GEL-ADDITION PROCESS CHEMICAL STUDIES

— Quarterly Progress Report No. 15 —

February - April 1970

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Gel-Addition Process Chemical Studies
— Quarterly Progress Report No. 15 —
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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 at the Babcock & Wilcox Nuclear Development Center. Progress for the 15th quarter, ending April 30, 1970, is reported. The program is sponsored by the U.S. Atomic Energy Commission under Contract No. AT(30-1)-3714.

Sufficient (U,Pu)O₂ fuel was prepared for the initial bundle to be irradiated in the EBR-II. About 450g of coarse ²³⁵UO₂-20% PuO₂ microspheres was formed in the unit-operation-scale spheroidizing system, and about 350g of fines was formed in laboratory-scale equipment. Since the first lot of coprecipitated ²³⁵UO₂-20% PuO₂ powder was found to have a high aluminum content, a new lot containing about 2.2 kg of powder was prepared. Sufficient fuel pellets and shards for the initial EBR-II irradiation test were fabricated from the second lot of powder.

A capsule in the "time-for-restructuring" series was placed in the BAWTR for an 81-day irradiation at 6 kW/ft. It was removed after 55 days when abnormal operating temperatures were indicated.

Post-irradiation examination of 10 BAWTR capsules was begun. A screening gamma scan indicated that there are no fuel pin ruptures or other failures that would produce an abnormal axial fission product distribution.

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1. INTRODUCTION

This is the 15th 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, 1970.

The work program for the contract establishes several technical tasks, including fuel preparation and fabrication, reactor testing in both thermal and fast neutron fields, and analytical support. For FY-1970 the prime objective is the preparation of fuel and the fabrication of 18 unencapsulated fuel pins for irradiation in the EBR-II as part of a 37-pin subassembly to be shared with ORNL. This fuel will be fully enriched UO_2 -20% PuO_2 in the form of sol-gel microspheres, shards (crushed pellets), and pellets. In addition, eight capsules containing UO_2 -20% PuO_2 in the same three fuel forms are scheduled for irradiation in the BAWTR for from 1 to 81 days at linear heat ratings of 6 to 20 kW/ft. By the end of the fiscal year, 15 of 17 capsules in a series designed to show the differences in restructuring kinetics of pellets and vibratory compacted spheres and shards will have been irradiated in the BAWTR. The examination of 10 of these capsules will be initiated in FY-1970 in the B&W hot cell facility.

During this report period sufficient (U,Pu) O_2 fuel was prepared for the initial bundle to be irradiated in the EBR-II. About 800g of $^{235}\text{UO}_2$ -20% PuO_2 microspheres was formed, and 2.2 kg of coprecipitated $^{235}\text{UO}_2$ -20% PuO_2 powder was prepared and fabricated into pellets and shards. Capsule GA-12, scheduled for an 81-day irradiation in the BAWTR at 6 kW/ft, was removed after 55 days when abnormal operating temperatures were indicated. Characterization of all fuel prepared for testing in the BAWTR and EBR-II was completed. The post-irradiation

examination of 10 BAWTR capsules in the "time-for-restructuring" series was initiated. Progress in all technical areas of the program is reviewed in the following sections.

2. RESULTS AND CONCLUSIONS

2.1. Fuel Preparation

The first lot of coprecipitated $^{235}\text{UO}_2$ -20% PuO_2 powder was found to have a high aluminum content. Since sufficient time and material was available, a new lot of about 2.2 kg of powder was prepared.

About 450g of coarse microspheres and 350g of fines were delivered for pin loading. The preparation of this material was unusually difficult, and the yield of acceptable product was low. Since the properties of the available $^{235}\text{UO}_2$ sol were changing during the sphere-forming runs, the difficulties are assumed to be related to poor-quality sol.

2.2. Fuel Fabrication

A new lot of $^{235}\text{UO}_2$ -20% PuO_2 pellets for the initial EBR-II irradiation test was prepared from the second lot of coprecipitated powder. These pellets were centerless ground and loaded into pins. In addition a new lot of $^{235}\text{UO}_2$ -20% PuO_2 shards was prepared. The vipac pins for the EBR-II test are being fabricated.

2.3. Irradiation Testing

Capsule GA-12, scheduled for an 81-day irradiation in the BAWTR at 6 kW/ft, was removed after 55 days when abnormal operating temperatures were indicated. The source of the problem is not known, but neutron radiographs indicate that the fuel train is still in the proper position and that there are no failed fuel pins.

The acceptable stainless steel tubing for the EBR-II pins was cut to length, and end caps were welded onto 23 tubes. All the internal components were received and inspected. The xenon tagging procedure has been submitted for approval, and construction of the gas handling system is nearly complete.

Post-irradiation examination of 10 BAWTR capsules is in progress. A screening gamma scan indicated that there are no fuel pin

ruptures or other failures that would produce an abnormal axial fission product distribution.

