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PUMP LOOPS USED FOR MATERIALS TESTING IN
HIGH TEMPERATURE AQUEOUS SOLUTIONS

AND SLURRIES

H. C. Savage

OAK RIDGE NATIONAL LABORATORY

operated by

UNION CARBIDE CORPORATION

for the

U.S. ATOMIC ENERGY COMMISSION

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ORNL-3449

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REACTOR CHEMISTRY DIVISION

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H. C. Savage

DATE ISSUED

JUN 24 1963

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FOREWORD

The pump loops used for materials testing in high temperature aqueous solutions and slurries have been described regularly in HRP Quarterly Progress Reports, beginning with the report in November 30, 1950 (ORNL-925) and continuing through the report for the period ending on January 31, 1960 (ORNL-2920). Descriptions of the engineering development of the loops and summaries of each of the loop experiments are included in the 34 reports of that progress-report series.

A number of people have contributed to the pump loop program during the past ten years. From the beginning the loop program has been under the general direction of E. G. Bohlmann, who has contributed substantially in establishing design criteria and operating procedures throughout this period. E. L. Compere and J. C. Griess have been responsible for establishing many of the operating procedures and techniques used.

The following individuals assisted in the mechanical design, construction, and operation: J. M. Baker, S. E. Bolt, R. B. Gallaher, D. W. Hubbard, D. T. Jones, R. A. Lorenz, T. H. Mauney, J. R. McWherter, R. M. Pierce, S. A. Reed, W. C. Ulrich, F. J. Walter, R. M. Warner, and J. F. Winesette.

Many persons have assisted and contributed substantially to the design and installation of instrumentation and controls, including: P. G. Herndon, R. C. Moore, R. M. Pierce, J. A. Russell, D. S. Toomb, and H. D. Wills.

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PUMP LOOPS USED FOR MATERIALS TESTING
IN HIGH TEMPERATURE AQUEOUS SOLUTIONS
AND SLURRIES

H. C. Savage

ABSTRACT

Pump loops designed to operate at temperatures and pressures to 320°C and 2,000 psia are described. These loops were used to circulate uranyl sulfate solutions and aqueous thorium slurries in out-of-pile tests for the aqueous homogeneous reactor program. As this phase of the aqueous homogeneous reactor program was completed, the loops were converted for use in studies of materials of interest to other types of reactors.

Solutions or slurries are circulated in the loops by means of a 100-gpm canned-motor centrifugal pump. The loop piping is of 1- or 1-1/2-in. size and pressurizers of 4-in. pipe are used where required for steam pressurization. Two of the loops were constructed of commercially pure titanium while all others were of type 304 stainless steel. Some loops are hydraulically pressurized by means of a feed pump and letdown valve arrangement. The nominal volumes of the 15 loops which have been constructed varies between 10 and 30 liters, and each loop was designed to contain a large number of corrosion test specimens of various types for exposure under a wide range of conditions.

Each loop test was usually of 200 to 500 hr duration, but some tests were operated continuously for more than 7,000 hr. Operating experience covering more than 350,000 hr of accumulated operating time in many hundreds of individual test runs is discussed.

The pump loops described have proved to be highly satisfactory systems in which to study reactor materials under a wide range of conditions which can be closely controlled to simulate those existing in nuclear reactors.

1. INTRODUCTION

The purpose of this report is to describe in some detail the pump-loop systems which were initially designed to obtain dynamic corrosion data and information on the chemical stability of the fuel solutions for the Aqueous Homogeneous Reactor Project at Oak Ridge National Laboratory. The dynamic test loop program was begun early in 1950 when one loop, designed to operate at elevated temperature and pressure, was placed in service. This first loop was used primarily to obtain data on the chemical stability and corrosiveness of a uranyl sulfate fuel solution proposed for the first Homogeneous Reactor Experiment (HRE-1). From this beginning

the dynamic test loop program expanded to include fifteen pump loops which were designed for continuous operation on a 24 hr/day, 7 day/week basis.

In addition to the studies with uranyl sulfate solutions the loops were modified to evaluate the corrosion, erosion, and handling properties of concentrated slurries of thorium oxide (up to ~1500 g of Th per kg of H₂O and/or D₂O) for the Aqueous Homogeneous Reactor Project.

A large number of corrosion test specimens of various types were used in the dynamic solution and slurry corrosion test program. The type of specimen used was, of course, dependent on the type of corrosion effect under study. One type of specimen was designed to obtain rather qualitative results where large numbers of test specimens of various types of material were required. Another type was designed to obtain more quantitative results on the effects of solution velocity on the corrosion rate. Still other specimens were used where information on stress-corrosion cracking was desired.

An important aspect of the dynamic corrosion test loops was the ability to evaluate the performance and reliability of component parts proposed for use in homogeneous reactor systems. In this category were operating tests of valves, pumps, pump-bearing materials, control instruments, oval ring flanges and other types of leak-tight, high-pressure closures. The fabrication of loops also aided in the development of improved welding techniques with subsequent evaluation under reactor operating conditions. Procedures and techniques used in loop operation have been valuable in preparing specifications for reactor operations. The experience gained in operation of forced-circulation loops of this type was of considerable value in the design, construction, and operation of the two experimental reactors, HRE-1 and -2.

