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ISOTOPE KILOWATT PROGRAM QUARTERLY PROGRESS REPORT
FOR PERIOD ENDING JUNE 30, 1971

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REACTOR DIVISION

ISOTOPE KILOWATT PROGRAM QUARTERLY PROGRESS REPORT
FOR PERIOD ENDING JUNE 30, 1971

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AUGUST 1971

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FOREWORD

In 1968 the Oak Ridge National Laboratory started work on a program to evaluate various types of radioisotope energy conversion systems for the production of 1 to 10 kw of electric power for terrestrial and under-sea applications. This program is being carried out for the U.S. Atomic Energy Commission Division of Reactor Development and Technology and the Naval Facilities Engineering Command. The first phase of the program was a parametric and engineering analysis comparing the principal isotope fuels and the principal types of energy conversion system that have been proposed for applications of this sort, and the preparation of a set of conceptual designs for the more attractive systems. That work was completed in the summer of 1969 and was reported in Ref. 1. In October of 1969 ORNL was asked to proceed with a detailed engineering study of the three most promising systems selected from those covered in Phase I of the program. These three systems now under study are a 2-kw(e) thermoelectric system, a 3-kw(e) steam Rankine cycle system, and a 5-kw(e) organic Rankine cycle system.

The first step in the effort was to evolve a program plan for a three-year effort to be carried out in calendar years 1970, 1971, and 1972. The conceptual designs presented in Task I were reexamined and possible improvements were considered with particular attention to the difficult development problems. A variety of engineering tests was considered as a means of evaluating the technology, solving the principal technical problems, and investigating engineering uncertainties that should be resolved before settling on the design of a prototype power plant. In view of the limited funds and the desirability of narrowing the field to a single Rankine cycle system, particular attention was given to the relative merits of the steam and organic Rankine cycle systems.

The first quarter of 1970 was devoted to firming up reference designs for the three types of system, selecting the most crucial experiments required to evaluate the technology, and settling on a program plan for the three-year effort.² With this first quarter's work as a foundation, the second quarter was then devoted to firming up the details of the reference designs, firming up details of the experiments to be conducted, and

preparation of topical reports covering the three reference designs with their associated experiments.³ The subsequent quarters have been devoted to the design and construction of the most urgent experiments.^{4,5} The remaining period will be devoted to the design and construction of additional test equipment and to tests designed to investigate vital questions in the technology. As the results of these tests become available, the reference designs will be revised accordingly. The objective is to evolve by December 1972 a set of designs for two prototype power plants with a clear delineation of the development program required in each case including firm estimates of the cost and time for the various programmatic steps.

This is the sixth in a series of quarterly progress reports. Topical reports present the work carried out in particular areas when key tasks are completed. When a topical report is issued essentially concurrently with a quarterly report, to avoid duplication only a very brief summary of its contents is included in the quarterly.

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ISOTOPE KILOWATT PROGRAM QUARTERLY REPORT
FOR PERIOD ENDING JUNE 30, 1971

1.0 SUMMARY

The organic capsule test continued during the quarter without interruption and has accumulated 4370 hr of operation as of the end of June.

The design review of the one-quarter scale organic fluid decomposition test loop was completed and the Quality Assurance Program plan was revised to meet requests from the ORNL Quality Assurance Office. The instrumentation application drawing was approved and drawings showing the electrical and instrumentation control systems were completed.

Tests with the glass mockup of the quarter-scale organic test loop demonstrate that the boiler flow stability is excellent even with one boiler tube operating at zero power and the other two at full power. Similarly, tests covering a wide range of other highly unbalanced operating conditions have disclosed no undesirable characteristics.

Procurement of hardware for the quarter-scale organic fluid evaluation test loop is underway. The heat block-shield unit has been completed, inspected, and loaded with SrTiO_3 . Radiation dose rates as a function of position are being determined. A purchase order was issued to Liquid Dynamics Corporation for two pumps. Fabrication of the piping has been delayed but will begin in July as soon as the full complement of final approvals have been obtained in conformance with the new Quality Assurance procedures.

