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THORIUM FUEL CYCLE DEVELOPMENT PROGRESS REPORT

OCTOBER 1970

NO. 15

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CHEMICAL TECHNOLOGY DIVISION
METALS AND CERAMICS DIVISION

THORIUM FUEL CYCLE DEVELOPMENT PROGRESS REPORT

October 1970

No. 15

I. HEAD-END REPROCESSING DEVELOPMENT

P. A. Haas, R. S. Lowrie, V. C. A. Vaughen

II. REFABRICATION DEVELOPMENT

R. G. Wymer, A. L. Lotts, R. E. Brooksbank, P. A. Haas

III. TURF EQUIPMENT DESIGN AND ENGINEERING

J. W. Anderson, J. W. Snider, J. D. Sease

IV. MATERIALS IRRADIATION

A. L. Lotts, T. N. Washburn, J. D. Sease, J. H. Coobs

NOVEMBER 1970

OAK RIDGE NATIONAL LABORATORY
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Previous issues of this report are as follows:

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4. ORNL-TM-2765, November 1969
5. ORNL-TM-2792, December 1969
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THORIUM FUEL CYCLE DEVELOPMENT PROGRESS REPORT

OCTOBER 1970

No. 15

Compiled by

The Staff of the Chemical Technology and
Metals and Ceramics Divisions

INTRODUCTION

The thorium fuel cycle development at ORNL is directed almost solely at HTGR fuels. These fuels consist of large blocks of graphite containing coolant channels and fuel and blanket holes. The fuel and blanket are made of microspheres of uranium or thorium compounds separately, or of mixtures of them in a single microsphere. The microspheres are coated with layers of pyrolytically deposited carbon and in some cases silicon carbide. The microspheres are retained in the holes in the graphite blocks in a bonded state.

Development work on all aspects of HTGR fuel recycle is in progress at ORNL. In addition, a major recycle development facility, the Thorium-Uranium Recycle Facility (TURF), has been built at ORNL, and the Coated Particle Development Laboratory (CPDL) has been put into operation in Building 4508. TURF is intended to be used as a development facility for fuel recycle. The CPDL is for engineering development studies leading to design of the equipment to be used in TURF.

I. HEAD-END PROCESSING DEVELOPMENT

The objective of this program is to evaluate head-end processes for converting irradiated HTGR fuels to a form suitable for recovery and decontamination of the thorium and uranium by a solvent extraction process. Small samples of irradiated and unirradiated fuel are studied to determine effects of irradiation on fuel reprocessing steps and to correlate with metallographic studies. An important objective is the determination of the amount of breakage of particle coatings and the resultant amount of cross-contamination of the fertile Th-^{233}U and fissile ^{235}U components in alternative processing steps. Mechanical systems are being developed for degrading the fuels and providing a material suitable for use in studies of the burn-leach steps using fluidized-bed or fixed-bed burners. The mechanical and burn-leach engineering development work is carried out using full-scale unirradiated fuel of the type to be used in PSC, and is designed to provide scale-up data for use in the design of pilot or full-scale processing plants.

1. Studies with Irradiated Fuels

(V. C. A. Vaughen, J. H. Goode, G. Davis)

Procurement of Dragon Fuels

The unirradiated compacts have arrived from Dragon. There has been a little difficulty in trying to correlate the code numbers on the compacts we received with the compacts we requested. We are presently trying to resolve the problem this has caused.

2. Head-End Engineering Studies

(R. S. Lowrie)

The head-end engineering studies comprise two principal areas of investigation - "mechanical" and "burn-leach." The general approach followed has entailed the comminution of a fuel element to produce suitable feed material for the burn-leach process. A number of

changes have occurred in the overall HTGR program during the past year and some of them, such as the recent decision of GGA to use bonded fuel sticks instead of loose particles in HTGR fuel elements, has led to changes in our ideas of the mechanical equipment required to reprocess spent reactor fuel.