2.4. Analytical Support

Characterization of all fuel prepared for testing in the BAWTR and EBR-II is now complete.

3. FUEL PREPARATION (R. V. Carlson)

The gel-addition irradiation program includes the exposure of spherical particles, angular particles, and pellets to the fast-reactor conditions obtainable in the EBR-II. Eighteen unencapsulated pins are scheduled for insertion in July 1970 as part of a 37-pin subassembly to be shared with ORNL. An additional 16 pins are required—10 will be loaded in the EBR-II at a later date, and six will serve as archive samples or replacements for rejected pins. The total fuel order for the EBR-II irradiation includes 1300g of sol-gel microspheres and 4100g of coprecipitated powder containing 20% PuO_2 in UO_2 enriched to 93% ^{235}U .

3.1. Preparation of $^{235}\text{UO}_2$ -20% PuO_2 Powder

Previously, it was reported that the FY-1970 mixed-oxide powder order was completed.¹ However, the $(^{235}\text{U}, \text{Pu})\text{O}_2$ fuel pellets prepared from this material had a high aluminum content (see Table 3-1). After checking the aluminum content in several materials at various stages of preparation, it was shown that the major source of the impurity was the high-density alumina balls used in milling the calcined powder. The effect of a high aluminum content on the irradiation tests is not clear. Since sufficient time and material were available, a new lot of powder was prepared, and tungsten carbide balls were used in the ball-milling step.

Nearly 2.2 kg of mixed oxide powder was prepared during the quarter by continuous coprecipitation of ammonium diuranate-plutonium hydroxide from a uranium-plutonium nitrate solution. The process flow-sheet was summarized previously.¹

Although the capacity of the continuous coprecipitation equipment is about 2 kg/shift, the fissile material mass limit of the glove box restricted the throughput to 315g (on a metal basis) per run. The

$\text{Pu}(\text{NO}_3)_4$ and $\text{UO}_2(\text{NO}_3)_2$ feed solutions were mixed on a weight basis from previously analyzed stock solutions. The compositions of these stock solutions are shown in Table 3-2. The pH was held at about 9.5 during precipitation to ensure homogeneity of the plutonium and uranium in the powder.^{2,3}

The data in Table 3-3 show that the weight loss averaged about 27% during calcination and that the average yield was about 87.5%. These values are in good agreement with previously reported data.¹ The principal source of material loss was holdup in the large equipment. After each run, except 8-B-72 and 8-B-75, the entire system was flushed with dilute nitric acid, and the resultant solution was stored for scrap recovery. For runs 8-B-73 and 8-B-76, which were made before the system was flushed, the yield was almost 100%.

The plutonium content of each batch of calcined powder was measured before the acceptable powder was blended into one lot. The specification calls for a $\text{Pu}/(\text{Pu}+\text{U})$ ratio of $20.0 \pm 0.5\%$. The data in Table 3-3 show that all batches were within these limits.

The seven batches of calcined powder were blended in a V-Cone blender, and the entire lot was ball-milled for 1.5 hours with tungsten carbide balls. After this treatment all the powder passed through a 200-mesh screen (75-micron openings).

3.2. Preparation of $^{235}\text{UO}_2$ -20% PuO_2 Microspheres

Sufficient $^{235}\text{UO}_2$ -20% PuO_2 microspheres for the EBR-II pins scheduled for insertion in July 1970 have been prepared.

3.2.1. Preparation of UO_2 and PuO_2 sol

Sufficient $^{235}\text{UO}_2$ and PuO_2 sol was prepared to complete the FY-1970 microsphere requirements.⁴ The UO_2 sol was somewhat lighter in color than is normally observed. In addition, the viscosity of the UO_2 sol began to increase within about one month, whereas a shelf-life of up to six months had been previously observed. The reason for this behavior is not known. Because of the limited time available for the completion of the fuel order, it was decided to use this sol.

Table 3-1. Impurities in First Lot of EBR-II Pellets

Element	Concentration, ppm metal	Element	Concentration, ppm metal
Carbon	13.4	Iron	16
Chloride	6.3	Lead	<2
Fluoride	0.9	Lithium	<5
Nitrogen	33	Magnesium	13
		Manganese	3
Aluminum	≈5000	Molybdenum	<6
Beryllium	<0.5	Nickel	<25
Bismuth	<1.0	Silicon	<12
Boron	<0.5	Silver	<0.5
Cadmium	<0.5	Sodium	13
Calcium	112	Tin	3
Chromium	<10	Vanadium	<2
Copper	32	Zinc	<10

Table 3-2. Composition of Stock Solutions for Coprecipitation

<u>Plutonium Nitrate</u>	
Plutonium, mg/g soln	154.9 ± 0.5
Free acid, M	5.2
²³⁸ Pu, wt %	0.047 ^(a)
²³⁹ Pu, wt %	90.497 ^(a)
²⁴⁰ Pu, wt %	8.344 ^(a)
²⁴¹ Pu, wt %	1.033 ^(a)
²⁴² Pu, wt %	0.079 ^(a)
<u>Uranyl Nitrate</u>	
Uranium, mg/g soln	217.1 ± 0.4
²³⁴ U, wt %	0.99 ± 0.01 ^(b)
²³⁵ U, wt %	92.68 ± 0.05 ^(b)
²³⁶ U, wt %	0.38 ± 0.01 ^(b)
²³⁸ U, wt %	5.95 ± 0.03 ^(b)

(a) Analysis performed on June 21, 1968.