Tests of the heat pipe at ORNL under a wide range of power and attitude conditions indicate peculiarities in the performance characteristics. Several hypotheses that have been advanced to explain all of these peculiarities have been investigated. Nondestructive diagnostic tests to determine the reason for its unusual behavior have included radiographs (which revealed a gas bubble in the lower end of the pipe) and eddy current tests. The results indicate that the anomalous behavior of the heat pipe has been caused by one or more bubbles of a non-condensable gas other than hydrogen (probably argon) trapped between the wick and

the outer tube. The potassium will be removed and, after a thorough bake-out, the pipe will be reloaded.

The thermoelectric module test at 3M was restarted on May 5 at 8:45 AM after replacing the heaters, but the hermetic seal at the base of the thermoelectric region failed about 2:30 that afternoon. The 3M Company is to repair the failed joint by the end of July.

Tests of the original wire screen thermal insulation indicated that paint on the wire might increase its emissivity, thus increasing its effective thermal conductivity. New wire screen free of paint and with a sharper melting point was procured and tested, but showed no improvement. Thin (.002 in) sheets of foil of alloy 5052 will be used in an effort to reduce the radiation loss in the next test.

All of the components for the heat block-shield test are in various stages of procurement except for the screen insulation and vacuum tank. Delivery of the major item, the heat block-shield assembly, is scheduled for September 1, 1971. Fabrication of the screen and tank will not be started until the screen thermal conductivity values are obtained with the foil.

2.0 ORGANIC SYSTEM

Capsule Tests

The organic fluid capsule decomposition tests continued at 600°F through the quarter and had accumulated a total of 4370 hr as of the end of the month. There have been no interruptions or problems in the course of the test.

One-Quarter Scale Organic Fluid Decomposition Test Loop

To supplement the results from the static capsule tests reported above, a one-quarter scale version of the organic Rankine cycle system is to be operated with heat fluxes, flow rates, temperature distribution, surface volume ratio, and other pertinent conditions as nearly similar to the full-scale 5 kw(e) system as possible.

Design

Many small delays have been experienced in obtaining all the design reviews and approvals required by the new Quality Assurance Program. Many of these have stemmed from the fact that the program is in the organizing process, and new rules and regulations appear every few weeks. These have led to many changes which, though minor, have each entailed delays. It is expected that the last hurdle in the approval process will be cleared early in July.

Full-Scale Organic Loop

Design work on the full-scale organic loop was started. The system will be essentially similar to the quarter scale loop but will have 12 boiler tubes instead of 3, a turbine-generator-pump unit, and an electrical load. It is being designed for operation in the full-scale heat block-shield.

Glass Loop

The boiling heat transfer and flow stability tests of the glass mockup of the three-tube organic boiler using Freon 113 have been completed. The boiler heat input was varied from about 3000 to 6000 Btu/hr-tube for three degrees of subcooling of the condensate: 22, 43, and 73°F. The boiler pressure varied with the heat input from about 23 to 35 psia. The corresponding saturation temperatures ranged from about 140 to 170°F. The boiler tube exit quality is shown as a function of heat input in Fig. 1. In general, the quality increases with heat input, but the curve for 22°F subcooling exhibits a maximum value at about 4500 Btu/hr. At low heat inputs the quality increases with the amount of subcooling, but the curves intersect, and at the higher heat inputs, the quality falls off as the amount of subcooling is increased. The range of quality is somewhat lower than was expected, but more analysis of the results will be required to determine how these data should be interpreted with respect to the performance of the Dowtherm A boiler.