As this phase of the Aqueous Homogeneous Reactor Program was completed, the loops were converted for use in the investigation of dynamic corrosion phenomena of interest to other types of reactors. For example, two loops have been modified to obtain data on the corrosion of aluminum under conditions of heat transfer at high heat fluxes.^{1,2}

Thus the pump loops have proved to be valuable for the study of the behavior of aqueous systems under a wide range of conditions which could be controlled to simulate those existing in nuclear reactors.

¹J. C. Griess, H. C. Savage et al., Effect of Heat Flux on the Corrosion of Aluminum by Water. Part I. Experimental Equipment and Preliminary Test Results, ORNL-2939 (Apr. 29, 1960).

²J. C. Griess, H. C. Savage et al., Effect of Heat Flux on the Corrosion of Aluminum by Water. Part II. Influence of Water Temperature, Velocity, and pH on Corrosion-Product Formation, ORNL-3056 (Feb. 10, 1961).

2. LOOP DESIGN

2.1 Solution Test Loops

A schematic diagram of a typical pump test loop is shown in Fig. 1. Figure 2 is a photograph of a typical loop. Referring to Fig. 1, it can be seen that the loop consists of a pump to circulate the solution, piping through which the solution circulates, and a pressurizer. The pressurizer is interconnected with the loop piping and has several functions: (1) provide system overpressure which may be either steam or gas, (2) provide expansion space for solution in the loop during heat-up, (3) provide space for gas (oxygen, hydrogen, etc.) that may be required for solution stability and/or corrosion studies, and (4) provide space for excess solution volume required for solution samples removed during operation.

Corrosion specimens could be placed in a number of different locations such as in the main loop piping circuit, in a bypass line, or in the pressurizer liquid or vapor space. The bulk solution velocity past those samples in the loop piping was regulated by means of orifice-plate restrictors as required. A description of the various types of corrosion specimens is given in Sec. 2.6.

The loops were designed to operate at temperatures and pressures up to $\sim 320^{\circ}\text{C}$ and ~ 2000 psia. All pumps used were the canned-motor type, Westinghouse Model 100A. However, some pump revisions were made for improved operating reliability of these pumps in our particular system. A description of these revisions and the operating experience with these pumps is given later.

Loop and pressurizer heating was accomplished by means of electric Calrod-type heating elements cast in an aluminum matrix around the outside wall of the loop and pressurizer piping as shown in Fig. 3. Loop cooling was normally done by means of a water spray or drip on the loop piping, as shown in Fig. 4. However, more recent loops were equipped with a cooling coil cast in the aluminum matrix along with the electric heating elements. Air, water, and air-water mixtures were used as the coolant medium in the cooling coils. Figure 5 is a drawing of the combination heater-cooler unit.

2.2 Slurry Test Loops

The slurry test loops, designed to circulate thorium oxide slurries, were similar to the solution loops described above. Figure 6 is a schematic diagram of a typical thorium oxide slurry loop. Figure 7 is a photograph of a typical slurry loop. The distinguishing feature of the loops used for slurry corrosion studies was the addition of a condenser in the pressurizer vapor space to supply condensate to continuously purge the pump bearings and prevent thorium oxide accumulation with resultant bearing wear. A slurry addition device was also incorporated as auxiliary equipment so that thorium oxide could be charged to the loop while operating at temperature and pressure.

Fig. 1. Schematic Diagram of 100A Solution Test Loop.

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Fig. 2. Photograph of 100A Solution Loop.

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Fig. 3. Loop Heater.

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Fig. 4. Water Spray Loop Cooler.

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Fig. 5. Combination Heater-Cooler Unit.

Fig. 6. Schematic Diagram of 100A Slurry Test Loop.

Fig. 7A. Photograph of 100A Slurry Pump Loop Showing Main Loop Piping.

Fig. 7B. Photograph of 100A Slurry Pump Loop Showing Pressurizer and Condenser.

2.2.1 Condensate System for Slurry Loops

The condensate system shown in Fig. 6 was used to provide a continuous purge of clean water to the pump bearings, thus preventing wear by the thorium oxide particles. This system was also used in conjunction with an addition tank to make thorium oxide additions to the loop during operation (Sec. 5.2).

By referring to Fig. 6, it can be seen that the condenser system consists of a condensate tank connected to the top of the pressurizer. A 1-in. sched-80 pipe was used for this purpose. A second 1-in. sched-80 pipe connected the condensate tank to the pressurizer at a point below the top of the pressurizer, but still in the vapor space. This second pipe was sloped slightly downward for gravity flow return of excess condensate to the pressurizer. Cooling coils were attached to the condensate tank between the top and side connections to the pressurizer. By maintaining the horizontal leg of the top connection at the temperature of the pressurizer, condensation of steam in this portion of the system was prevented. The steam in the steam-gas mixture from the pressurizer was condensed in the upper, vertical part of the condensate tank and collected in the lower section of the tank. The residual gas, along with any overflow of condensate, was returned to the pressurizer via the horizontal connection, sloped downward toward the pressurizer to allow drainage. Thus thermal circulation of a steam-gas mixture was established through the condenser circuit. The rate of steam-gas circulation and amount of condensate produced was regulated by the capacity of the cooling coils and the driving force which resulted from the difference in density between the steam-gas mixture in the pressurizer, the residual gas in the condensate system, and the height of the condenser.

The particular condensate system now in use was designed to provide 10 liters per hr of clean condensate. It also had the advantage that at a given set of operating conditions in a loop, the condensation rate was essentially constant no matter how small or how large (up to the system capacity) the rate of withdrawal of the condensate.