2.1 TURF Process Flowsheet Calculations (R. S. Lowrie)

Detailed process flowsheets have been prepared showing the compositions of inlet and outlet streams for the head-end, acid-thorex solvent extraction, sol formation, and microsphere preparation steps during the hot demonstration run in TURF.

During the past month we have been analyzing the problems associated with handling and disposing of the various process waste streams from the head-end operations. It is clear that gaseous wastes will be our overriding problem. The flowsheets and waste problems will be presented and discussed in next month's report. In the meantime, we have resumed fluidized bed burner studies. Here, our major problem is getting prototype fuel to work with.

II. REFABRICATION DEVELOPMENT

1. Particle Preparation

The fuel material of primary interest for ORNL recycle studies is the ThO_2-UO_2 particle, which has a thorium-to-fissile uranium ratio of about 4.2. The uncoated fuel particles are to be microspheres 350 ± 100 μ in diameter, made by the sol-gel process. Fuel preparation includes development and demonstration of all process steps involved in making remotely the ThO_2-UO_2 microspheres. The steps include demonstrating reliable, remote processes for reproducibly mixing $Th(NO_3)_4$ and $UO_2(NO_3)_2$ solutions in the desired thorium-to-uranium ratio, preparing the mixed, stable ThO_2-UO_3 sol in concentrations exceeding 1 g-mole of oxides per liter, forming ThO_2-UO_3 gel microspheres, and converting them to dense ThO_2-UO_2 in good yield.

Experimental studies of processes and equipment for preparation of sols and microspheres are reported here. The present emphasis is on processes, procedures, and prototype equipment for preparation of oxide microspheres in the Thorium-Uranium Recycle Facility (TURF). Tests of flowsheets and procedures to be used for test materials preparation are also included.

1.1 Sol and Microsphere Preparation Development (P. A. Haas)

The still used for alcohol purification and recycle in the Bldg. 4505 sphere preparation system was rebuilt with larger heat exchangers and a larger liquid-vapor disengaging section. The rebuilt still will be adequate for larger flows to allow either lower equilibrium water concentrations in the 2EH or higher sol feed flows to the sphere forming column. It will also be more suitable for test of recovery of 2EH by complete distillation; tests in the previous still were limited by excessive carryover of surfactants--probably by foaming.

1.2 Sphere-Forming Column Chemistry (W. D. Bond, J. W. Snider, P. A. Haas)

Our previous studies have shown that pH, surfactant concentration and water content of 2-ethyl-1-hexanol (2EH) are important variables in the microsphere forming process. We are presently examining methods for improving the control of pH and surfactant concentration during continuous operation of sphere forming columns. Our present system for controlling the water content is adequate. For water content control, a portion of the used solvent is heated at 150-160°C to remove most of the water, and the desired water content is obtained by mixing this relatively dry 2EH with the remaining wet solvent.

In the past, sphere forming columns have been operated continuously by making periodic additions of surfactants to off-set the losses of surfactant caused largely by reaction of the nitric acid (extracted from the sol) with the surfactants during removal of water by distillation. In addition to reactions during distillation, a relatively small amount of reaction occurs in the forming column as evidenced by slow discoloration of the solvent when the forming column is batch operated without a distillation system. The pH or acid contents of the 2EH that have been employed in continuous operation, have been mainly those governed by the amount of acid extracted from the sol which can subsequently be maintained in the 2EH with the distillation system operating. When higher acid contents are required, nitric acid must be added to the 2EH and reactions with surfactants and 2EH are increased. Laboratory studies have shown that reactions of nitric acid during distillation could essentially be eliminated by removal of the acid prior to the distillation step by ion exchange. The acid content of the alcohol could then be maintained by adding nitric acid to the alcohol after the removal of water by distillation. We are presently evaluating this procedure as a means of controlling the pH and surfactant concentration of the UNOP sphere forming column during continuous operation. Studies are continuing on the effect of surfactant and water content, and the pH of 2EH on sphere forming using different sols in batch operated laboratory forming columns.

