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TITANIUM PUMP LOOP FOR AQUEOUS
SOLUTIONS AT HIGH TEMPERATURES

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OAK RIDGE NATIONAL LABORATORY

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TITANIUM PUMP LOOP FOR AQUEOUS SOLUTIONS AT HIGH TEMPERATURES

J. M. Baker

S. E. Bolt

ABSTRACT

A titanium pump loop, designed to circulate aqueous solutions at temperatures and pressures up to 370°C and 3000 psia, was constructed. It is to be used to study the chemical stability of uranyl sulfate fuel solutions of interest to the Fluid Fuels Reactor Program. The total loop volume was minimized so that about 2 liters of solution was sufficient for loop operation.

The equipment includes (1) a sampling system to remove solution samples from the loop while operating at elevated temperature and pressure; (2) a hydroclone to separate and remove any solids and/or heavy-phase material formed; and (3) provisions for installation of corrosion test specimens in the main loop stream. All equipment performed satisfactorily at design conditions in tests with water.

1. INTRODUCTION

In the aqueous homogeneous reactor program at Oak Ridge National Laboratory, the chemical stability and corrosiveness of the uranyl sulfate fuel solutions at elevated temperatures has been the subject of a large number of investigations.^{1,2} These studies have been carried out in both small-scale autoclaves and in dynamic pump loops. The pump loop tests more nearly duplicate the dynamic conditions which exist in an aqueous homogeneous reactor as compared with static or rocking autoclaves; therefore, a pump loop capable of operation with uranyl sulfate solutions at temperatures and pressures higher than those of previous tests was desired to extend these studies.

The design of the type 347 stainless steel and titanium loops operated in the past has limited their operation to temperatures and pressures below ~330°C and 2000 psi. In order to extend the studies with

¹J. A. Lane, H. G. MacPherson, and F. Maslan, eds., Fluid Fuel Reactors, chap. 3, "Properties of Aqueous Fuel Solutions," pp 85-120, Addison-Wesley, Reading, Mass., 1958.

²W. L. Marshall et al., HRP Quar. Progr. Rept. July 31, 1960, ORNL-3004, pp 49-60.

uranyl sulfate fuel solutions to temperatures near the critical temperature of water (372°C), a pump loop was constructed to withstand temperatures and pressures to 370°C and 3000 psia. Titanium was chosen as the material of construction since it is substantially more corrosion-resistant to acid-containing uranyl sulfate solutions than type 347 stainless steel. The use of titanium also eliminates the introduction of soluble stainless steel corrosion products into the solution under test.

This report describes the various features of the loop and the calibration tests with water made to demonstrate the capability of the system to operate at design conditions.

2. DESCRIPTION OF LOOP

The loop is similar in design to the HRP in-pile loops.³ A 5-gpm canned-rotor pump is used to circulate the solution. A combination heater-cooler unit controls the main-stream temperature. Corrosion-specimen holders are mounted in the main loop piping, and a horizontally mounted pressurizer with a separate preheater is located in a bypass stream off the main loop piping. A hydroclone⁴ is installed in another bypass line to remove heavy phases which might form during operation at elevated temperatures. The main loop piping is 1/4-in. sched-80 pipe.

Also included are provisions for removing samples of the fuel solution, either from the main loop stream, the hydroclone underflow pot (where solids or heavy-phase material is collected), or from the pump-rotor cavity while the loop is operating at temperature and pressure.

A schematic diagram of the loop is shown in Fig. 1. A tabulation of all drawings used in the fabrication of the loop, auxiliary equipment, and instrument panel is included in the Appendix, along with a bill of material identifying the types of titanium used. Several different grades of titanium were used for two reasons: (1) availability of acceptable material, and (2) strength requirements for the larger components.

³H. C. Savage et al., In-Pile Corrosion Test Loops for Aqueous Homogeneous Reactor Solutions, ORNL-2977 (Nov. 10, 1960).

⁴P. A. Haas, Hydraulic Cyclones for Application to Homogeneous Reactor Chemical Processing, ORNL-2301 (Nov. 18, 1957).

Fig. 1. High Pressure Titanium Loop.