A 1/4-in. tube connected the bottom of the condensate tank to the rear of the pump. The condensate flowed to the rear of the pump as a result of the pressure difference between these two points in the system. In the event of loss of this pressure difference, a pulsafeeder pump (positive displacement) was incorporated in the circuit and was used as required. Similarly, the condensate reservoir was connected to a slurry addition device which is described below.

2.2.2 Slurry Addition System

A slurry addition system, Fig. 6, was incorporated in the slurry loops as a standard piece of auxiliary equipment. The slurry addition tank was connected by means of 1/4-in. tubing to the main loop at the suction side of the pump. Condensate used to flush the addition tank entered at the top flanged closure. By means of the slurry addition

system, the concentration of thorium oxide in the system was increased, while the loop was in operation, in the following manner. The slurry addition tank, valved off from the loop, was filled with the desired quantity of thorium oxide slurry. The top flange of the tank was closed, pressurized, and checked for leak-tightness. The 1/4-in. line from the top of the slurry tank to the loop-condensate holdup tank was then opened, after which the 1/4-in. line from the bottom of the slurry addition tank to the pump suction line was opened. The differential pressure between the condensate tank and pump suction forced slurry into the loop, and the slurry was replaced by clean water from the condensate system. Thus the slurry in the addition tank was replaced by an equal volume of water from the loop. In the event of plugging of the 1/4-in. line from the slurry tank to the pump suction, the pulsafeeder, shown in Fig. 6, was used to apply sufficient pressure to unplug this line. The feed to the pulsafeeder was taken from the loop condensate tank so that it was not necessary to add additional water to the loop during operation of the slurry addition system. A high-pressure rotometer and a regulating valve were also provided in the addition system for measuring and adjusting the flow of condensate through the slurry addition tank.

2.3 Pumps

Circulation of the solution or slurry in the test loops was accomplished with a Westinghouse Model 100A, canned-motor, centrifugal pump. In general these pumps delivered 100 gpm at a head of 250 ft and were designed to withstand system pressures to 2500 psig. The pump motor is a 3-phase, 3400-rpm induction type. The pump motor is totally enclosed, and the rotor and bearings operate in the solution being pumped. In the case of thorium slurry, the rotor and bearing region is purged with condensate as described above. Electrical stator windings, cooled by circulating oil, are protected from the solution or slurry by a 0.040-in.-thick metal diaphragm. The rotor is canned in a metal sheath. All parts of the pump in contact with solution or slurry were made of materials resistant to the corrosive action of the solution or slurry. Figure 8 is a drawing of this pump.

Labyrinth rings in the thermal spacer that separated the pump impeller from the motor prevent excessive mixing of the hot solution in the impeller housing with the cooler solution in the rotor and bearing region. At solution temperatures up to 300°C, the temperature of the solution in the rotor and bearing region is approximately 80 to 110°C.

Seal rings are installed around the front and rear hubs of the closed impeller to reduce pumping loss. These rings are replaceable and can be fabricated from any corrosion-resistant material.

Since the pump thrust-bearing surfaces are small and would not carry a large thrust load, it was necessary to remove or reduce the pump thrust load by hydraulic balance of the impeller under operating conditions. The thrust load was balanced by the combined action of the seal rings at the impeller hubs and radial pads or ribs on the thermal spacer at the rear

Fig. 8. Modified Westinghouse Model 100A Pump.

of the impeller and on the housing at the front of the impeller. Four pads, approximately 2 5/16-in. long, 1-in. wide, and 1/8-in. thick, made by depositing weld metal on the casing walls or by mechanically attached metal ribs, are placed 90 degrees apart on the front and rear impeller casing walls (see Fig. 8). The front and rear pads are 45 degrees out of phase. Leakage through small holes drilled through the impeller hub or eye and past the impeller seal rings tend to equalize the pressure on both sides of the impeller; whereas the weld pads increase the hydraulic pressure between the impeller shroud and casing as the impeller moves closer to the particular casing. The impeller, therefore, seeks an axial balance position. Usually 0.030 to 0.090 in. freedom of movement of the rotor between the front and rear thrust bearings is provided for this purpose. In practice, the position of the impeller, and thus the rotor, is changed by positioning of the impeller until the rotor runs free of the thrust bearing surfaces. The leakage of solution past the impeller hub seal rings can also be changed by increasing or decreasing the clearance between hub and rings, and thus changing the hydraulic pressure balance, as an aid in removing the thrust load. Without the above-mentioned adjustments, thrust loads of more than 200 lb could be attained and would cause excessive thrust bearing wear.

The pump impeller is a closed-vane type (Fig. 9). The vane configuration is shown in Fig. 10. The impeller normally used had a diameter of 9-in. However, its diameter could be reduced by machining when lower head and/or flow rates were desired. The diameter of the impeller could also be reduced in order to pump solutions of higher density without exceeding the pump motor rating. Impellers have been fabricated from several materials as required for corrosion resistance. Among the materials used were type 347 stainless steel, titanium, and Zircaloy-2.

The pump stator is a 3-phase, 220-v induction type, delta-wound with class A Formvar insulation. The pump output could be adjusted, if desired, by changing the motor speed with a variable frequency motor generator set. Cooling oil is circulated through passages in the stator and then through an external oil-water heat exchanger to prevent overheating of the stator windings. Approximately 20,000 Btu/hr were removed in cooling the stator.