Procurement

Efforts to procure the low flow-high head canned motor pumps for the facility yielded a number of bids. After reviewing these the order was placed with the Liquid Dynamics Corporation. The pumps will have

Fig. 1. Boiler Tube Exit Quality Versus Heat Input for Freon 113 in Glass Loop.

casing fabricated from a stainless steel forging rather than a casting, the pump shaft will be stainless steel with a tungsten carbide coating, and the bearings will be hardened cast iron. Two pumps of this type are now operating with Dowtherm A. Liquid Dynamics presently has a pump of this design in a test loop at the factory that has over 3000 hr of operation, 1500 hr of which have been in one continuous run, without maintenance. A similar pump is also in operation in the field and according to information from the buyer has given satisfactory performance during intermittent operation for a period of approximately two months.

The heat block-shield assembly has been fabricated, inspected, and delivered to the Isotopes Division. The SrTiO_3 fuel capsules have been fabricated and loaded in the shield. A radiation dose survey over the exterior of the shield has been completed and the dose rates have been found to be satisfactory. A detailed calibration of the dose rates in the holes for the boiler tubes will be completed in July.

3.0 THERMOELECTRIC SYSTEM

Heat Pipe Tests

Testing this quarter has been concerned with determining the cause of the anomalous behavior of the heat pipe. The instrumentation difficulties that were experienced in the previous quarter have been corrected and good heat balance data have been obtained. However, a number of peculiarities in the heat pipe performance have been noted. The temperature structure in the evaporator region varies erratically depending on the inclination of the heat pipe and the heat loading. There are four thermocouples in the evaporator region, one in each of the intervals between the three heaters, and one at either end of the heated section. With the heat pipe in any position between vertical with the condenser end up and horizontal, the temperature is essentially uniform throughout the length and the heat transport capacity is high. If the evaporator end of the heat pipe is raised above the condenser end, however, the temperature distribution diverges and the heat transport capacity of the heat pipe drops sharply. See Fig. 2.

Fig. 2. Axial Temperature Distribution for the Heat Pipe in Two Typical Positions, i.e., with the Condenser End Inclined Upward with a Slope of +1.5 deg and with the Condenser End Inclined Downward with a Slope of -10 deg.

Possible Causes of Trouble

In attempting to diagnose the difficulty, the first thought was that there might be an inert gas, particularly hydrogen, present in the heat pipe. J. E. Kemme of Los Alamos, an outstanding expert on heat pipe performance, visited ORNL for two days and assisted in the diagnosis; he favored the theory that there was noncondensable gas in the heat pipe. Another hypothesis advanced was that the wick structure was not adequately sealed to the wall at the evaporator end of the heat pipe. As a consequence, the wick could lose its prime if the condenser were lower than the level of the boiler. In fact, even with the condenser above the evaporator, the temperature structure in the evaporator region could become erratic at high powers.

The presence of an inert gas bubble between the wick and the heat pipe wall could also act to break the suction formed by the capillary pores in the wick and thus would negate the strong pumping effect of these $30\text{ }\mu$ pores. If this occurs with the condenser above the evaporator, gravitational forces will still act to recirculate the liquid, and there may be no observable effect on the heat transport performance. However, if the condenser is below the evaporator, gravitational forces will act to resist recirculation. The only force acting to produce recirculation from the condenser to the evaporator would be the capillary force acting on the free liquid surface between the wick and the heat pipe wall. The nominal radial gap at this point is 0.017 in., but the wick is free to be eccentric, hence the gap on the close clearance side is probably only three to five mils. Inasmuch as the $30\text{ }\mu$ pore size is about 1 mil, the capillary force would drop to perhaps 25% and the heat transport capacity of the heat pipe would be cut by the same factor. Note that the wick is made up of 10 or 12 layers of screen, hence capillary forces within the wick will act to distribute the liquid circumferentially around the wick.

If one examines Fig. 2 with this rationale in mind, it appears that the experimental data can be explained in this way. The large increase in temperature of the evaporator when it was raised above the condenser implies a pronounced increase in heat losses and consequently a reduction in the heat input to the heat pipe. Similarly, the drop in the condenser

temperature--from 420°C to 320°C--indicates a reduction in the heat transport rate of perhaps 50%.