(b) Analysis performed on July 10, 1968.

Table 3-3. Data From Preparation of Coprecipitated Powder

Batch No.	Weight of dried cake, g	Weight of calcined product, g	Weight loss during calcination, %	Pu/(Pu+U), % ^(a)	Yield, %
8-B-70	409.5	305.0	25.5	20.12 ± 0.06	85.3
8-B-71	368.0	275.5	25.1	20.04 ± 0.11	77.1
8-B-72	456.7	274.7	39.9	20.16 ± 0.06	76.8
8-B-73	444.0	341.5	23.1	20.01 ± 0.06	95.5 ^(b)
8-B-74	416.7	325.0	22.0	20.03 ± 0.06	90.9
8-B-75	411.5	308.2	25.1	19.96 ± 0.08	86.2
8-B-76	483.0	358.0	25.9	20.19 ± 0.03	100.1 ^(b)

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

(b) System was not flushed with acid before run.

3.2.2. Microsphere Formation

The initial effort to prepare ²³⁵UO₂-20% PuO₂ microspheres in the unit-operation-scale spheroidizing system was unsuccessful because of an inability to determine a satisfactory surfactant concentration. Since no difficulty was encountered with the laboratory-scale system, which does not contain a still for organic regeneration, the large system was operated with the organic bypassing the still. The operating procedure has been summarized previously.⁴

About 450g of coarse microspheres and 350g of fines are available for pin loading. The preparation of this material was unusually difficult because the required surfactant concentration often changed, not only between runs but also within a run. Since the properties of the available ²³⁵UO₂ sol were changing (e.g., the viscosity was increasing), the difficulties are assumed to have been related to poor-quality sol. The data from the runs that yielded microspheres are summarized in Tables 3-4 and 3-5. The average yield of acceptable coarse spheres is comparable to that obtained with the small column,^{5,6} but the best yield is substantially greater than has been achieved previously.

The plutonium content of each batch of spheres was measured and found to vary over a wider range than encountered in prior

work at B&W. A Pu/(Pu+U) ratio of $20.0 \pm 0.5\%$ was the target. However, the ratio for the fine fraction ranged from 17.9 to 22.6%; for the coarse fraction it ranged from 16.8 to 24.3%. The calculated average Pu/(Pu+U) ratio is 19.1% for the fines and 20.9% for the coarse spheres. The calculated average is 20.4% for the quantity of each blended size fraction required in a fuel pin.

The unusually wide range in plutonium content of the microspheres is thought to be due to the poor-quality $^{235}\text{UO}_2$ sol available. As pointed out earlier, the $^{235}\text{UO}_2$ sol tended to become viscous within a short period of time. In addition, solids (probably gel) slowly settled during storage. Apparently the procedure used to resuspend the solids before the withdrawal of each batch from the master lot was not adequate.

It is felt that the technical objectives of the EBR-II irradiation experiment can be met if these microspheres are used. In addition, it is felt that the use of this material would pose no additional hazard to the reactor itself. Therefore, this material was released for pin loading.

Table 3-4. Sphere Formation Data

<u>Coarse fraction</u>	<u>Average yield from all runs, %(a)</u>	<u>Yield from best run, %(a, b)</u>	<u>Yield from worst run, %(a, b)</u>
Rounded product (420-590 μ)	42.6	67.2	26.8
Non-round scrap	11.5	4.9	29.3
Off-size scrap	45.9	19.7	65.5
<u>Fine fraction</u>			
Product (<44 μ)	97.8	100	92.3
Off-size scrap	2.2	0	7.7

(a) All yields are calculated on the basis of recovered material.

(b) All data are not necessarily from the same run.

Table 3-5. Size Distribution of Spheres Prepared

<u>Coarse fraction, μ</u>	<u>Average from all runs, %</u>	<u>Run with highest yield of product, %</u>	<u>Run with lowest yield of product, %</u>
>590	2.6	1.4	0.7
500-590	17.6	44.8	1.2
420-500	26.3	28.7	2.9
350-420	13.9	14.3	15.8
210-350	21.3	6.8	62.7
149-210	12.1	1.1	14.0
<149	6.2	2.9	2.7
<u>Fine fraction, μ</u>			
>44	2.2	0	0
37-44	10.6	8.4	20.5
<37	87.2	91.6	79.5

Note: Data include rounds and non-rounds.

