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SUBJECT: Thorium Oxide Slurry Filtration: Preliminary Tests of a 3-Micron
Poroloy Sintered Filter; Runs 11, 12 and 13 in Loop L-4-24S

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SUMMARY

A Poroloy sintered-metal filter unit which was placed in a bypass arrangement in a 5-gpm loop, L-4-24S, for runs 11 and 12 was used to separate water from a flowing stream of thorium slurry. During runs 11 and 12, the filter unit prevented the accumulation of thorium in the horizontal pressurizer during 490 hr of operation at temperatures up to 250°C and circulating stream concentrations up to 490 g Th/kg H₂O.

Run 13 was made with the main stream of the loop flowing through the center of the filter. The flow velocity through the center of the filter was sufficiently high to prevent a filter cake from building up, and consequently the filter unit was rendered ineffective. Microscopic examination of the filter screen revealed no other reason for the ineffectiveness of the filter in run 13 other than the lack of filter cake. Further testing with and without cake build-up is scheduled for work in the pump test stand and in L-4-24S.

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INTRODUCTION

Since the 5-gpm loop is a very well-instrumented loop for both in- and out-of-pile operation and is relatively inexpensive to fabricate, it seems highly desirable to use a 5-gpm loop as a slurry loop. All the preliminary runs on a converted 5-gpm loop which have been reported previously^(1,2) resulted in slurry settling in the pressurizer where the velocity is very low (approximately 0.1 ft/sec). The pressurizer in the 5-gpm loop is mounted horizontally in order for the loop package to fit into an experimental hole in the LITR. To eliminate slurry settling in the pressurizer, methods to produce a pressurizer feed of essentially clear water were investigated. Initial attempts to use a hydroclone as this separation device were unsuccessful. The minimum overflow concentration which was consistently attained was in the 25-50 g Th/kg H₂O range, with a loop concentration in the 200-300 g Th/kg H₂O range. This concentration is considered too high for use as a feed to the in-pile pressurizer. The hydroclone has been used successfully to remove slurry from 100A loops.⁽³⁾ The circulating stream continually flows through the hydroclone for that application, while for the use here the slurry must of necessity make a single pass through the separator. The next type separator to be investigated was the sintered metal filter which is described in this report.

TEST PROCEDURE

Equipment Used:

1. The Poroloy filter, Fig. 1, is a cylinder 0.630-in. ID, 0.685-in. OD, 12-in. long made of woven wire sintered after being wound on a mandrel. The

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Fig. 1. Sintered Metal Filter Unit Arranged for Slurry Flow Through Annulus

screen tube of 3-5 micron average pore size was mounted inside a 1-in. sch-40 pipe of 1.049-in. ID.

2. The 5-gpm pump test stand used in the first test consists of a 5-gpm pump with a 10-liter supply tank and connecting piping. It contains a differential pressure cell for measuring head output of pump and sampling valves for sampling circulating fluid. There are air connections for pressurizing the supply tank up to ~80 psig. The test stand was built for room temperature tests only (Fig. 2).

3. The L-4-24S 5-gpm loop is built essentially like the in-pile loops except the horizontal pressurizer is of bolted rather than all-welded construction. Runs L-4-24S-11, L-4-24S-12 and L-4-24S-13 were made with piping arrangements as shown in Fig. 3, 4 and 5.

4. The thoria used for these tests was LO-40 1600°C-fired thoria. Average particle size was 1.2 μ (Table I).

Table I
Particle Size Distribution of ThO₂
Batch LO-40 Fired at 1600°C

<u>Original Slurry</u>		<u>Residue Left in Pressurizer, Run No. 11</u>	
<u>Particle Size</u> <u>(μ)</u>	<u>Distribution</u> <u>(per cent)</u>	<u>Particle Size</u> <u>(μ)</u>	<u>Distribution</u> <u>(per cent)</u>
< 5	100	< 1.5	98
< 3	97	< 1	80
< 2	86	< 0.7	47
< 1.5	69	< 0.5	13
< 1	30	< 0.2	3
< 0.7	7		
< 0.5	1		
Average	1.2 μ	Average	0.71 μ