The surface tension of the alkali metals is reduced if the oxygen content is increased. Further, a film of chromium oxide would tend to reduce the wetting action. Either (or both) of these effects could act to break the capillary film in the wick and produce essentially the same effect as outlined above for a gas bubble.

Non-destructive Tests

To investigate these problems it seemed eminently desirable to evolve a set of non-destructive tests that could be employed for inspecting heat pipes in production. Thus the first step taken was to remove the heat pipe from the test stand and radiograph it. When examining the films it was noted that the interior details of the heat pipe were easy to discern. The annulus between the porous tube and the outer tube can be seen but it is not possible to tell whether or not the annulus and porous tube were filled with potassium. The pool of excess potassium at the bottom was easily seen and the amount of excess potassium was estimated.* It appears that the excess pool contains ~0.9 in.³ more than one would expect if the wick and annulus were completely full, thus implying a void behind the wick. The radiographic films also revealed the presence of a void about 11/16 in. in diam between the tapered plug and the end cap at the fill-end as shown in Fig. 3.

The heat pipe was then subjected to a phase-sensitive eddy current test. This test measures the local conductivity of the material being tested. In this case we hoped that it might show the effects of whatever is causing the anomalous behavior of the heat pipe. Inquiries indicate that this technique has not been used to study heat pipes. In the absence of prior experience or a test standard, it is difficult to analyze the results of such a test. The test did indicate that there was a basic eccentricity in the pipe (probably the eccentricity of the

*When the heat pipe was filled initially it was loaded with 20% excess potassium to insure an adequate supply of the working fluid.

Fig. 3. Heat Pipe - Evaporator End.

porous tube in the outer tube) with other eccentricities superimposed over various portions of the length along the pipe. The eddy current test will be repeated when the fault has been found and corrected. It may be that such a test will prove to be an effective check on the conditions of a heat pipe.

Tests in a Muffle Furnace

At this point it was decided that the time lost in installing and removing the heat pipe from the test facility could be reduced by conducting the performance check tests in a tilting muffle furnace in the Materials Compatibility Laboratory at X-10. By using this facility it has proved possible to reduce the turn-around time between the various operations on the heat pipe and the analytical tests following each of the operations from about a week to about one day. After an initial performance test to familiarize ourselves with the heat pipes operation in the muffle furnace, the joint between the wick and the outer wall at the evaporator end of the heat pipe was rerolled. (See Fig. 3) The heat pipe was then reinstalled in the muffle furnace, but the performance was found to have deteriorated. A new radiograph showed that the second rolling operation had acted to drive the plug out from under the rolled region. Fortunately, there was still room for a second rolling operation farther toward the end of the heat pipe, and this operation was carried out. Retesting of the heat pipe showed that the performance was restored to the level obtained before the end plug had been dislocated. The following operations were carried out on the heat pipe during June in an effort to eliminate hydrogen and/or argon which (as a non-condensable gas) was thought to be the cause of the trouble:

1. Vacuum outgas at 1600°F for 13 hr to allow any hydrogen to diffuse through the heat pipe wall.
2. Performance test the heat pipe.
3. Vacuum outgas again at 1600°F for 13 hr.
4. Performance test the heat pipe.
5. Cut off the pinched-off portion of the fill tube and install a valve. Connect the heat pipe to a vacuum system and re-evacuate to remove any non-condensable other than hydrogen, e.g., argon.

6. Performance test while connected to vacuum system.

Each of the above operations resulted in some improvement in the heat pipe performance, but none of them achieved a complete cure for the loss in output that occurred when the condenser end of the heat pipe was dropped below the evaporator end.