4. FUEL FABRICATION (A. H. Bremser, J. M. Kerr)

4.1. Oxide Characterization (A. H. Bremser)

Sorbed gas release values were obtained for the third EBR-II blanket pellet firing, the new lots of fuel pellets and shards (see section 4.2), and the fibrous plug material. These results are as follows:

Material	Gas release, cc/g @ STP			
	Run 1	Run 2	Run 3	Average
EBR-II fuel pellets, lot 2	0.006	0.004	0.006	0.005
EBR-II fuel shards, lot 2	0.041	0.044	0.050	0.045
EBR-II blanket pellets, firing 3	0.003	0.006	0.006	0.005
EBR-II fibrous plug (Fiberfrax)	0.39	0.81	0.47	0.56

Microstructures and alpha autoradiographs were obtained for the new fuel pellets. A typical microstructure is shown in Figure 4-1, and a typical alpha autoradiograph is shown in Figure 4-2.

4.2. Fabrication of EBR-II Specimens (L. J. Ferrell)

A new lot of $^{235}\text{UO}_2$ -20% PuO_2 powder was produced (see section 3.1). Two batches of pellets were pressed and fired from this material—one to be used in pellet pins and one to be crushed into shards. The former were centerless ground, washed, and dried. They were sorted by measuring length and weight and by using a nomograph (Figure 4-3) to ensure that each accepted pellet was within the density specification of $93 \pm 2\%$ of theoretical. The nomograph has two lines, one for the minimum diameter-maximum density relationship and one for the maximum diameter-minimum density relationship. If the pellet's length

and weight fell within the area bounded by the two lines, the pellet was accepted. This time-saving method was possible because of the very close tolerances (± 0.25 mil) maintained during centerless grinding. All pellets produced in this manner were stacked, weighed, and loaded into EBR-II cladding along with the appropriate blanket and insulator pellets and other internals. Data on these pellet pins are as follows:

		Pin						
		D-12	D-14	D-9	D-17	D-16	D-6	D-4
UO ₂ blanket pellets	Length, in.	6.060	6.060	6.059	6.060	6.058	6.057	6.059
	Weight, g	37.082	37.144	37.054	37.164	37.157	37.173	37.055
Fiberfrax	Length, in.	1/16	1/16	1/16	1/16	1/16	1/16	1/16
Bottom insulators	Length, in.	0.4903	0.5007	0.5010	0.4990	0.4872	0.5046	0.5072
	Weight, g	2.9824	3.1127	3.1159	3.0858	3.0180	3.0775	3.0955
Fuel	Length, in.	13.483	13.458	13.494	13.457	13.440	13.543	13.458
	Weight, g	75.00	74.74	75.01	74.76	74.46	75.41	74.82
	Smear density	82.73	82.92	82.78	82.79	82.70	82.83	82.50
Top insulators	Length, in.	0.5055	0.5016	0.4995	0.4960	0.4980	0.5018	0.5010
	Weight, g	3.1036	3.0995	3.0776	3.0761	3.0833	3.1018	3.0950
Fiberfrax	Length, in.	0.0625	0.0625	0.0625	0.0625	0.0625	0.0625	0.0625
Nickel reflector	Ident No.	D-7	D-4	D-3	D-6	D-17	D-1	D-21
Spring	Ident No.	D-7	D-4	D-3	D-6	D-17	D-1	D-21

Note: 1. Diameters of the fuel pellets range from 0.2045 to 0.2050 inch.
 2. Diameters of the insulator and the blanket pellets are 0.214 ± 0.002 inch.

The second batch of fired pellets was crushed into the appropriate size fractions for vibratory compaction. Fabrication of the vipac pins is in progress.

4.3. Fabrication of BAWTR Specimens (J. M. Kerr, L. J. Ferrell)

All specimens for the BAWTR "time-for-restructuring" series have been prepared.⁷ The characterization data for each fuel lot are shown in section 6.1. The fuel lot contained in each pin is summarized in Table 4-1.

Figure 4-1. Photomicrograph of
EBR-II Fuel Pellet
(Lot 2)

Figure 4-2. Alpha Autoradiograph
of EBR-II Fuel Pellet
(Lot 2)