As originally purchased from Westinghouse, the 100A pumps contained an Inconel diaphragm separating the pump stator and rotor. In the uranyl sulfate solutions, leakage of solution through the Inconel diaphragm into the stator windings was encountered as the result of corrosion damage. Replacing the Inconel diaphragms with type 347 stainless steel eliminated this problem.

Another source of trouble with the pumps, as originally purchased, was loosening of the front stub shaft of the rotor. In the original design this stub shaft, on which the impeller is mounted, was press-fitted into the iron core of the rotor (see Fig. 11). By redesigning the rotor such that the stub ends passed completely through the rotor body (Fig. 12), loosening of the shaft with a resultant dynamic unbalance of the rotor and impeller assembly was eliminated.

Fig. 9. 100A Pump Impeller. (a) Impeller front. (b) Impeller back.

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Fig. 10. Impeller Drawing Showing Vane Configuration.

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Fig. 11. Original Rotor Design with Press-Fitted Stub Shafts.

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Fig. 12. Modified Rotor with Solid Shaft Construction.

The sleeve-type bearings and journal bushings, which are lubricated by the pumped solution, have been the subject of investigation to improve bearing operating life. The original bearings were Graphitar No. 14 (United States Graphite Co.) with journal bushings of titanium or tungsten carbide with a nickel binder. Titanium and tungsten-carbide bushings were unsatisfactory in the test loops because of excessive corrosion. The original carbide journal bushings were replaced with bushings of cast Stellite 98M2 or Stellite 6 (Haynes Stellite Co.). Most of the pumps now in use contain Graphitar No. 14 bearings and Stellite 98M2 journal bushings, a combination which has given fair service with a mean life expectancy of 2000 to 4000 hr.

Recently bearings and journal bushings of pure sintered aluminum oxide have been under test in several of the model 100A pumps. This combination, installed as shown in Fig. 13, appears superior to the Graphitar No. 14 - Stellite 98M2 combination^{3,4} in that mean bearing life expectancy was increased to more than 10,000 hr based on experience to date.

2.4 Pipe Flanges

A large number of flanged joints were used in the loop piping to provide maximum flexibility for the installation of corrosion test specimens. The flanged joints were also useful as inspection ports to examine interior surfaces. The specially designed joint used on the corrosion test loops is shown in Fig. 14. This type of joint has the advantages of fabrication simplicity, material and space economy, and minimum cracks for solution holdup and/or flow disturbance. It proved to be a very satisfactory, leak-tight joint in this application.

2.5 Sampling Devices

Samples of the loop solution were taken regularly during all operating periods. Special sampling equipment was required since the loops operated at temperatures and pressures up to 320°C and 2000 psia. A high-pressure sampling tank, usually of 30- to 60-ml volume, was used. The sampling tank had a valved opening at each end plus another side opening with a tube fitting as shown in Fig. 15. This side opening was connected to the loop through a valve. A sample was obtained by closing the valves at the ends of the tank and then opening the loop valve. As soon as the tank was filled with solution, the loop valve was closed. After the solution in the tank had cooled, the end valves were opened and the sample drained into a sample bottle. All of the sampling operations were performed through the loop safety shielding by means of extension handles attached to the valves. Solution samples for dissolved gas analysis were obtained

³The Franklin Institute, Investigation of Materials for Water-Lubricated Thrust Bearings -- March 5, 1951 - June 30, 1953, Report F-2243.

⁴H. C. Savage, Sintered Alumina as a Pump Bearing and Journal Material ORNL CF-57-11-122 (Nov. 26, 1957).

Fig. 13. Aluminum Oxide Bearings and Journal Bushings. (a) Aluminum oxide journal bushing. (b) Aluminum oxide bearing.

Fig. 14. 100A Loop Flange Joint.

Fig. 15. Solution Sampling Tank.

similarly except that the tank for sampling had no side opening. An evacuated tank was connected to the loop sampling valve through one of the end valves of the tank (see Fig. 16), and the sample was taken by opening the loop valve and the tank valve. As soon as the tank was filled, these two valves were closed so that the solution and any gas it contained were sealed in the sample tank. After cooling, the tank was connected to an Orsat gas analyzer for determination of the quantity of gas.

The amount of oxygen dissolved in solution was of primary interest since it was required to maintain chemical stability of the uranyl sulfate solution. In some loop tests the amount of dissolved oxygen was maintained at a level of 10 ppm or less. At this low concentration a modified calorimetric Winkler method instead of the Orsat gas analyzer was used for the oxygen determinations.

2.6 Corrosion Test Specimens

To facilitate dynamic corrosion testing of various materials, sample holders were incorporated either in a bypass line or in the main loop piping. A large number of these sample holders could be installed in each loop (see Fig. 1) since two holders containing up to 14 test specimens each could be installed in each of the three flanged sections (Fig. 17) of the main loop piping. Each holder was made in two halves which were clamped together after the test specimens were installed. The inside flow area of the holders had either a round or rectangular cross section and the flow channel was either straight or tapered. Test specimens in the form of flat coupons or round pins (Fig. 18) were used in these holders.

2.7 Hydroclones

Liquid cyclones, or hydroclones, were used for separation and removal of particulate matter (1- to 10- μ size) resulting from corrosion products in the loops.⁵ These hydroclones were also used to separate and identify heavy-phase liquids which could be produced in the event of chemical instability of the solution under test.