Recovery of 2EH for Recycle (W. D. Bond, C. W. Greene)

We are studying a distillation process for purifying 2EH for recycle to the sphere-forming column. The 2EH is recovered from surfactants and degradation products by distilling 2EH and water from the used solvent at 180°C and then the water content is adjusted by a second distillation at 160°C. Results continue to show that the recovered 2EH + H_2O is free of surface active materials. However, gas chromatographic analyses show that the recovered 2EH is of 99.39% purity as compared to 99.93% for the 2EH as received from the manufacturer. This amount of impurity is similar in amount to the larger amounts of surfactant we add. Thus, it may be important. The impurity is judged to have a lower boiling point than 2EH from its order of elution in the gas chromatographic analysis. Experiments are in progress to remove the impurity by further distillation.

1.3 Equipment Development and Design (P. A. Haas, C. C. Haws)

We are reviewing the flowsheet requirements for firing sol-gel spheres in order to select the equipment for development studies. For ThO_2-UO_2 spheres, we need to demonstrate the firing flowsheets for 12 kg/day of oxides in TURF. A continuous, push-through type of muffle furnace is practical for this scale of operation. We can probably demonstrate operation of this type of continuous furnace as easily as we can demonstrate the long-term durability and dependability of the alumina pot and hydraulic transfers which we used for the last ThO_2-UO_2 preparations. The continuous firing is preferred for ease of scale-up. Therefore, we plan to purchase a standard push-through furnace of about 4 in. ID for testing.

Recent firing tests for large UO_2 spheres show a need for better atmosphere control than is possible in our batch muffle furnace. We are adapting high density alumina tubes for test firing of 1/2 to 1 kg batches of UO_2 spheres.

1.4 Preparation of Test Materials (P. A. Haas)

Fabrication of test elements for HTGR head-end reprocessing studies will require 5 to 10 kg of 100 μ natural UO_2 spheres. We plan to prepare these particles from CUSP sols in our non-fluidized column system. The cooling water piping to the still for this column was altered to allow operation with hot alcohol. For n-hexanol, operation at 39°C gave less of the pitting or dimple-type distortion than operation at 28°C, but the difference was small. While our column was not tall enough to complete gelation in 2EH at 28°C, mass transfer calculations indicate that we can make these spheres in 2EH at 40°C.

2. FUELED GRAPHITE FABRICATION DEVELOPMENT

(F. J. Furman, W. H. Pechin, C. B. Pollock,
J. M. Robbins, and J. D. Sease)

We are developing processes and equipment for the refabrication of HTGR fuel as detailed in the National HTGR Recycle Development Program Plan. The fuel consists of microspheres of thorium and/or uranium as the oxide or carbide, coated with multiple layers of pyrolytic carbon and silicon carbide. These particles are loaded into hexagonal graphite logs which contain both fuel and coolant holes.

Our work is divided into particle coating, particle handling and inspection, fuel stick making, and element assembly. Last report period we concentrated on particle coating development. We continued the work reported last period on modifying the prototype coating furnace and continued a series of tests to establish load limits of a 5-in.-diam coating furnace, paying particular attention to coating HTGR reference recycle particles with a minimum of soot inclusions and faceting. We began setting up a fuel stick fabrication line.

2.1 Particle Coating

We are investigating how variation of the parameters used in coating reference recycle HTGR fuel particles affects the quality of coatings and furnace cone life. Specifically, we are interested in minimizing faceting and sooting in propene coatings. We have established the densities which result from variation of propene flux and furnace temperature for a 700 g starting load for our 5-in.-diam laboratory coater. The resultant curves are shown in Fig. 2.1. These curves apply for coating with no dilution of the hydrocarbon gas. We do not yet have sufficient quantitative data to analyze the effects of these coating parameters on particle shape, but we now have methods for shape determination as described in Section 2.2 and we will collect the same type of information for particle shape as is shown for coating density in Fig. 2.1.

