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GEL-ADDITION PROCESS CHEMICAL STUDIES
-- Quarterly Progress Report No. 11 --

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ABSTRACT

The development of a sol-gel process and related processes for preparing (U,Pu)O₂ fast reactor fuels has continued in Babcock & Wilcox's laboratories. Progress for the eleventh quarter, ending April 30, 1969, is reported. The program is sponsored by the U.S. Atomic Energy Commission under Contract No. AT(30-1)-3714.

The preparation of UO₂-20% PuO₂ sol-gel spherical fuel in two size ranges for thermal reactor testing is complete. Two uranium enrichments—depleted UO₂ and 20% enriched ²³⁵UO₂—were used to prepare materials that will yield two different heat rates with fuel of the same U/Pu ratio. Also completed are the six initial capsule irradiation tests in a series that will show the differences in the restructuring kinetics of pellets and vibratory compacted spheres and shards. The heat rate varied from about 5 to 20 kW/ft, and the exposure time varied from about 1 to 9 EFPD. All the capsules are cooling pending hot cell examination. The seventh capsule in the series has been fabricated, and it is scheduled for exposure in May 1969.

The evaluation of procedures for the determination of moisture content and oxygen stoichiometry in mixed oxide fuels is complete. The average relative standard deviation from a series of oxygen-to-metal analyses was found to be 0.176%. All scheduled oxide characterization and analytical chemistry on the fuels for thermal reactor testing during fiscal year 1969 is complete, and the results are summarized.

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1. INTRODUCTION

This is the eleventh progress report on a continuing program of experimental work on sol-gel and related methods for preparing mixed uranium-plutonium oxide fuels for fast reactor application. The program is sponsored by the U.S. Atomic Energy Commission under Contract No. AT(30-1)-3714. This report covers the period from February 1 to April 30, 1969.

The work program for this contract establishes several technical tasks including fuel preparation and characterization, reactor testing in both thermal and fast neutron fields, and analytical support. For fiscal year 1969 more limited objectives were set on a schedule that was delayed at AEC request. Specifically, sol-gel pilot plant construction work was halted, fast reactor testing was rescheduled to begin in fiscal year 1970, the examination of reactor-exposed fuels was postponed until 1970, and thermal reactor testing was slowed.

During this report period, work continued on the fabrication of pins for thermal reactor exposure. Fuels of three different classes—pellets, shards (crushed pellets), and sol-gel spheres—are being fabricated for thermal reactor exposures which will show the restructuring characteristics of all three materials under virtually identical conditions. Six capsule exposures have been completed, and a seventh is scheduled for May 1969.

The progress and status of all the technical areas of the program are reviewed in the following sections.

2. RESULTS AND CONCLUSIONS

2.1. Fuel Preparation

All UO_2 -20% PuO_2 fuel requirements for thermal reactor testing were completed during the last quarter. A topical report covering the fuel preparation campaigns has been written and is scheduled for release during the next quarter.⁶

2.2. Oxide Characterization

Ten additional fuel pins have been fabricated for assembly into capsules for thermal reactor testing, bringing the total fabrication work in fiscal year 1969 to 31 pins containing either pellets, shards (crushed pellets), or sol-gel spheres. The difficulties in preparing acceptable sintered pellets from coprecipitated powder that had been stored for about a year and had a high oxygen-to-metal (O/M) ratio (about 2.4) were alleviated by introducing a 1-hour soak at 1000 C into the firing schedule and by increasing the N_2 -6% H_2 flow rate.

The smeared density in all the fuel rods prepared measured $82 \pm 2\%$ TD. The measured sorbed gas content was ≤ 0.03 cc/g for pellets, ≤ 0.14 cc/g for shards, and ≤ 0.08 cc/g for sol-gel spheres. The results from ceramographic and alpha autoradiographic examinations of the fuel materials indicated that the materials contained a single phase and that the plutonium was uniformly distributed.

2.3. Irradiation Testing

Irradiation testing in the thermal flux of the BAWTR is confined by budget limitations to studies of the restructuring characteristics of three fuel forms—pellets, shards, and sol-gel spheres. The initial six exposures in the restructuring series have been completed and are cooling pending hot cell examination. The next capsule is scheduled for exposure in May 1969.