In the thorium oxide slurry loops, hydroclones were particularly useful in removing thorium oxide from the loop while operating at elevated temperature and pressure and, in conjunction with the thorium oxide addition system (Sec. 2.2.2), provided a means of regulating the thorium oxide inventory.

For thoria removal and addition the hydroclone was mounted on top of the addition tank which also served as an underflow pot. This unit was connected to the loop through four lines as shown in Fig. 19 to provide

⁵P. A. Haas, Hydraulic Cyclones for Application to Homogeneous Reactor Chemical Processing, ORNL-2301 (Nov. 18, 1957)

Fig. 16. Gas Sampling Tank.

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Fig. 17. Corrosion Test Specimen Assembly as Installed in Loop.

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FLAT-PLATE-TYPE SPECIMENS

TEFLON INSULATION

PIN-TYPE CORROSION SPECIMENS

Fig. 18. Corrosion Test Specimens.

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Fig. 19. Hydroclone as Connected for Slurry Removal from Pump Loop.

maximum operating flexibility. When removing thorium from the loop, a bypass stream of ~1 gpm was circulated through the hydroclone and the solids removed by the hydroclone were collected in the underflow pot. A typical rate of removal of ThO_2 from a slurry loop by means of a hydroclone at elevated temperature and pressure is shown in Fig. 20.

The thorium collected in the underflow pot could be returned to the loop, if desired, by shutting off the hydroclone feed and then displacing the slurry collected in the pot with water from the condensate tank, thus transferring slurry back to the loop via the addition line from the bottom of the tank.

2.8 High-Pressure Letdown and Feed Systems

A letdown valve and diaphragm feed pump were incorporated in some of the loops so that the loop pressure could be controlled hydraulically. This feature was desirable in order to remove the pressure dependence on steam and gas pressure and was particularly useful in loop tests where very low gas (oxygen, etc.) concentration (10 ppm or less) was required; for example, in boiling tests on heated-metal surfaces. The feed pump and letdown valve also provided a means for continuous addition, removal, and/or control of various additives to the solution under test. Within the capacity of the feed pump, the letdown rate was controlled by an air-operated valve from the loop pressure controller.

3. LOOP CONSTRUCTION

The pump loops were designed and operated at temperatures and pressures proposed for aqueous homogeneous reactors (up to 320°C and 2000 psia). In addition, the materials used in the loops were carefully controlled and inspected for quality in order to determine the degree of quality control required for those materials to be used in the reactor itself. Fabricating procedures, particularly welding procedures for the type 347 stainless steel piping, proposed for use in the reactor were also followed.

3.1 Materials

Most of the loops were constructed entirely of type 347 stainless steel, although two loops were of commercially pure titanium. Records were kept of the source, heat number, chemical analyses, and physical properties of all materials used in construction of the loops. For the 347 stainless steel, representative samples of each heat or lot were subjected to the boiling nitric acid test in accordance with ASTM A262-55T and were required to show an average corrosion rate of not more than 2 mils per month. The stainless steel was sensitized prior to test by heat treatment at 1250°F for one hour followed by air-cooling.

Fig. 20. Removal of ThO₂ from a Pump Loop with a Hydroclone.

3.2 Final Tests

After final assembly each loop was hydrostatically pressure-tested in excess of operating pressure, checked for leaks, and the flow rates through the main loop piping and all bypass lines were measured and adjusted as required for each loop test. At this point the thermocouples were installed, pressure gauges and/or cells calibrated, and all instrumentation and pump and heater electrical circuits tested. The hydraulic balance of the pump rotor was adjusted so that the rotor "floated" free of the front and rear thrust bearing surfaces as described in Sec. 2.

4. INSTRUMENTATION AND CONTROL

Instrumentation associated with the pump loops was designed to (1) continuously monitor, and in most cases record, operating variables such as temperature, pressure, and flow, (2) automatically control loop temperature and pressure, and (3) provide safety interlocks to prevent excessive temperature and/or pressure. A photograph of a typical loop control panel is shown in Fig. 21.

4.1 Loop Temperatures and Pressures

Loop temperatures were measured at a number of locations by means of thermocouples attached to the outside surfaces of the pipe or inserted into thermowells. Thermocouple wells were normally used for those thermocouples which controlled the loop main stream and pressurizer temperatures. The temperature of the loop main stream was continuously indicated and recorded on a full-scale electronic instrument which also controlled the temperature by regulating the loop cooling water flow through a pneumatically operated control valve. Heat input to the loop was by the electric Calrod-type heaters and pump work. The pressurizer temperature was controlled by means of the pressurizer heaters.

Loop pressures were continuously indicated and recorded by means of a pneumatic pressure transmitter. On those loops which were hydraulically pressurized, the loop pressure was maintained by the combined action of a high-pressure feed pump and letdown system. Normally the feed rate was held constant and the total system pressure was controlled by the letdown rate through a pneumatically operated valve actuated by the pressure indicator and recorder. On most loops the system pressure was achieved by steam pressurization and/or the combined pressures of steam and gas overpressure in the pressurizer vapor space. In these loops the pressure was controlled by the pressurizer temperature.