A likely explanation for the loss of performance when lowering the condenser is that a non-condensable gas other than hydrogen (most likely argon) has been trapped between the back of the porous tube (or wick) and the wall of the outer tube. Such a gas bubble could have entered when the heat pipe was backfilled with argon following the heater failure of the previous quarter. (The heater arced to the heat pipe wall leaving a thin spot that was repaired by building it up with weld metal.) To eliminate this trapped gas, the potassium will be distilled from the heat pipe, the heat pipe will be thoroughly baked out and potassium will be reloaded into the unit. The necessary hardware and preparation for this next step has been made and it should be carried out early in July.

Effects of Radiation

Conversations with J. E. Kemme from Los Alamos during a visit to ORNL around the first of April disclosed some interesting points. There has been considerable question from time to time as to whether intense radiation would effect nucleation of a liquid metal operating in a heat pipe and possibly lead to the formation of a vapor blanket in the superheated liquid between the annulus and the wick in the type of heat pipe chosen for the ORNL work on this program. To resolve this question Los Alamos built a one meter long heat pipe of niobium filled with lithium following the type of construction employed in the ORNL heat pipes. This unit was installed in the UTREX reactor at Los Alamos and operated upside down at 1100°C. The heat pipe started well with no problems and the power rose to its normal value in two successive starts. The test was discontinued for lack of funds, but clearly demonstrated that intense neutron and gamma radiation did not break the capillarity by inducing boiling in the superheated liquid in back of the wick.

Use of a Getter

J. R. Kemme also stated that when the metal vapor condenses in the condenser section of the heat pipe it tends to remove all impurities from the condenser region and transport them to the evaporator section. The Los Alamos work on this problem indicates that in lithium-filled heat pipes a zirconium getter in the filler stem is very helpful and a yttrium getter in the filler stem is even better. The latter material should also be effective in potassium. Future heat pipes in this program should include a getter system.

Thermoelectric Module

The breakage of heater leads caused by sulphur and cadmium contamination when the thermoelectric module was being subjected to its initial tests at the vendor in March led to an extensive detailed examination of the parts involved to determine the extent to which sulphur might have attacked the thermoelectric module.⁶ An intensive examination of the components in the heater region of the thermoelectric module indicated that there was general mild sulphur contamination throughout the region but that it was most severe at the heater end. Other than the loss of time, the principal ill effect appeared to be possible embrittlement of the nickel hot shoe in the vicinity of the seal weld between the hot shoe and the 0.020 in. thick end bell which seals the region around the junctions and prevents air or moisture from attacking the junction material. It was finally decided that there was no better course of action under the circumstances than to replace all of the components that had been exposed to the sulphur and cadmium attack, where possible, and thoroughly abrade and clean the balance prior to reassembly and startup of the system.

On May 6, 1971, the 3M Company restarted the system at 8:45 AM. That afternoon, about 2:30 PM, it was noticed that the pressure in the thermoelectric generator was decreasing. 3M Company personnel continued to operate the system until 4:45 PM. At that time they turned off the electrical heaters and allowed the unit to cool off. At the request of ORNL, the 3M Company had installed a valve on the top plate

of the generator and were thereby able to maintain an atmosphere of pure argon in the generator until it had reached room temperature so that the thermoelectric junctions themselves were not damaged. The total operating time was 18 hr. The hermetic seal apparently failed as a result of excessive sulphur embrittlement of the nickel hot shoe.

The system was being operated at a temperature level that would bring the hot junction temperature in the generator to 1200°F. (The normal operating temperature of the hot junction will be 1000°F. The 1200°F temperature is necessary for part of the 3M Company's proprietary processing operations.) Information has been received from 3M Company indicating that the heat pipe temperatures were about 1500°F.

The 3M Company is repairing the unit and expects to complete the repairs and acceptance tests and have it ready for delivery to ORNL by the end of July 1971.