Figure 4-3. Pellet Density Nomograph

Table 4-1. Fuel Contained in BAWTR Pins

<u>Type of fuel</u>	<u>Pins prepared</u>
Depleted (U, Pu)O ₂ pellets	
Firing 1	C-16, C-17, C-42
Firing 2	C-1, C-3, C-102, C-103
Firing 4	C-10, C-19, C-109, C-110, C-114, C-117
20% ²³⁵ U-enriched (U, Pu)O ₂ pellets	
Firing 1	C-99, C-100
Firing 2	C-84, C-87, C-88, C-89, C-97
Depleted (U, Pu)O ₂ shards	
Firing 1	C-23, C-139
Firing 2	C-53, C-84A, C-126, C-128, C-129, C-137
Firing 4	C-31, C-34, C-75, C-83
20% ²³⁵ U-enriched (U, Pu)O ₂ pellets	
Firing 1	C-45, C-59
Firing 2	C-43, C-49, C-58, C-64, C-65
Depleted B&W (U, Pu)O ₂ microspheres	C-24, C-68, C-69, C-73, C-74, C-78, C-80, C-132, C-141, C-146
20% ²³⁵ U-enriched B&W (U, Pu)O ₂ microspheres	C-46, C-51, C-82, C-125, C-134, C-138
ORNL (U, Pu)O ₂ microspheres	C-52, C-122

5. IRRADIATION TESTING (E. N. Harbinson, C. J. Baroch)

The gel-addition irradiation task⁸ is divided into three subtasks: (1) time required for in-pile restructuring, (2) sorbed gas behavior, and (3) EBR-II irradiation. The scope and the progress of each of these subtasks are described.

5.1. Time Required for Inpile Restructuring

The effect of heat rate and time on the restructuring characteristics of sol-gel $\text{UO}_2\text{-PuO}_2$ spheres and of angular particles and pellets prepared from coprecipitated $\text{UO}_2\text{-PuO}_2$ powder will be evaluated. A total of 17 capsules will be irradiated in 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.

Capsule GA-12 was scheduled for 81 days' irradiation at 6 kW/ft. The exposure began in January and continued until March 27, 1970, when the readouts from the NaK and ΔT thermocouples became erratic for a short period and then indicated unacceptably high operating temperatures. Therefore, the capsule was removed from the BAWTR after only 55 days' irradiation. Neutron radiographs of the capsule indicate that the fuel train is still in the proper position. No failed fuel pins could be detected. Although no electronic problem in the temperature readout system has been found, this is thought to be the source of the unusual readouts. The problem will be investigated further before we decide whether or not to reinsert the capsule. Table 5-1 shows the conditions under which GA-12 was irradiated, as well as the data from the previously irradiated capsules.

Five additional capsules (GA-5, GA-7, GA-16, GA-18, and GA-19) were assembled during the quarter. All of the capsules that have been

assembled are described completely in Table 5-2. Capsules GA-5, GA-7, GA-18, and GA-19 will be irradiated simultaneously for 27 days during May 1970.

Table 5-1. Operating Conditions for BAWTR Capsules

Capsule No.	Exposure time, d	Linear heat rate, kW/ft		NaK temperature, F	
		Design	Actual	Design	Actual
GA-15 ^(a)	3	6	5.0 - 6.5	1040	1140
GA-12	81 ^(c)	6	4.8 - 6.0	1040	1015 - 1025
GA-14 ^(a)	3	10	7.5 - 10.3	1080	950 - 1110
GA-11	1	15	15.5 - 17.0	1100	1040 - 1060
GA-13 ^(a)	3	15	15.2 - 16.1	1100	1140 - 1170
GA-2	9	15	15.3 - 16.7	1100	1080
GA-4	1	20	17.2 - 19.6	1150	965 - 1045
GA-8 ^(a)	3	20	17.0 - 19.8	1150	1050 - 1140
GA-10	3	20	20.0 - 22.0	1150	1120 - 1160
GA-6	9	20	17.0 - 20.0	1150	1060 - 1120
GA-9 ^(b)	5	6	6.2 - 7.3	1040	1140 - 1225

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

(b) Capsule GA-9 was to be irradiated for 81 days, but was removed after 5 days of operation because of indicated abnormal temperatures.

(c) Irradiation suspended after 55 days of operation because of indicated abnormal temperatures.

Table 5-2. Assembly Data for BAWTR Capsules

Capsule Numbers	Pin Numbers
GA-2	C-23, C-24, C-42
GA-4	C-49, C-51, C-100
GA-5	C-59, C-84, C-138
GA-6	C-45, C-97, C-134
GA-7	C-10, C-126, C-146
GA-8	C-43, C-82, C-99
GA-9	C-3, C-31, C-73
GA-10	C-46, C-88, C-122
GA-11	C-15, C-74, C-139
GA-12	C-53, C-68, C-110
GA-13	C-80, C-83, C-103
GA-14	C-34, C-78, C-102
GA-15	C-1, C-69, C-75
GA-16	C-52, C-87, C-125
GA-18	C-114, C-132, C-137
GA-19	C-19, C-84A, C-141

5.2. Sorbed Gas Behavior

The pressure created by the sorbed gas released from particulate fuel during irradiation will be determined. If practicable, 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 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 design linear heat rates of 6, 10, 15, and 20 kW/ft. No work has been performed on this subtask because of funding limitations.