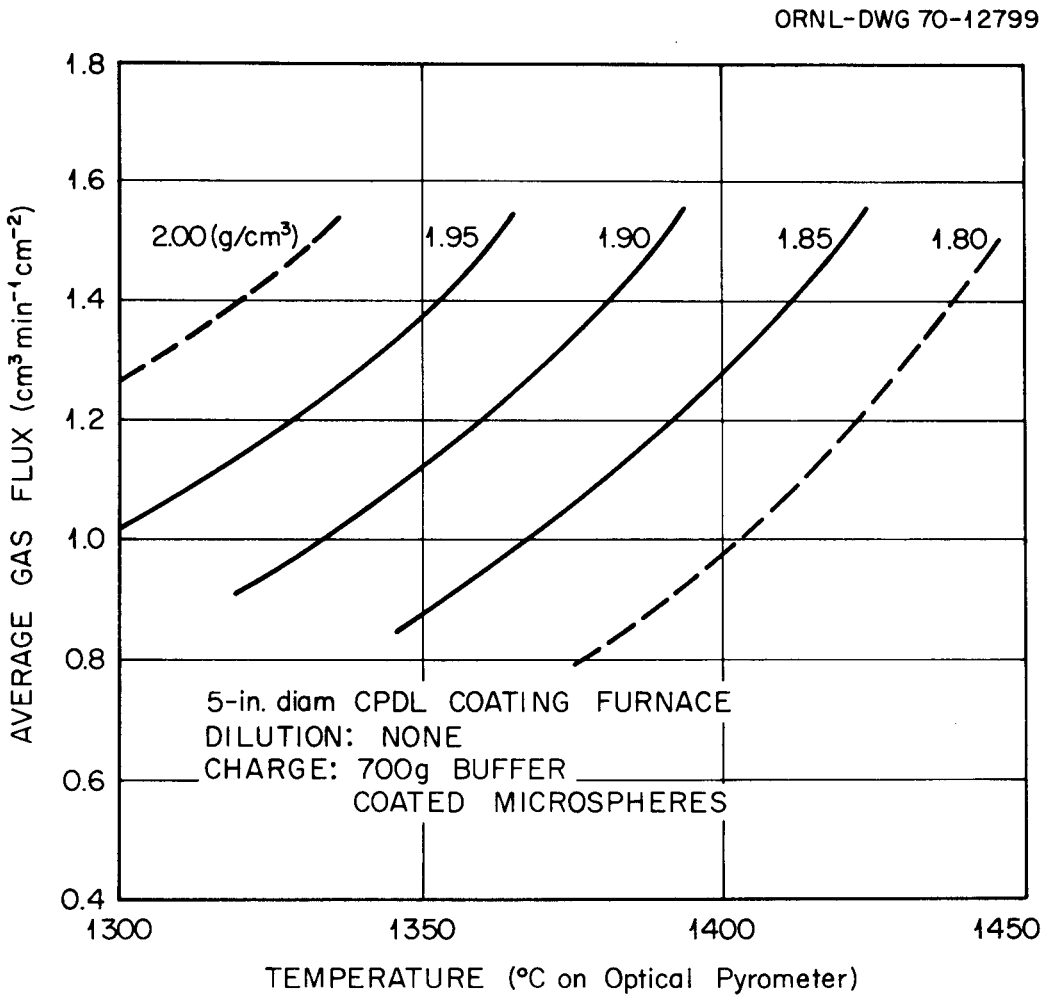

Fig. 2.1. Density of Propene Coatings.

2.2 Particle Inspection

We have been investigating the measurement of particle shape. Two items are of interest: the geometric shape of the final particle and the variation of coating thickness at various points on the final particle. The particle shape is of interest because of its effect on the packing of particles into molds prior to injection of binder in the fabrication of fuel sticks. The variation of coating thickness from point to point on the particle is of interest because of its expected influence on the irradiation performance of the particle. The shape of a solid particle is most easily characterized by the ratio of its maximum-to-minimum dimension. For simplicity we measure the maximum and minimum dimensions of the two-dimensional projection of the particle on a microradiograph. On the same microradiograph we can measure the maximum and minimum coating thickness of each particle.