2.1 Pump

The circulating pump is an ORNL 5-gpm canned-rotor pump of all-welded construction and was fabricated entirely of titanium with the exception of the bearings and journal bushings, which are aluminum oxide. The pump is identical to those used in the HRP in-pile pump loops except that the impeller diameter was increased from 3-1/4 in. to 4 in. in order to increase the pump output, and the thicknesses of the pump containment can and impeller housing were increased as required for the increased pressure. The head-capacity curve for this pump is shown in Fig. 2. In the same figure the flow rates in the loop pressurizer circuit and the hydroclone unit vs flow in the main loop piping are also shown. From these curves a main loop flow of 4.5 gpm is indicated, with corresponding flows of 35 and 15 cc/sec through the hydroclone and pressurizer respectively.

Fig. 2. Head-Capacity Curve for Titanium Loop Pump.

2.2 Loop Heater-Cooler

The heating unit located in the main loop stream consists of four 1000-w Calrod-type electric heating elements cast in an aluminum matrix around the main loop piping. A cooling coil is also cast in this aluminum matrix, and air-water mixtures are passed through the coil for cooling. By means of this dual-purpose unit, loop operating temperatures can be controlled from room temperature to 370°C.

2.3 Corrosion Test Specimens

Corrosion test specimens can be mounted in two special titanium holders in the main loop stream. Each holder contains twelve $1/2 \times 1/2 \times 0.060$ in. flat coupon specimens as shown in Fig. 3. The tapered flow channels of these holders give a velocity distribution of 7 to 34 fps (at the loop flow of 4.6 gpm) across the corrosion specimens and allow

a determination of the effect of fluid velocity on corrosion rates. Lap-joint flanges with 0.10-in.-thick titanium gaskets are used to mount these corrosion specimen holders in the loop.

Fig. 3. Corrosion Specimens, Holder, and Flow-Velocity Distribution.

2.4 Pressurizer

The cylindrical pressurizer is mounted in a horizontal position in a bypass line and is connected to the loop at each end by means of 0.250-in.-OD $\times$ 0.150-in.-ID tubing. Two tubes of capillary dimensions are attached to the pressurizer end-caps in the upper vapor space, and a thermocouple well extends into the lower liquid space. The capillary tubes are used for pressure measurements and for gas additions to the loop.

The pressurizer volume is 4.7 liters, and that of the loop piping and hydroclone underflow pot is 1 liter. This relatively large pressurizer volume permits volumetric expansion of the solution in the loop as the temperature is increased from room temperature to a temperature near critical. It also provides a large reservoir so that many samples of the solution may be removed from the loop without the necessity of replacing the solution. A heating jacket is installed around the pressurizer body to minimize heat loss and is used to maintain equilibrium thermal conditions for improved accuracy of temperature-pressure measurements.

2.5 Hydroclone

A 0.10-in. hydroclone is mounted directly across the discharge and suction of the pump, and a bypass stream from the loop passes through the hydroclone for separation of solids and/or the heavy-liquid phase. The inside diameter of the hydroclone at the feed port is 0.4 in., and it contains 0.10-in.-diam underflow and overflow ports. Any material separated is collected in a 420-cc underflow pot mounted directly under the hydroclone, where it can be sampled or removed. The temperature of the underflow pot can be maintained at loop temperature, if desired, by means of a heating jacket.

2.6 Temperature and Pressure Control

The temperatures of the loop and pressurizer are controlled by the loop and pressurizer heaters, and the power supply to these heaters is in turn controlled by air-operated Variacs. A thermocouple connected to

a recorder-controller controls the air supply to the loop-heater Variac. More accurate control of the pressurizer temperature is required for two reasons: (1) uncontrolled temperature excursions at temperatures near 370°C could exceed the critical temperature, and (2) partial pressures of oxygen or other gases and steam in the pressurizer are determined from accurate measurements of the total pressure and temperature which require that insofar as possible steady-state thermal conditions be maintained.

3. LOOP PRETREATMENT RUNS

In order to remove all traces of oil, grease, and foreign material, a 3% trisodium phosphate solution was circulated in the loop at 100°C for 4.5 hr. After rinsing, this was followed by a similar run with 5% nitric acid at 100°C for 25 hr.