5.3. EBR-II Irradiations

The primary objective of the EBR-II irradiations is to obtain data on the operational characteristics of particulate fuels irradiated in a fast reactor. The effect of heat rate and burnup on the overall performance of sol-gel spherical particles and of angular particles and pellets prepared from coprecipitated UO_2 -20% PuO_2 powder will be compared and evaluated. This subtask includes the irradiation of 30 fuel pins in a 19-pin encapsulated subassembly in row 7 of the EBR-II, and the irradiation of 28 unencapsulated pins in a 37-pin subassembly to be shared with ORNL. The heat rating for the encapsulated and the unencapsulated fuel pins will be approximately 8 to 10 kW/ft and 14 to 16 kW/ft, respectively.

The type-316 stainless steel tubing, which passed ultrasonic inspection,⁹ was cut to 38-inch lengths and inspected for unacceptable dimensional deviations in both outside and inside diameters. Bottom end caps were welded onto 23 accepted tubes. These welds were helium-leak-checked and radiographically inspected. The welds meet the acceptance criteria for EBR-II irradiations.¹⁰ All of the internal components were received and inspected.

The xenon-tagging procedure was completed and submitted to the EBR-II staff for approval. Construction of the gas-handling system is nearly complete, and testing is scheduled for the next quarter.

5.4. Post-Irradiation Examinations (R. E. Womack)

Post-irradiation examination of 10 of the capsules withdrawn from the BAWTR has begun. Instrumentation leads and standpipes have been removed, and the capsules have been cut to a standard length in preparation for NaK-reaction operations. All capsules have been subjected to a screening gamma scan in an effort to detect fuel pin ruptures or other failures that would produce an abnormal axial fission product distribution. No such condition is evident in any of the capsules. All capsules have been placed into the alpha-box, and capsule disassembly operations are under way.

6. ANALYTICAL SUPPORT (D. H. Schmitt)

6.1. Characterization of BAWTR Fuel (R. H. Dodd, R. J. Boden)

Characterization of all fuel prepared for the BAWTR irradiation program has been completed. These data are shown in Tables 6-1 through 6-6.

Most of the impurity levels are well within those given in typical fast reactor fuel specifications. However, the carbon content of the shards and the fine microspheres is high, possibly due to CO₂ sorption during storage. This phenomenon will be investigated later. The aluminum content of the pellets and shards is high, presumably because of the alumina balls used in the milling step during powder preparation (see section 3.1). In section 4.3 the fuel is identified with the exact pins used in the irradiation program.

6.2. Characterization of EBR-II Fuel and Insulators (R. H. Dodd, R. J. Boden)

The characterization data for the second lot of EBR-II fuel prepared during FY-1970 are shown in Tables 6-7 through 6-10. The data for the EBR-II UO₂ insulator pellets are shown in Tables 6-11 and 6-12. The impurity levels are generally well within those given in typical fast reactor fuel specifications.

Table 6-1. BAWTR Fuel Pellet Characterization

Analysis	Depleted (U,Pu)O ₂ pellets ^(a)				20% ²³⁵ U-enriched (U,Pu)O ₂ pellets ^(a)	
	Firing 1	Firing 2	Firing 3 ^(c)	Firing 4	Firing 1	Firing 2
Pu/(Pu+U), wt %	19.93 ± 0.66	19.93 ± 0.66	19.93 ± 0.66	19.93 ± 0.66	19.57 ± 0.12	19.57 ± 0.12
²³⁵ U content, wt %	<0.7 ^(b)	<0.7 ^(b)	<0.7 ^(b)	<0.7 ^(b)	19.68 ± 0.02	19.68 ± 0.02
O/M, atom ratio	1.986 ± 0.004	1.970 ± 0.002	1.975 ± 0.006	1.975 ± 0.006	1.971 ± 0.004	1.977 ± 0.003
Moisture, ppm oxide	2.8 ± 1.3	1.9 ± 3.0	4.6 ± 0.7	4.6 ± 0.7	1.8 ± 0.4	3.0 ± 0.3
Sorbed gas, cc/g	0.021	0.010	0.013	0.013	0.030	0.030
Density, % theor	91.04	92.55	86.99	92.66	91.52	95.90
Nitrogen, ppm metal	20.5 ± 2.5	29.8 ± 3.6	15.6 ± 2.0	21.1 ± 11.2	24.1 ± 7.5	24.1 ± 7.5
Carbon, ppm metal	33.5 ± 3.0	51.3 ± 10.1	44.9 ± 10.2	6.42 ± 13.2	29.0 ± 4.6	29.0 ± 4.6
Chloride, ppm metal	17.8 ± 8.9	14.2 ± 6.9	5.8 ± 0.8	5.0 ± 2.7	6.0 ± 0.7	6.0 ± 0.7
Fluoride, ppm metal	7.1 ± 0.3	1.4 ± 0.3	0.6 ± 0.2	5.1 ± 1.1	1.2 ± 0.4	1.2 ± 0.4

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

(b) ²³⁵U content was not determined but should be less than normal.