4.2 Safety Devices and Interlocks

The safety devices and instrument interlocks incorporated in the pump loops were designed not only for personnel protection but also to prevent damage to the loop components and experimental equipment. In addition to the normal precautions against loop rupture at elevated temperature and

Fig. 21. 100A Pump Loop Panel Board.

pressure, it was necessary to protect against the accidental release of uranyl sulfate and thorium oxide into the experimental area. For this purpose the entire loop was enclosed in shielding made from 1/8-in.-thick steel plates bolted to steel framework. All joints on this enclosure were gasketed to minimize leakage. Further, this enclosure was maintained at a slight negative pressure through an exhaust system which contained a Cambridge Absolute filter element. On the loop itself a rupture disc was incorporated in a line discharged to an appropriate drain line. For those loops which were steam-pressurized, the loop and pressurizer heaters were cut off automatically if safe operating limits were exceeded. Loop and pressurizer heaters were also instrumented to cut off in the event of loss of fluid circulation by pump failure.

For a canned-motor pump it was necessary to maintain a positive suction head to prevent boiling and/or cavitation in the impeller region, which would lead to loss of lubricant for the pump bearings in addition to loss of flow. Thus the control system automatically shut off the pump and heaters in the event system pressure was lost. The canned-motor pump stator windings were oil-cooled, and the temperature in the pump rotor and bearing region was maintained at temperatures below about 125°C in order to prevent bearing damage. To prevent misoperation of the pump cooling system, the instrumentation was interlocked such that the pump and loop heaters could not be turned on unless the pump cooling oil was circulating and pump temperatures were below the specified limit.

5. OPERATING CONDITIONS AND PROCEDURES

All loops were designed to operate at temperatures and pressures to 320°C and 2000 psia. Within these limits operating conditions and procedures for each loop run varied widely and in each case depended on the experimental objectives.

In addition to the corrosion data from the test specimens, important information was obtained on the chemical stability of the aqueous fuel solution (usually uranyl sulfate solution) and handling characteristics of the slurry suspensions.

The performance and reliability of loop components such as pumps, valves, and pipe joints were also of interest since most of these components were similar and in some cases duplicates of those designed for use in an aqueous homogeneous reactor. Thus the operating conditions and procedures were closely controlled throughout each loop run to obtain meaningful data on corrosion, fuel stability, and component reliability.

5.1 Preparatory Operations

After final assembly, each loop was operated initially with a 3 wt % trisodium phosphate solution, circulated at ~100°C for several hours, to degrease and clean the loop piping. After rinsing, this was followed by a 5 wt % nitric acid solution, which was also circulated at 100°C. The

nitric acid solution was sampled regularly and analyzed for corrosion-product buildup to verify the high quality of the materials of construction. During this period all thermocouples, flowmeters, control instruments, heaters, and loop volumes were checked and calibrated.

5.2 Solution and Gas Addition to Loop

The solution to be tested was added to the loop by evacuating the loop to subatmospheric pressure and then the required amount of solution was drawn into the loop from a tared reservoir for careful measurement of the quantity added.

When gas overpressure was required, that quantity of gas (oxygen, hydrogen, etc.) calculated to produce the desired concentration in solution at operating temperature and pressure was added to the pressurizer vapor space before the loop was brought to its operating temperature. This procedure was used for the loop runs in which gas concentrations of >200 ppm were desired. In these cases gas partial pressures of 50 to 400 psi (at operating temperature) were used and the total system pressure was the sum of the gas and steam pressures.

In cases requiring low (0 to ~200 ppm) concentrations of gas in solution, the loop was completely filled with solution and hydraulically pressurized by means of a diaphragm feed pump. The dissolved gas concentration was then maintained by continuous addition from a high-pressure gas cylinder or gas-sparging the contents of the feed tank, which was kept at ambient temperature and at or near atmospheric pressure.

5.3 Operation with Slurry

When slurry tests were first begun, thorium oxide slurry was charged to the loops under vacuum in a manner similar to that used for solutions. This proved to be a very tedious and unsatisfactory procedure because of plugging of the charging lines. Removal of the thorium oxide slurry at the end of each test run proved to be equally unsatisfactory. This required draining under pressure with continuous water-flushing while circulating to prevent the solids from settling out in the loop piping.

Loop operation with thorium oxide slurries was greatly facilitated by the use of a hydroclone in conjunction with the condensate system and slurry addition system. With this equipment the loop was first brought to operating temperature and pressure with water. Slurry was placed in the addition tank and was added to the loop by displacement with condensate as described in Sec. 2.2.2. When a run was to be terminated, the slurry circulating in the loop was routed through the hydroclone, which discharged the solids back into the addition tank (see Fig. 19) where they were collected and retained. It was found that at least 99% of the solids could usually be removed from the loop by the hydroclone. Figure 20 shows the removal rate for a typical removal of thorium oxide from a loop, operating at elevated temperature and pressure, by means of a hydroclone.

6. OPERATING EXPERIENCE

The first pump loop was placed in service in November, 1950. Additional loops were built to meet the program requirements until a total of fifteen loops was available. Until 1955 the loops were used almost exclusively for dynamic corrosion testing with uranyl sulfate solutions proposed for use as fuel for an aqueous homogeneous reactor. In March, 1955, one loop was modified for operation with thorium oxide slurries in support of a program to obtain information on the corrosion and erosion of various materials exposed to circulating slurry and to gain experience in the handling of the thorium slurries proposed as a blanket material in a two-region aqueous homogeneous breeder reactor. Subsequently three loops were used to circulate thorium oxide slurries.