4.0 THERMAL FUSE AND INSULATION

Thermal conductivity measurements on a test specimen of the original aluminum alloy (alloy 5056) at reduced pressures have been completed. The thermal conductivity at five pressure levels is shown as a function of the mean insulation temperature in Fig. 4. The results indicate that natural thermal convection contributes to the thermal conductivity at pressures above about 1 psia, but at pressures below that the thermal conductivity falls off because of the effect of the increased molecular mean free path on the thermal conductivity of the air.

All of the results give higher values of thermal conductivity than those predicted from theoretical considerations. It is thought that these differences may be accounted for by the effects of an aluminum pigmented paint, which had been applied to the screen during manufacturing. Although an attempt was made to remove the paint in a hot cleaning solution, it was not successful, and apparently much of the paint remained on the screen. The paint boiled off the screen during testing, leaving a dark deposit on the screen, and condensed on the cooler outer layers of the insulation. The paint may increase the

Fig. 4. Thermal Conductivity Versus Mean Insulation Temperature at Reduced Pressures of Aluminum Screen Insulation.

thermal conductivity of the screen in two ways. First, the dark deposit on the screen in the hot portion increases the emissivity of the wire and increases the radiant heat transfer. Second, the solid and liquid residue decreases the contact resistance between the wires and increases the heat transfer by conduction. The predicted values of thermal conductivity for an assumed emissivity of 1.0 for the wire were in close agreement with the measured values at 1.25 psia, indicating that it is quite possible that the paint caused a high emissivity.

New screen made from alloy 1100 was obtained and a new test specimen was prepared. The insulation specimen was made with an ID of 1 5/8 in. and an OD of 5 5/8 in. It was wound on a cylinder made from a single layer of heavy stainless steel wire screen. The inner screen has a wire diameter of 0.032 in. on a mesh of 4 x 4 wires per inch and has an ID of 1 1/2 in. The stainless steel screen was painted with PT404A ceramic silicone paint to give it a high emissivity and to protect it from possible attack by the aluminum at high temperature. Thermal conductivity tests at three pressure levels were disappointing; the test results were the same as those for the original screen material. Contrary to what was expected, the lower emissivity of the bright surface of the wire in the new specimen did not reduce the high values of thermal conductivity found earlier with the old specimen that had been discolored from decomposition of a coating of aluminum-pigmented paint. Apparently the effective emissivity of the screen matrix is controlled by the geometry and void volume fraction, and is virtually independent of the surface emissivity of the wire itself. Analysis of the data indicates that an effective emissivity of 0.9 - 1.0 would be required to yield the measured values of thermal conductivity.

While the thermal conductivity is low enough to be adequate for the organic system, further improvement is needed to keep the heat losses to the desired level with a reasonable insulation thickness for the higher temperature conditions of the thermoelectric system. Since the radiant heat transmission through the insulation is higher than was expected, it appears that the greatest improvement could be achieved by placing a number of solid sheets of aluminum foil spaced

several layers of screen apart in the insulation. In order to prevent overheating of the block, the aluminum foil would be made of an alloy having a lower melting temperature than the screen. The first layer of foil would be placed inside several layers of screen so that, when the screen began to melt, the foil would reach its melting point when several layers of screen were still intact between the foil and the heat block. It is hoped that this will give a high effective emissivity during the entire process of meltdown.

It is now planned to incorporate aluminum foil in the next test specimen. An order has been placed for 5052 alloy foil with a 0.002 in. thickness and a 48 in. width. This material is expected to be delivered the last week of July. In the meantime, the thermal conductivity and meltdown tests should be completed on the present specimen.

5.0 HEAT BLOCK-SHIELD TEST

The contract for the 17,000 lb full-scale heat block-shield unit has been placed and delivery is scheduled for September 1, 1971. The contract includes inspection and certification that all dimensional tolerances have been met. An ORNL inspector will be sent to the vendor in Dayton, Ohio, to observe the final inspection. The simulated fuel capsules are being made of centrifugally cast 310 stainless steel pipe. These will enclose the electric heaters. Delivery is scheduled for August 15, 1971.

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