(c) This firing was not used because the density was below specifications.

Table 6-2. Trace Metallic Impurities in BAWTR Fuel Pellets

Element	Depleted (U, Pu)O ₂ pellets				20% ²³⁵ U-enriched (U, Pu)O ₂ pellets	
	Firing 1	Firing 2	Firing 3	Firing 4	Firing 1	Firing 2
Aluminum	>500	>500	>500	>500	>500	>500
Beryllium	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
Bismuth	<1	<1	<1	<1	<1	<1
Boron	0.5	<0.5	<0.5	<0.5	0.5	0.5
Cadmium	3	2	<0.5	<0.5	2	2
Calcium	50	33	30	9	16	16
Chromium	8	8	<5	27	<5	<5
Copper	>100	>100	>100	170	>100	>100
Iron	62	25	21	39	65	65
Lead	49	44	3	2	31	31
Lithium	<1	<1	<1	--	<1	<1
Magnesium	18	12	13	48	9	9
Manganese	<1	<1	<1	3	<1	<1
Molybdenum	<6	100	40	<34	30	30
Nickel	<25	<25	<25	<11	<25	<25
Silicon	<10	15	<10	28	<10	<10
Silver	<1	<1	<1	<0.5	<1	<1
Sodium	36	11	6	45	16	16
Tin	<3	<3	<3	10	<3	<3
Titanium	8	<5	<5	<5	<5	<5
Vanadium	<1	<1	<1	<1	<1	<1
Zinc	54	100	<10	<10	30	30

(a) Values given are in ppm of metal and are the mean of four determinations.

Table 6-3. BAWTR Fuel Shards Characterization.

Analysis	Depleted (U,Pu)O ₂ shards ^(a)			20% ²³⁵ U-enriched (U,Pu)O ₂ shards ^(a)	
	Firing 1	Firing 2	^(a) Firing 4	Firing 1 ^(c)	Firing 2 ^(c)
Pu/(Pu + U), wt %	19.93 ± 0.66	19.93 ± 0.66	19.93 ± 0.66	NM	NM
²³⁵ U content, wt %	<0.7 ^(b)	<0.7 ^(b)	<0.7 ^(b)	NM	NM
O/M, atom ratio	2.000 ± 0.009	1.984 ± 0.003	1.977 ± 0.007	NM	NM
Moisture, ppm oxide	17.3 ± 7.7	37.1 ± 18.3	19.8 ± 6.1	NM	NM
Sorbed gas, cc/g	0.105	0.064	0.094	0.139	0.051
Nitrogen, ppm metal	41.5 ± 3.7	41.5 ± 3.7	13.8 ± 5.4	NM	NM
Carbon, ppm metal	226.5 ± 45.8	226.5 ± 45.8	35.0 ± 15.9	NM	NM
Chloride, ppm metal	13.6 ± 3.4	13.6 ± 3.4	5.5 ± 2.2	NM	NM
Fluoride, ppm metal	1.3 ± 0.5	1.3 ± 0.5	4.5 ± 0.6	NM	NM

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

^(b)²³⁵U content not determined but should be less than normal.

^(c)Not measured; assumed to have about the same values as the pellet firings No. 1 and 2 from which they were prepared.

Table 6-4. Trace Metallic Impurities in BAWTR Fuel Shards

Element	Depleted (U, Pu)O ₂ shards			20% ²³⁵ U-enriched (U, Pu)O ₂ shards	
	Firing 1	Firing 2	Firing 4	Firing 1 ^(b)	Firing 2 ^(b)
Aluminum	>500	>500	>500	NM	NM
Beryllium	<0.5	<0.5	<0.5	NM	NM
Bismuth	<1	<1	<1	NM	NM
Boron	1	1	<0.5	NM	NM
Cadmium	1	1	<0.5	NM	NM
Calcium	48	48	<11	NM	NM
Chromium	<5	<5	<23	NM	NM
Copper	>100	>100	86	NM	NM
Iron	73	73	36	NM	NM
Lead	24	24	6	NM	NM
Lithium	<1	<1	NM	NM	NM
Magnesium	33	33	21	NM	NM
Manganese	3	3	5	NM	NM
Molybdenum	<6	<6	<34	NM	NM
Nickel	<25	<25	<11	NM	NM
Silicon	<10	<10	7	NM	NM
Silver	<1	<1	<0.5	NM	NM
Sodium	23	23	<4	NM	NM
Tin	<3	<3	2	NM	NM
Titanium	<5	<5	<11	NM	NM
Vanadium	<1	<1	<1	NM	NM
Zinc	38	38	<10	NM	NM

(a) Values given are in ppm of metal and are the mean of four determinations.