To date more than 330,000 hr of pump-loop operation with uranyl sulfate and 25,000 hr with thorium oxide slurries have been accumulated. Loop tests at temperatures to 340°C have been made. Each loop test was usually of 200 to 500 hr duration, but some tests were operated continuously for more than 7,000 hr.

The basic loop design changed very little over the ten-year period, and most changes were in the nature of auxiliary equipment added for special tests. The basic loop components such as pressurizer, pump, pipe sections which contained the corrosion test specimens, closure flanges and gaskets were completely interchangeable between all loops. This feature greatly facilitated repair and maintenance operations.

Over-all operation of the loops has been entirely satisfactory. Brief comments on operating experience and observations are given below.

6.1 Pumps

Initially considerable difficulty was encountered with the model 100A canned-motor pumps. Pump failures were frequent and usually resulted from excessive bearing wear or electrical failure of the stator windings. Occasionally leaks developed in the metal (Inconel) diaphragm used to isolate the oil-cooled stator windings from the fluid being pumped in the loops. A pump repair program was initiated which resulted in certain changes in the pumps. These changes, along with careful inspection, assembly, and test procedures, increased the average pump life from ~2000 hr to ~15,000 hr. The modifications included: (1) type 347 stainless steel was substituted for the Inconel diaphragms; (2) the rotor shaft was made in one piece and passed through the rotor body; (3) Stellite 98M2 was substituted for the titanium and tungsten carbide journal bushings. Further improvement was later realized by the use of pure sintered aluminum oxide for both the journal bushings and bearings.⁴ Stator winding life was improved by careful fabrication and inspection. Final assembly

⁴H. C. Savage, Sintered Alumina as a Pump Bearing and Journal Material, ORNL CF-57-11-122 (Nov. 26, 1957).

of the pumps was in accordance with rigid procedures and by experienced personnel. Each rotor and impeller unit was dynamically balanced before use. After installation in the loop the pump impeller was hydraulically balanced as described in Sec. 2.3 to remove all thrust load from the bearings.

Periodic checkout of the pumps (usually between each test run) became routine procedure and did not significantly delay or interfere with the test program.

6.1.1 Pump Bearings

The majority of pumps in use in the dynamic corrosion test loops were equipped with Graphitar No. 14 bearings (United States Graphite Co.) and Stellite 98M2 (Haynes Stellite Corporation) journal bushings. Almost all bearing failures were the result of wear of the Graphitar. In general the front bearings were worn much faster than the rear, the average life of the rear bearing being about three times as long. Among the reasons for greater wear of the front bearings are: (1) higher bearing load, (2) higher temperature of the front bearing which is located just behind the thermal barrier, and (3) loss of hydraulic thrust balance of the rotor-impeller assembly usually resulted in a load on the front bearing and caused wear on the front bearing thrust surface.

The results of a survey of bearing failure are shown in Table 1, a tabulation of the number of failures within various time ranges. A maximum life of 8,000 hr for a front Graphitar No. 14 bearing was observed and 17,000 hr for a rear bearing. It can be seen that a large proportion of the bearings lasted more than 2000 hr. Since bearing replacement was a relatively simple operation, and since most of the test runs were of 200 to 500 hr duration, bearing replacement has not been a serious problem.

Table 1. Duration of Graphitar Bearing in Westinghouse Model 100A Pumps

Operating Time (hr)	Bearing Life			
	Front		Rear	
	No.	%	No.	%
<1,000	7	27	1	9
1,000 - 2,000	7	27	2	18
2,000 - 4,000	5	19	2	18
4,000 - 6,000	5	19	3	28
6,000 - 10,000	2	8	1	9
>10,000	0	0	2	18
Total	26	100	11	100

However, it was found that bearings and journal bushings of pure sintered aluminum oxide virtually eliminated bearing failures in the 5-gpm in-pile loop pumps where bearing reliability and long life were essential. Bearings and journal bushings of this material have also been used in the model 100A pumps with similar success, particularly in those pumps used to circulate thorium oxide slurries. Some 24,000 hr of operation in several 100A pumps equipped with aluminum oxide bearings and journal bushings have been accumulated. Only one failure has occurred, and this was due to a cracked journal bushing.

6.1.2 Pump Impellers

Initially pump impellers were of type 347 stainless steel. Occasionally excessive corrosion occurred, particularly in highly concentrated uranyl sulfate solution (>100 g of U per liter). An example of impeller corrosion under these conditions is shown in Fig. 22. For service in concentrated uranyl sulfate solutions titanium impellers were used because of superior corrosion resistance. However, on two occasions burning and melting of titanium impellers occurred. This phenomenon occurred under oxidizing conditions (oxygen gas was dissolved in solution to a concentration of ~ 1000 ppm) and at the time of failure there was evidence of pump cavitation which undoubtedly resulted in a local oxygen atmosphere in the impeller region. The area affected was at the impeller hub where rubbing action against the seal rings probably removed the protective oxide film on the titanium.⁵ Oxidation must have been very rapid and intense, since portions of the oxidized titanium were melted and thrown from the hub.⁶ The hub of one titanium impeller melted in this manner is shown in Fig. 23. This impeller had been in use for more than 12,000 hr under a variety of conditions prior to failure.

The two titanium impeller failures did not preclude continued use of this material, but care was taken to prevent cavitation and rubbing of these impellers.

Impellers of cast Zircaloy-2 have also been used and this material appeared to be more resistant to the erosive action of thorium oxide slurries than either stainless steel or titanium in this application. An example of the erosive action of thoria slurry on a type 347 stainless steel impeller is shown in Fig. 24.