4. LOOP TESTS WITH WATER

After the pretreatment, the loop was charged with water under vacuum and operated at elevated temperature and pressure to demonstrate its ability to operate at these temperatures and to measure the degree of accuracy to which the temperatures could be controlled. The accuracy of the pressurizer thermocouple and evidence of equilibrium thermal conditions were determined by means of a "steam calibration." This consists in measuring the pressurizer temperature and pressure and comparing with steam-table values. During calibration the pressurizer was operated at temperatures to 360°C, and the loop at 2 to 3°C below this in order to prevent boiling in the main loop stream. For purposes of calibration, the pressure reading was assumed to be correct, since very accurate calibrations of the pressure cells were made, and any error between the pressurizer temperature and pressure was attributed to the thermocouple. The data obtained in this manner are given in Table 1 and show that in the temperature range 300 to 360°C the pressurizer-thermocouple error varied between +0.4 and -1.7°C. This error was considered acceptable.

Table 1. Steam Calibrations of the Pressurizer Thermocouple

Pressurizer Temperature (°C)	Loop Temperature (°C)	Pressure (psia)	Pressurizer Thermocouple Error (°C)
300	297	1246	-0.5
320	317	1642	-0.6
340	337	2144	-1.2
360	357	2709	-1.7
360	356	2700	+0.4

5. CONCLUSIONS

Test results indicated that the loop is entirely satisfactory for operation at temperatures and pressures to at least 365°C and 3000 psia. No difficulties were encountered in the operating tests with water.

6. ACKNOWLEDGMENTS

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P. G. Herndon	Instrumentation
C. H. Wodtke	Welding Design and Assembly
J. R. Hart	Assembly of the Loop and Components

7. APPENDIX

7.1 Construction Materials for High-Temperature Titanium Loop

Component	Material	Nominal Room Temperature Tensile Properties		
		Ultimate (psi)	Yield (psi)	Elong. in 2 in. (%)
Loop piping	1/4-in. sched-80 seamless pipe, commercially pure titanium*	50,000	40,000	22
Pressurizer circuit	1/4-in.-OD x 0.049-in.-wall seamless tubing, Ti grade II	74,000	59,000	19
Hydroclone circuit	1/4-in.-OD x 0.049-in.-wall seamless tubing, Ti grade II	74,000	59,000	19
Capillaries	0.090-in.-OD x 0.020-in.-wall seamless tubing, commercially pure titanium*	50,000	40,000	22
Pump				
Housing	4-1/2-in.-OD bar, hot-rolled, annealed, Ti-110AT	115,000	110,000	10
Scroll	6-1/4-in.-OD bar, forged, Ti-110AT	115,000	110,000	10
Discharge nipple	3/4-in.-OD bar, Ti-75A	80,000	70,000	15
Capillary adapter	1/4-in.-OD rod, Ti-45A	50,000	40,000	22
Bearings and journals	Kearfott 99% Al ₂ O ₃			
Sample holder and stub ends	2-1/2-in.-OD bar, Ti-55A	55,000	45,000	20
Pressurizer				
Tube fitting	1/4-in.-OD rod, RC-70	80,000	70,000	15
Caps	4-1/2-in.-OD bar, Ti-110AT	115,000	110,000	10
Body	Pipe, 3-13/16-in.-OD x 0.281- in.-wall, Ti-110AT (HRT mockup pressurizer stock - Bridgeport Brass Company)	115,000	110,000	10
TC well	1/4-in.-OD x 0.083-in.-wall seamless tubing, commercially pure*	50,000	40,000	22
Hydroclone				
Cap, body, and pot	2-in.-OD bar, Ti-55A	55,000	45,000	20

*Assumed to be A-40 titanium for design purposes.

7.2 Drawing List

Drawing Number	Title
TD-D-3675	Modified ORNL Canned-Rotor Pump Assembly
TD-D-3677	Modified ORNL Canned-Rotor Pump Housing and Details
TD-D-3681	Modified ORNL Canned-Rotor Pump Stator
TD-D-3685	Hydroclone
TD-D-3686	Hydroclone Heater
TD-D-3687	Piping Layout
TD-D-3688	Flowsheet
TD-D-3695	Sample Holder
TD-E-3678	Heater-Cooler
TD-E-3683	Pressurizer
TD-E-3684	Pressurizer Heater
D-27003	Thermal Shield Assembly
D-27004	Rotor Assembly
D-27006	Details - Pump
TD-D-5408	Instrument Flowsheet
TD-D-5409	Panel Layout
TD-D-5410	Power-Circuit Schematic
TD-D-5411	Control-Circuit Schematic
TD-D-5412	Relay and Terminal-Box Diagram
TD-D-5413	Instrument Contact Wiring Diagram

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