2.4. Analytical Support

The furnace tube of the moisture analyzer was modified by placing the inert gas inlet tubulation farther away from the heating coil. This modification eliminated the loosening of the Tygon inlet connection that was caused by excessive heating during operation near 600 C. The procedure for moisture analyses was modified to permit the separate determination of "loosely held" and "tightly held" water.

The precision of the B&W method for measuring the O/M ratio in mixed uranium and plutonium oxides was determined. An average relative standard deviation of 0.176% was found.

3. FUEL PREPARATION (E. J. Kosiancic)

3.1. Status of Fuel Preparation Work (R. V. Carlson)

All fuel requirements for the gel-addition irradiation program in the BAWTR were completed in laboratory-scale equipment during the last reporting period. The fuel order includes 2570 grams of coprecipitated powder^{1,2} and 1108 grams of sol-gel microspheres^{3,4} containing 20% PuO_2 in depleted UO_2 , as well as 1288 grams of coprecipitated powder⁴ and 746 grams of sol-gel microspheres⁵ containing 20% PuO_2 in UO_2 enriched to 20% ^{235}U . A topical report covering the fuel preparation campaigns has been written and is scheduled for release during the next quarter.⁶

3.2. Pilot Plant Equipment (R. N. Gurley)

Two unfinished unit-operation-scale systems are in a standby status: a PuO_2 sol thermal denitrator and a spheroidizing system for preparing mixed-oxide gel from input UO_2 and PuO_2 sols. Because of reduced funding in the gel-addition program, no work has been performed on these systems during this report period.

4. OXIDE CHARACTERIZATION (J. M. Kerr)

4.1. Material Characterization

The oxide characterization work on all the fuel to be irradiated in the BAWTR during fiscal 1969 was completed. Table 1 summarizes the results from the oxide characterization and analytical chemistry work performed on this fuel.

4.2. Fabrication Studies

Five additional fuel pins each of pellets and shards have been fabricated for assembly into capsules for irradiation in the BAWTR (see section 5). The material in each of these ten pins is nominally 20% PuO_2 and 80% UO_2 ; the uranium is depleted in its ^{235}U content. As mentioned previously, difficulty was encountered in achieving the required densities, diameters, and O/M ratios in the sintered depleted pellets.⁷ The normal firing schedule yielded pellets with low densities and large dimensions, apparently because the material was not being completely reduced at the proper time and temperature in the cycle.⁸ One possible explanation for this difficulty in sintering is that the coprecipitated powder used to prepare the depleted pellets had been stored for about a year; the powder had a high O/M ratio (about 2.4, compared with a more typical value of near 2.2), which may have been a result of air oxidation. The sintering problem was solved by introducing a 1-hour soak at 1000 C into the firing schedule and by using an increased gas (N_2 -6% H_2) flow of 6 liters per minute. The remainder of the pelletizing process flow sheet remained the same.⁶ Table 2 shows the physical properties of the fabricated depleted pellets.

All work planned under this task for fiscal year 1969 is now complete. A topical report on the fabrication of the pellet, shard, and sphere specimens for the BAWTR irradiations will be issued during the next quarter.⁹

