.6
	250	990		282		590	-	-	1.86	0.32	5.6
	249	1056		330		364	0.21	0.19	1.59	0.27	3.5
	248	1038		336		429	0.20	0.20	1.66	0.27	3.5
	250	930		450		243	0.12	0.11	0.96	0.23	4.4
	250	930		525		606	0.21	0.22	1.82	0.34	4.2
	-	-	↓	547		428	0.21	0.23	1.85	0.38	5.5

* 700 g ThO_2 added at hr 23.

REFERENCES

(1) R. M. Warner, et al., HRP Dynamic Corrosion Studies, ORNL CF-56-11-40, November 2, 1956.

see also HRP Quarterly Progress Reports; July 31, 1956 (ORNL-2148); October 31, 1956 (ORNL-2222).

(2) S. A. Reed, et al.; HRP Dynamic Corrosion Studies; ORNL CF 57-10-95, October 23, 1957.

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