Fig. 2. Diagram of Filter Test Stand

Test No. 1

The filter unit was installed in the pump test stand per Fig. 2. The slurry feed stream was fed into the annulus around the outside of the filter screen. The filtrate traveled from circumference to center and out through a rotometer back to suction side of the circulating pump. The loop was charged with enough LO-40 1600°C-fired thoria to produce a circulating stream concentration of approximately 300 g Th/kg H₂O. Samples taken after 3 hr of room temperature operation showed a loop concentration of 300 g Th/kg H₂O and a clear filtrate flow to the pressurizer. After 40 hr of operation at room temperature at a loop concentration of 416 g Th/kg H₂O, the filtrate was still clear. The run was continued for 85 hr at room temperature. The entire run was made with 10 psig air pressure on the supply tank and a 40-ft head drop across the filter unit. The only difficulty encountered was the temperature in the back of the circulating pump exceeding the desired level of 50 to 75°C. This difficulty, which had been noticed in previous runs on the pump test stand, was relieved by venting gas from the back of the pump.

Test No. 2

Test No. 2 of the Poroloy-sintered filter was run no. 11 in loop L-4-24S. The filter unit was connected into the loop in a horizontal position in a bypass line (Fig. 3), with practically all of the head output of the pump (40 ft H₂O) as pressure drop across the filter. The filtrate traveled from circumference to center of filter and on to the pressurizer; therefore, the pressurizer flow rate was proportional to the efficiency of the filter. After a brief shakedown run on water at 30 and 250°C, the temperature was lowered and slurry added via the addition tank. The pressurizer flow rate was ascertained by a rough heat balance. A loop

Fig. 3. Loop L-4-24S With Filter Unit for Run No. 11

sample taken after 20 hr of room temperature operation contained very little thorium. A beta-gamma survey revealed that most of the slurry had settled in the annulus around the filter. After 63 hr of operation at room temperature and three slurry additions, the loop concentration was found to be 475 g Th/kg H_2O , and the filtrate flow to the pressurizer was apparently clear water. The loop temperature was increased. Samples at 100 and 200°C showed 159 g Th/kg H_2O . After 17 hr of 250°C operation, the concentration in the circulating stream had dropped so low that a sample appeared as milky water.

A beta-gamma survey showed additional slurry had collected in the filter annulus. The loop was cooled to room temperature and the concentration increased to 245 g Th/kg H_2O after 164 hr of operation. This concentration was not maintained. A sample taken at 188 hr showed only 155 g Th/kg H_2O . The loop was shut down, drained, and rinsed in preparation for piping changes prior to further testing. The book inventory indicated 320 g of thorium in the loop; but drainage and rinses accounted for only 280 g of thorium, so there must have been a holdup of approximately 40 g in the filter unit and loop components. The inlet end of the horizontal pressurizer was removed to check for slurry which might have settled along the bottom of the pressurizer. This is a very low velocity region in the 0.1 ft/sec range. There was a small amount of fine sediment along the bottom of the horizontal pressurizer. Analysis revealed this sediment to be 0.7-micron average particle size rather than the 1.2-micron average particle size of the original slurry. Particle size distribution is shown in Table I.

Test No. 3

Test No. 3 of the Poroloy filter unit was run no. 12 in loop L-4-24S. The filter was arranged in the loop per Fig. 4, with the filter unit inclined at an angle to facilitate the flow of slurry through the annulus. Additional thermocouples were added to measure pressurizer heater temperatures to allow a better measurement of the Δt across the pressurizer heater to obtain a more reliable measure of flow to the pressurizer by means of a heat balance. After a short shakedown run on water at room temperature and 250°C, slurry was added via the addition tank with the loop operating at room temperature. Upon reheating the loop to 250°C, most of the slurry settled out in the filter unit as indicated by a loop sample and beta-gamma survey. After three more additions of slurry and 122 hr of room-temperature operation, the loop concentration had increased to 438 g Th/kg H₂O. A fifth addition was made and loop brought up to 250°C. Both run 11 and run 12 were made with 200-300 psi oxygen overpressure at 250°C. A heat balance around the pressurizer heater showed the filtrate or pressurizer flow rate to be 5 cc/sec. After 168 hr of operation at 250°C, the loop concentration was 487 g Th/kg H₂O and the filtrate clear. The run was continued at 250°C to 195 hr of operation, at which time it was cooled down to correct instrument trouble which was causing loss of heater power. While trying to correct this difficulty, the temperature in the back of the pump exceeded the desired level by 40°C. The pump was stopped, and upon attempting to restart the pump, the power meter went off scale and the motor did not turn over. An electrical check showed the stator windings to be satisfactory. The pump was removed from the loop and the pump seal weld cut open to inspect the pump