If the kernels are essentially round (shape factor = 1.000), then the particle shape factor can be determined from the ratio of maximum-to-minimum coating thickness. If shape is determined in this manner, however, the shape factor is also a function of the coating thickness. The analytical relationship is

$$F_3 = \frac{T_{max}}{T_{min}} = \frac{F_1\left(\frac{D_{av}}{D_k}\right)-1}{\left(\frac{D_{av}}{D_k}\right)-F_1}$$

where

T_{max} = maximum coating thickness
T_{min} = minimum coating thickness
D_{av} = average particle diameter
D_k = average kernel diameter
F_1 = ratio of maximum particle diameter to minimum particle diameter

The point here is that a given T_{max}/T_{min} ratio represents a more irregularly shaped particle when the coating thickness is large than it

does for a thinly coated particle. At the present time we are primarily concerned with the effects of coating parameters on particle shape; therefore, we will judge particle shape on the coating thickness ratio T_{max}/T_{min} as measured from the microradiograph. This is a somewhat more laborious method than the tilting plate which was described in the last report of this series and we hope a comparison of data from the two methods will allow us to rely more heavily on the tilting plate, especially in situations where great precision is not required.

2.3 Fuel Stick Fabrication

We are constructing an HTGR fuel stick fabrication line with a production capacity of up to 400 in. of fuel sticks per day. The line will consist of a coated particle batch blender for preblending the fissile and fertile particle batches separately, a combination fissile-fertile particle blender and fuel stick mold loader, fuel stick molds, mold heater, pitch injector, and fuel stick ejector. The batch blender has been designed and the parts are being fabricated. The combination mixer-loader has been fabricated. Initial testing will begin this month. The fuel stick molds are available, although they were originally for 12-in. fuel sticks and must be modified for shorter sticks. A mold heater is available. The pitch injector is being fabricated from a booster pump. The pump is being mounted and the auxiliary hardware assembled. The pump for the fuel stick ejector is on hand. We expect to complete most of the equipment fabrication during the next month and to begin testing the individual components.

III. TURF EQUIPMENT DESIGN AND ENGINEERING

(A. L. Lotts, J. W. Anderson, J. D. Sease, J. W. Snider, and J. M. Chandler)

The objective of this part of the program is to design, to construct, and to perform operational testing of three pilot plants to demonstrate recycle of HTGR fuels. These pilot plants are the Head-End Reprocessing Pilot Plant, Acid Thorex Pilot Plant, and the Refabrication Pilot

Plant. The Head-End Pilot Plant and the Refabrication Pilot Plant will be installed in Building 7930 and the Acid Thorex Pilot Plant will be installed in Building 3019. The design of these pilot plants will include processing equipment, materials handling equipment, waste treatment and disposal equipment, and modifications of the facilities of Buildings 7930 and 3019. These engineering efforts will include major processes and equipment involved in recycle Head-End, Solvent Extraction, Sol-Gel, and Refabrication, as well as Special Tasks such as waste and material handling and shipping. These are reported subsequently under their respective subsections.

General project activities during the reporting period included a continued effort toward updating the National HTGR Recycle Development Program Plan and the preparation of simplified process flow diagrams, task schedules, budget estimates, and illustration figures to be included in the program plan. The program letter containing budget information for the development program was received from RDT. A budget review was prepared summarizing the allocation of funds for the task within the program.

1. Head-End Reprocessing Equipment

Flowsheets summarizing the process proposed for the pilot plant were completed and made ready for inclusion in the program plan. A space utilization study for the equipment to be installed in Cell G, Building 7930, was begun and preliminary equipment layouts were developed utilizing Cell G and a portion of Cell C. To prepare these equipment layouts, some very preliminary conceptual equipment designs were prepared for estimating space requirements.

2. Solvent Extraction Equipment

Flowsheets summarizing the process proposed for the Acid Thorex Pilot Plant were completed and made ready for inclusion in the program plan. The overall schedule for performing the reprocessing demonstration task was completed for inclusion in the program plan.