Table 1. Analytical and Characterization Results for BAWTR Irradiation Specimens

Type of material	²³⁵ U content, wt % U	$\frac{\text{Pu}}{\text{U + Pu}}$, wt % ^(a)	Moisture content, ppm oxide		O/M, at. ratio ^(a)	Sorbed gas, cc/g	Particle density, % TD ^(a)	Ceramographic examination	α -Autoradiographic examination	Pin numbers prepared
			at 100 C	at 600 C						
420-590 μ spheres	0.258 $\pm$ 0.009	19.52 $\pm$ 0.32	5.5-9.1	11.2-18.0	1.976 $\pm$ 0.005	0.036-0.039 ^(d)	95.8 $\pm$ 0.6	Single phase	Homogeneous	C-69, C-73, C-74, C-78, C-80
<44 μ spheres	0.258 $\pm$ 0.009	19.71 $\pm$ 0.17	20.7-33.0	49.1-69.6	1.987 $\pm$ 0.002	--	NM	--	--	--
420-590 μ spheres	19.68 $\pm$ 0.02	19.06 $\pm$ 0.11	3.5-6.4	9.1-16.7	1.974 $\pm$ 0.003	0.028-0.078 ^(d)	96.2 $\pm$ 0.9	Single phase	Homogeneous	C-51, C-82
<44 μ spheres	19.68 $\pm$ 0.02	19.20 $\pm$ 0.17	10.2-28.3	41.1-57.3	1.983 $\pm$ 0.004	--	NM	--	--	--
Pellets (firing #1)	19.68 $\pm$ 0.02	19.57 $\pm$ 0.12	0.4-0.8	1.6-2.1	1.971 $\pm$ 0.004	NM	--	Single phase	Homogeneous	C-99, C-100
Shards (firing #1)	19.68 $\pm$ 0.02	NM ^(c)	NM	NM	NM	0.139	--	--	--	C-45, C-59
Pellets (firing #2)	19.68 $\pm$ 0.02	19.57 $\pm$ 0.12	1.1-1.6	2.7-3.1	1.977 $\pm$ 0.003	0.030	--	Single phase	Homogeneous	C-84, C-87, C-88, C-89, C-97
Shards (firing #2)	19.68 $\pm$ 0.02	NM	NM	NM	NM	0.051	--	--	--	C-43, C-49, C-56, C-64, C-65
Pellets (firing #4)	=0.3 ^(b)	19.93 $\pm$ 0.66	NM	1.9-3.7	1.986 $\pm$ 0.004	0.013	--	--	--	C-15
Shards (firing #4)	=0.3 ^(b)	19.93 $\pm$ 0.66	NM	12.9-24.1	2.000 $\pm$ 0.009	0.105	--	--	--	C-139
Pellets (firing #5)	=0.3 ^(b)	19.93 $\pm$ 0.66	NM	0.9-4.7	1.970 $\pm$ 0.002	0.010	--	Single phase	Homogeneous	C-1, C-3, C-102, C-103
Shards (firing #6)	=0.3 ^(b)	19.93 $\pm$ 0.66	NM	26.8-33.2	1.989 $\pm$ 0.002	0.064	--	Single phase	Homogeneous	C-31, C-34, C-75, C-83

(a) Errors are for 95% confidence limits on the mean.

(b) ²³⁵U content was not determined, but it is probably less than normal.

(c) NM: not measured.

(d) Measured on a sample containing 75% 420-590 μ spheres plus 25% <44 μ spheres.

Table 2. Depleted Pellet Property Data

Diameter, in.

Average	0.208
Range	0.211 - 0.206

Length, in.

Average	0.244
Range	0.239 - 0.250

Weight, grams

Average	1.4260
---------	--------

Density, % TD

Average	93.38
One standard deviation	1.38

5. IRRADIATION TESTING (C. J. Baroch)

5.1. Introduction

The gel-addition irradiation task, as described previously,¹⁰ is divided into three subtasks:

1. Time Required for In-Pile Restructuring.
2. Sorbed Gas Behavior.
3. EBR-II Irradiations.

The scope and progress for each of these subtasks are described in this section.

5.2. Time Required for In-Pile Restructuring

In this subtask, the effect of heat rate and time on the restructuring characteristics of sol-gel $\text{UO}_2\text{-PuO}_2$ spheres and on angular particles and pellets prepared from coprecipitated $\text{UO}_2\text{-PuO}_2$ will be evaluated. A total of 17 capsules will be irradiated in the thermal flux of the BAWTR. The time required for restructuring at linear heat rates of about 6, 10, 15, and 20 kW/ft will be determined. The program will include a comparison of the irradiation behavior of spherical sol-gel particles prepared by ORNL and B&W.

Five capsules (GA-11, GA-8, GA-13, GA-14, and GA-15) were irradiated during this report period. Table 3 shows the conditions under which these capsules were irradiated, as well as the previously reported data for capsule GA-2.¹¹ The fabrication of capsule GA-4 is complete, and it is scheduled for exposure in May 1969.