Fig. 4. Loop L-4-24S With Filter Unit for Run No. 12

bearings. Upon disassembling the pump, both Al_2O_3 journals were found to be cracked, while the Al_2O_3 bearings were in good condition. The diametrical clearance had increased 0.001 in. in 1078 hr of operation, 900 of which had been in slurry service without the use of purge water being supplied to the back of the pump. (4)

Drains and flushes accounted for 522 g of Th, while the "book value" was 605 g. This means a holdup of 83 g in the filter, core, pressurizer, or other loop components.

Test No. 4

Test No. 4 was run 13 in loop L-4-24S. This run was made with the Poroloy filter unit modified as Fig. 5 to permit the bulk flow of the loop to pass through the center of the filter per Fig. 6.

After a brief shakedown run on water at 200 and 250°C, slurry was added to the loop at 28, 48 and 71 hr of operation to bring the loop inventory up to 438 g Th/kg H_2O (with the loop cooled to room temperature for each addition). The loop was operated at 250°C without further sampling until 240 hr of loop operation. During this last 140 hr of operation, the only apparent change was that the filtrate flow rate from the filter to the pressurizer decreased from its original 22 cc/sec to 10.5 cc/sec and then remained constant. This flow rate was determined by a heat balance around the pressurizer heater. After 240 hr of operation a sample indicated the circulating slurry had dropped to 261 g Th/kg H_2O . This trend continued until the concentration was less than 25 g Th/kg H_2O at 310 hr. The loop temperature was lowered to 95°C and the circulating stream recovered

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Fig. 5. Sintered Metal Filter Unit Arranged for Slurry Flow Through Center

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Fig. 6. Loop L-4-24S With Filter Unit for Run No. 13

to 317 g Th/kg H₂O. The loop was operated at 95°C for 30 hr, then raised back to 250°C; 75 hr later the concentration was down to almost zero. Radiographs made with the loop operating at 250°C revealed a quantity of slurry settled out in the core and pressurizer. The pressurizer samples had been clear throughout the run. Since the pressurizer sampling station is downstream from the pressurizer proper, the samples must have contained only supernatant after the slurry had settled out in the horizontal portion of the pressurizer. The temperature was dropped to 95°C for 60 hr of operation. Samples taken at 478 hr just prior to shutdown gave a concentration of 173 g Th/kg H₂O. The loop was shut down and drained.

Removal of the pressurizer end plates revealed a layer of slurry 1/8- to 1/4-in. thick along the bottom of the 1 1/2-in. horizontal section of the pressurizer. Analysis of this sediment gave essentially the same particle size distribution as the original charge. The filter unit was disassembled for examination. Microscopic examination failed to show any cracks or large holes through the filter element. This would indicate the necessity of a cake build-up on the filter to successfully separate slurry and water. The layer of slurry deposited in the pressurizer covered the end of the thermocouple probe which extends into the pressurizer. This may explain the steady, although slight, drop in the pressurizer temperature as indicated by this thermocouple during the latter part of the run.

Further Tests

Further tests to better evaluate the importance of a cake build-up on the filter will be made on the pump test stand at room temperature. Outcome of these tests will determine which tests are to be made at high temperature and pressure in loop L-4S-24S. Similar tests will be made on other filter materials which are now on order.

SUMMARY OF OPERATIONS

Test No. 1 (made in pump test stand)

Purpose: To check possibility of separating water from thorium slurry.

Charge: 500 g Th/kg H₂O; 2 liters IO-40 1600°C thorium.

Temperature: Room temperature.

Pressure: 10 psig.

Flow Rate: Main stream 5 gpm; filtrate 1 cc/sec.

Length of Run: 85 hr.

Operation: Operation satisfactory, since it proved a clear filtrate could be produced under these conditions.

Test No. 1, which was carried out in the pump test stand, demonstrated satisfactorily that the sintered filter unit was capable of separating water from a flowing stream of thorium slurry at room temperature and 10 psi. These results were deemed adequate to warrant further testing of the unit in an in-pile type loop at higher temperature and pressure.