3. Sol-Gel Equipment

Flowsheets summarizing the process proposed for this part of the Refabrication Pilot Plant were completed and made ready for inclusion in the program plan. The overall schedule for performing both the development and the demonstration portions for this program task was completed and made ready for inclusion in the program plan.

4. Refabrication Equipment

Included in this subtask is the remainder of the reprocessing equipment to be installed in the Refabrication Pilot Plant not included in Subsection 3 above. General work in this area during the reporting period included the development of a revised schedule of this subtask to be included in the program plan. A preliminary drawing list was prepared and a system for numbering documents that will be developed during the design of pilot plant equipment was established. Meetings were held with program personnel to establish a series of steps to guide the design work. During these meetings it was agreed that the proposed fuel stick fabrication machine described in the last report of this series would be designed in detail, fabricated, and tested.

4.1 Refabrication Equipment Design

(W. A. Pate, F. C. Davis, and D. D. Cannon)

The proposed fuel stick fabrication unit is undergoing final design and will be detailed for fabrication. This work will require ∿5 to 6 weeks to complete the drawings and specifications required for shop fabrication. The conceptual design of the remotely operated fuel element loading machine is progressing. This concept should be completed by the end of the month. The problem of obtaining a representative fuel particle sample from a large batch is being investigated. Two types of samples are being considered at this time. The first is a multistage riffler and the second is a multistage

conical divider. It is planned that development equipment will be designed so that both concepts can be tested through laboratory testing.

5. Special Tasks

5.1 Material Handling

A brief study was prepared with a budget estimate of the cost involved in performing a very minimum demonstration of head-end reprocessing. This demonstration was based upon reprocessing through the Head-End Pilot Plant only a hundred spent fuel elements of the Ft. St. Vrain type. To perform the required demonstration in the Refabrication Pilot Plant, either ^{233}U material would be taken from the storage facility in Building 3019 or the fuel elements would be fabricated using ^{235}U instead of ^{233}U.

A space utilization study was also made for using the facilities available in ORNL Building 7503 for receiving, handling, storage, and shipping of fuel. That study was based upon the assumption that the program would take beneficial occupancy of the facility and would clear out and renovate five cells which would be used as storage area for spent fuel elements being shipped from the Ft. St. Vrain Reactor for head-end reprocessing in Building 7930. Budget estimates were also prepared for this study to include cleanup of the cells in the facility with the modification and addition required to that facility, as well as those of Cell G and E in Building 7930.

IV. MATERIALS IRRADIATION

1. HTGR Recycle Fuels Irradiation

(T. N. Washburn, R. B. Fitts, and A. R. Olsen)

The irradiation tests on the HTGR recycle program have two main objectives: (1) to provide irradiated fuel for head-end process studies, and (2) to provide irradiation proof tests of

the products of coated particle process development for the Thorium-Uranium Recycle Facility. The test conditions of interest include fuel temperatures between 600 and 1300°C, burnup to 20% FIMA in the $(Th,U)O_2$ particles, and fast fluence exposures up to 8×10^{21} neutrons/cm^2.

The first two stages in this program were implemented this year. They are: (1) the start of accelerated burnup rate capsule irradiation and (2) irradiation of eight fuel elements in the Peach Bottom Reactor.

1.1 Capsule Irradiations

(A. R. Olsen)

Fabrication of the experimental capsule components is ∿70% complete. The mockup shroud was assembled onto the capsule satisfactorily so fabrication of the actual shrouds has been initiated. One shroud of each material, hafnium and Zr–40% Hf, has been preformed, etched, and welded. They are currently being annealed prior to final outer diameter sizing. All other capsule hardware components have been machined and preassembly of certain components has been initiated.

1.2 Large Scale Irradiations

(R. B. Fitts)

The initial set of six RTE's is operating satisfactorily in the Peach Bottom Reactor. The reactor has now (to October 20, 1970) accumulated 83.8 days of full power operation since the installation of the RTE's, which is 9% of that planned for the highest burnup elements and 28% of that for the low burnup elements.

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