5.3. Sorbed Gas Behavior

In this subtask the pressure created by the sorbed gas released from particulate fuel during irradiation will be determined. If practical, each capsule will contain two pins in which the gas pressure is continuously monitored. One pin will contain spherical sol-gel particles,

and the other will contain angular coprecipitated particles. The gas release characteristics of spherical sol-gel particles prepared by ORNL and by B&W will be determined. By varying the linear heat rate, it should be possible to determine the amount of gas released as a function of average fuel temperature. The fuel pins will be irradiated at nominal linear heat rates of 6, 10, 15, and 20 kW/ft. No work has been performed on this subtask because of funding limitations.

5.4. EBR-II Irradiations

The irradiations in EBR-II will provide performance data on particulate fuels under the fast reactor conditions obtainable in EBR-II. In this task the effect of heat rate and burnup on the overall performance of pellets, spherical particles, and angular particles will be evaluated. The smeared density for all three materials will be $82 \pm 2\%$. This subtask includes the irradiation of one 19-pin encapsulated subassembly in row 7 of the EBR-II and the irradiation of 18 unencapsulated pins in a subassembly to be shared with ORNL. The scope of the EBR-II irradiations is described in BAW-3714-9.¹¹ At AEC request, work on this subtask has been deferred until fiscal 1970.

Table 3. BAWTR Capsule Irradiation Conditions

<u>Capsule No.</u>	<u>Exposure time, days</u>	<u>Linear heat rate, kW/ft</u>		<u>NaK temperature, F</u>	
		<u>Design</u>	<u>Actual</u>	<u>Design</u>	<u>Actual</u>
GA-2	9	15	15.3-16.7	1100	1080
GA-11	1	15	15.5-17.0	1100	1040-1060
GA-8 ^(a)	3	20	17-20	1150	1050-1140
GA-13 ^(a)	3	15	~15.0	1100	1140-1170
GA-14 ^(a)	3	10	7.5-8.7	1080	950-1110
GA-15 ^(a)	3	6	5-6	1040	1140

^(a) Capsules GA-8, GA-13, GA-14, and GA-15 were irradiated simultaneously.

6. ANALYTICAL SUPPORT (D. H. Schmitt)

6.1. Moisture Analysis

The moisture analysis instrumentation includes a Type 23-321 AMA Solids Moisture Analyzer which was built by Consolidated Electrodynamics Corporation and modified by B&W in several ways.^{12,13} One of the modifications was the incorporation of a redesigned furnace tube made of heavy-wall Vitreosil, clear, fused quartz. Experience with this furnace tube shows that operation for more than about 15 minutes at temperatures exceeding 600 C causes loosening of the nitrogen sweep-gas inlet connection, a length of polyvinyl chloride (Tygon) tubing. As a result, a significant quantity of moisture (from samples containing several hundred micrograms of water) is lost from the system before being measured. To alleviate this source of error, a new furnace tube, in which the inlet tubulation is located 50 mm from the heated zone, was installed; with the other tube, this distance was 15 mm.

The moisture determination procedure has been modified slightly to obtain an indication of the relative amounts of "loosely held" and "tightly held" water. First, the moisture evolved at any requested temperature between 100 and 200 C is determined; then the additional moisture evolved at the requested maximum temperature (generally near 600 C) is determined.

No further development of this method is planned. A formal Babcock & Wilcox Analytical Method for moisture determination will be written during the next quarter.

6.2. Oxygen-to-Metal Ratio Analysis

The Fisher Scientific Company Thermogravimetric Analysis (TGA) System, Model 10-560-100, has been adapted for use in determining the oxygen-to-metal ratios in uranium oxide, plutonium oxide, and mixed

uranium-plutonium oxide. The method is based on oxidation-reduction reactions of the oxides by a flowing gas stream and measurement of the accompanying weight changes. The B&W procedure¹⁴ is adapted from a method developed by W. L. Lyon.¹⁵ The data in Table 4 show the relative precision that can be expected for O/M analyses of mixed uranium-plutonium oxides using this method. The average relative standard deviation computed from these data is 0.176%. The data indicate that the relative precision obtained by the B&W method is adequate to meet current fuel specifications.