⁵Fred E. Littman, Frank M. Church, Reactions of Titanium with Water and Aqueous Solutions, Stanford Research Institute Project No. SD-2116, Final Report, March 15, 1957 - June 15, 1958.

⁶J. C. Griess, et al., Report on Corrosion of Titanium Impeller Number One, ORNL CF-55-9-65 (Sept. 15, 1955).

Fig. 22. Corrosion of Type 347 Stainless Steel Pump Impeller in Concentrated Uranyl Sulfate Solution.

Fig. 23. Hub of Titanium Impeller After Damage by Oxidation and Melting.

Fig. 24. Erosion of Type 304 Stainless Steel Pump Impeller by Thoria Slurry.

6.2 Loop Piping, Welds, and Flanges

The primary flow circuits of all loops were of either 1-in. or 1 1/2-in. sched-80 pipe. Generally 3/8- or 1/2-in. sched-40 pipe was used for the various bypass circuits. All pressurizers, except those in the titanium loops, were of 4-in. sched-80 pipe. Each loop contained about twelve 1 1/2-in. flanged joints designed to eliminate crevices on the inside bore (refer to Fig. 14).

Welding was by the Heliarc process and all welds were carefully inspected by visual examination, dye-penetrant, and x-ray techniques to guarantee high quality. Special care was taken to ensure full penetration of each weld but without excessive "bleed-through".

Two loops were fabricated entirely of commercially pure titanium and all others were of type 347 stainless steel.

6.2.1 Loop Leaks and Ruptures

From the beginning of the loop test program all failures in the loop piping have been carefully documented. When required, the section in which failure occurred was submitted for metallographic examination to determine the cause of failure. Occasionally loops were completely dismantled and representative samples from various regions of the loop were also carefully examined metallographically for evidence of corrosion damage and/or other latent defects which might be attributed to operational history.

Only one massive rupture has occurred in loop piping, and this was in one of the titanium loops. The rupture occurred in the pressurizer (1-in. sched-40 pipe) made of titanium, and the point of failure was directly under a radiant-type clamshell heater used to maintain pressurizer temperature. The failure occurred when an excessive amount of solution was inadvertently removed from the loop, emptying the pressurizer, and this resulted in overheating of the titanium pipe wall. This produced a ductile failure characterized by swelling of the pipe as shown in Fig. 25.

With the above exception, all failures in loop piping have been in the nature of cracks which resulted in slow leaks. Eight such failures have occurred during the more than 350,000 hr of loop operation. Six were stress-corrosion cracks which in most instances were caused by use of chloride-containing process water for the spray cooler. In these cases stress-corrosion cracking was initiated on the outside surface of the pipe where the water scale deposits served to concentrate chloride (up to 500 ppm). In four of the six cases failure did not occur until ~20,000 hr of operation was accumulated. Table 2 lists all failures documented during operation of the pump loops.

Fig. 25. Titanium Pressurizer on Loop G After Rupture.

Table 2. Tabulation of Failures in Loop Piping

Loop No.	Hours of Operation	Temperature (°C)	Pressure (psig)	Pipe Size and Material	Type Failure	Comments
A	20,000	200-320	200-1500	1 1/2-in. sched-80, Type 347 SS	Stress-corrosion crack	Crack initiated on the inside surface of the pipe. Various uranyl sulfate solutions had been circulating in the loop.
CS	320	300	~1400	1 1/2-in. sched-80, Type 303 SS	Intergranular corrosion	Loop circulated oxygenated thoria slurry. Failure occurred near weld. Type 303 SS was used in error, and welding of this material is known to produce susceptibility to corrosion.
D	20,000	100-300	100-1400	1-in. sched-40, Type 347 SS	Stress-corrosion crack	Crack initiated on outside of pipe which was subjected to cooling-water spray. Scale on outside of pipe contained 250 ppm Cl ⁻ .
G	-	100-300	100-1400	1-in. sched-40 Titanium	Ductile rupture	Pipe wall ruptured due to overheating.
C	8,000	100-300	100-1400	1/2-in. and 3/8-in. sched-40, Type 347 SS	Stress-corrosion cracks	Cracks initiated on outside of pipe which was exposed to wetting by process water.

Table 2 (continued)

Loop No.	Hours of Operation	Temperature (°C)	Pressure (psig)	Pipe Size and Material	Type Failure	Comments
H	19,700	200-300	200-1400	1 1/2-in. sched-80, Type 347 SS	Stress-corrosion crack	Crack initiated on outside of pipe exposed to water-spray cooler. Scale on pipe contained 500 ppm Cl ⁻ .
I _a	3,000	200-300	200-1300	1/2-in. sched-40, Type 347 SS	Stress-corrosion crack	Crack initiated on inside pipe wall. Loop circulating boiler water (H ₂ O + 60 ppm PO ₄ ³⁻ as Na ₃ PO ₄ + D ₂). Last 800 hr with 50 ppm Cl ⁻ added.
I _b	20,000	250	1000	1 1/2-in. sched-80, Type 347 SS (machined from bar stock)	Fatigue	Failure occurred under carbon steel flange welded on outside of pipe.
K	33,800	250	1000	1 1/2-in. sched-80, Type 347 SS (machined from bar stock)	Fatigue	Failure occurred under carbon steel flange welded on outside of pipe.

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