Test No. 2 (run 11 in L-4-24S loop)

Purpose: To test sintered metal filter at temperature and pressure.

Charge: 425 g Th/kg H₂O; IO-40 1600°C thorium.

Temperature: Room temperature to 250°C.

Pressure: 100-1100 psi (oxygen + steam)

Flow Rate: Main stream 5 gpm; filtrate 5 cc/sec.

Length of Run: 190 hr.

Operation: Filter worked satisfactorily in that a clear filtrate was produced. There was a large holdup in the filter annulus and some loop components. There were small deposits of fine thoria left in the horizontal pressurizer.

Test No. 2, run 11 in the L-4-24S loop, demonstrated the filter capable of separating water from slurry both at room temperature and 250°C. There was a holdup in the annulus of the filter which represented a sizable portion of the loop inventory, although it did not seem to hinder the filtrate flow. The separation was satisfactory with loop concentrations up to 500 g Th/kg H₂O with a 40-ft head drop across the filter unit. Figure 7 is a plot of the operating data.

Test No. 3 (run 12 in L-4-24S loop)

Purpose: To further test sintered filter at temperature and pressure.

Charge: 500 g Th/kg H₂O; LO-40 1600°C thoria.

Temperature: Room temperature to 250°C.

Pressure: 100-1100 psi (O₂ + steam).

Flow Rate: Main stream 5 gpm; filtrate 5 cc/sec.

Length of Run: 290 hr.

Operation: Operation satisfactory except large holdup in filter unit annulus. Near conclusion of run the pump became gas bound; and due to failure to have a cutout jumper open the temperature in the back of the pump, greatly exceeded desired operating level. This resulted in cracked Al₂O₃ journals on the rotor shaft. Figure 8 is a plot of the operating data.

Fig. 7. Plot of Operating Temperature and Sample Analysis Loop L4-24S Run-11

Fig. 8. Plot of Operating Temperature and Sample Analysis Loop L4-24S Run-12

Test No. 3, run 12 in loop L-4-24S, was made with a similar piping arrangement as that used in run 11 except the filter unit was inclined at an angle to facilitate the movement of slurry through the filter unit annulus to reduce the holdup.

The results were practically the same as run 11 with a large holdup in the filter unit. These tests show it is possible to separate a clear filtrate from a flowing stream of thorium slurry at a concentration of 500 g Th/kg H₂O and at temperatures up to 250°C by using a sintered metal filter. The filtrate flow rate was approximately proportional to the viscosity of water.

Test No. 4 (run 13 in L-4-24S loop)

Purpose:	To test filter with total flow through its center at temperature.
Charge:	438 g Th/kg H ₂ O; LO-40 1600°C thorium.
Temperature:	Room temperature to 250°C.
Pressure:	100-1100 psi (O ₂ + steam)
Flow Rate:	Main stream 5 gpm; filtrate 10.5 cc/sec.
Length of Run:	480 hr.
Operation:	Continual loss of slurry from main loop also steady, but slight drop in temperature inside the pressurizer. Radiographs showed slurry deposited in bottom of pressurizer and in core. Disassembled screen checked satisfactorily; therefore, a filter cake seems necessary for the separation to progress.

Test No. 4, run 12 in the L-4-24S loop, was made with the bulk slurry flow through the center of the filter. The velocity of the main stream prevented a cake build-up, and as a result the filter was ineffective. Radiographs taken when

the loop was in operation at 250°C showed slurry settled out in both pressurizer and core sections. This would indicate the necessity of permitting a cake build-up to aid filtration and a redesign of the core before in-pile testing can be considered.

FUTURE TESTS

Further testing to better evaluate the importance of the cake build-up and attempts to control the amount of this build-up will be made. Other tests with different filters, which are now on order, will be made as soon as possible.

CONCLUSIONS

It is possible to produce, by means of a sintered-metal filter, a filtrate of essentially clear water to be used as a feed stream to a horizontal pressurizer for an in-pile loop. This filtrate offers a good possible purge stream for circulating pumps and instrument lines. Tests to date indicate the necessity of a filter cake to aid separation. This cake would have to be kept at a minimum for in-pile work; therefore, further development work is required.

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