No further development of this method is planned. A formal Babcock & Wilcox Analytical Method for O/M ratio determination will be written during the next quarter.

6.3. Analytical Sample Load

During this quarter, 17 samples were analyzed, representing a total of 58 individual analyses. These analyses included measurement of total uranium, total plutonium, moisture, O/M ratio, and the isotopic composition of uranium.

Table 4. Relative Precision for O/M Ratios of Mixed Uranium-Plutonium Oxides as Determined by the Babcock & Wilcox Method

Type of material	$\frac{\text{Pu}}{\text{Pu} + \text{O}}$ wt %	^{235}U content, wt %	No. of O/M analyses	Mean O/M, O/M units	Range, O/M units	Standard deviation, O/M units	Relative standard deviation, %	95% confidence limits on the mean, O/M units
420-590 μ microspheres	19.52	0.258	4	1.976	0.007	0.0030	0.15	0.0048
420-590 μ microspheres	19.00	19.68	4	1.974	0.004	0.0020	0.10	0.0032
<44 μ microspheres	19.71	0.258	4	1.987	0.003	0.0015	0.08	0.0024
<44 μ microspheres	19.20	19.68	4	1.983	0.004	0.0023	0.12	0.0037
One pellet prepared from co-ppt oxide	19.59	19.68	4	1.971	0.007	0.0036	0.18	0.0057
One pellet prepared from co-ppt oxide	19.51	19.68	4	1.977	0.006	0.0027	0.14	0.0043
Composite sample of 7 pellets prepared from co-ppt oxide	19.63	$\approx 0.3^{(a)}$	4	1.970	0.005	0.0022	0.11	0.0035
Composite sample of 7 pellets prepared from co-ppt oxide	19.63	$\approx 0.3^{(a)}$	4	1.986	0.011	0.0046	0.23	0.0073
Shards prepared from co-ppt oxide	19.63	$\approx 0.3^{(a)}$	4	1.984	0.005	0.0022	0.11	0.0035
Shards prepared from co-ppt oxide	19.63	$\approx 0.3^{(a)}$	5	2.000	0.020	0.0101	0.50	0.0125
One pellet prepared from dry-blended oxide	24.93	0.7 $^{(a)}$	4	1.992	0.005	0.0022	0.11	0.0035
One pellet prepared from dry-blended oxide	24.93	0.7 $^{(a)}$	4	1.993	0.005	0.0024	0.12	0.0038
One pellet prepared from dry-blended oxide	24.93	0.7 $^{(a)}$	4	1.994	0.007	0.0036	0.18	0.0057
Dry-blended oxide powder	24.79	0.7 $^{(a)}$	4	1.998	0.018	0.0082	0.41	0.0130
Dry-blended oxide powder	10.28	0.7 $^{(a)}$	3	2.023	0.003	0.0021	0.10	0.0052

^(a) ^{235}U content was not determined by the Babcock & Wilcox Analytical Chemistry Laboratory.

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Canoga Park Area Office
P.O. Box 951
Canoga Park, California 91305

32. M. Napack
United Nuclear Corporation
Grasslands Road
Elmsford, New York 10523

33. The Babcock & Wilcox Company (39)

Baroch, CJ	Holt, CD
Barringer, HS	Johnson, CR
Bishop, WN	Kerr, JM
Breazeale, WM	Kingsley, RS
Burkart, CA	Kosiancic, EJ
Carlson, RV	Library (2)
Central Files (6)	Littrell, LW (2)
Cokal, EJ	Lynch, ED
Croft, MW	Moncrief, EC
Deuster, RW	Nitti, DA
Dewell, EH	Poor, HH/Alliance
Dodd, RH	Probert, PB/Barberton
Engelder, TC	Schmitt, DH
Esleeck, SH	Travis, CC/TRG
Grant, SP	Webb, RA
Gurley, RN	Whitaker, BW