(b) Not measured; assumed to have about the same values as the pellet firings No. 1 and 2 from which they were prepared.

Table 6-5. BAWTR Fuel Microsphere Characterization

Analysis	Depleted (U, Pu)O ₂ microspheres(a)		20% ²³⁵ U-enriched (U, Pu)O ₂ microspheres(a)		ORNL (U, Pu)O ₂ microspheres(a)	
	420-590 μ	<44 μ	420-590 μ	<44 μ	420-590 μ	<25 μ
Pu/(Pu + U), wt %	19.52 $\pm$ 0.32	19.71 $\pm$ 17	19.00 $\pm$ 0.11	19.20 $\pm$ 0.17	20.74 $\pm$ 0.08	20.01 $\pm$ 0.15
²³⁵ U content, wt %	0.258 $\pm$ 0.009	0.258 $\pm$ 0.009	19.68 $\pm$ 0.02	19.68 $\pm$ 0.02	20.16 ^(b)	20.08 ^(b)
O/M, atom ratio	1.976 $\pm$ 0.005	1.987 $\pm$ 0.002	1.974 $\pm$ 0.003	1.983 $\pm$ 0.004	1.994 $\pm$ 0.001	1.997 $\pm$ 0.003
Moisture, ppm oxide	14.0 $\pm$ 5.2	59.0 $\pm$ 17.0	12.0 $\pm$ 10.2	50.5 $\pm$ 12.6	7.0 $\pm$ 1.2	67 $\pm$ 15
Sorbed gas, cc/g	0.038 ^(c)	0.038 ^(c)	0.053 ^(c)	0.053 ^(c)	0.016 ^(c)	0.016 ^(c)
Density, % theor	95.8 $\pm$ 0.6	NM	97.3 $\pm$ 0.7	NM	97.1 $\pm$ 0.1	NM
Nitrogen, ppm metal	42.3 $\pm$ 22.0	49.1 $\pm$ 14.7	12.5 $\pm$ 3.8	74.0 $\pm$ 40.7	11.5 $\pm$ 2.9	17.8 $\pm$ 1.6
Carbon, ppm metal	58.8 $\pm$ 5.0	294.2 $\pm$ 33.2	11.8 $\pm$ 2.4	245.8 $\pm$ 33.0	10.2 $\pm$ 3.3	21.2 $\pm$ 4.6
Chloride, ppm metal	12.1 $\pm$ 1.0	19.8 $\pm$ 8.3	6.0 $\pm$ 2.2	6.0 $\pm$ 2.1	5.3 $\pm$ 0.7	9.7 $\pm$ 1.3
Fluoride, ppm metal	1.2 $\pm$ 0.2	1.4 $\pm$ 0.1	0.5 $\pm$ 0.2	1.6 $\pm$ 0.3	0.7 $\pm$ 0.3	0.9 $\pm$ 0.8

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

(b) ²³⁵U content determined by ORNL.

(c) Sorbed gas analyses were performed on a mixture of 73 wt % coarse and 27 wt % fine microspheres in order to simulate the fuel composition in a pin.

Table 6-6. Trace Metallic Impurities in BAWTR Fuel Microspheres^(a)

Element	Depleted (U,Pu)O ₂ microspheres		20% ²³⁵ U-enriched (U,Pu)O ₂ microspheres		ORNL (U,Pu)O ₂ microspheres	
	420-590 μ	<44 μ	420-590 μ	<44 μ	420-590 μ	<25 μ
Aluminum	215	190	305	275	11	31
Beryllium	<0.15	<0.5	<0.5	<0.5	<0.25	<0.25
Bismuth	<1	<1	NM	<1	<1	<1
Boron	<0.5	<0.5	1.7	<0.5	<0.4	3
Cadmium	<0.5	<0.5	<1	<0.5	<0.5	4
Calcium	125	96	NM	125	21	115
Chromium	16	<50	<20	<50	<9	<100
Copper	85	13	46	<2	<2	20
Iron	430	350	620	250	26	106
Lead	<1	<1	<1	<1	<1	<1
Lithium	<1	<1	NM	<1	<0.5	<0.5
Magnesium	17	22	23	35	<2.5	6
Manganese	3	13	3	9	<1	3.5
Molybdenum	32	25	<25	<6	100	4
Nickel	<25	<25	<20	<25	<10	58
Silicon	400	450	250	200	33	120
Silver	<1	<1	<1	<1	12	11
Sodium	13	9	24	12	<1	<1
Tin	7	<3	3	10	4	42
Titanium	7	8	<15	<5	<5	20
Vanadium	<1	<1	<1	<1	<1	<1
Zinc	30	<10	<20	<10	<50	<50

(a) Values given are in ppm of metal and are the mean of four determinations.

Table 6-7. EBR-II Fuel Pellet and Shard Characterization^(a)

Analysis	Fully enriched (U,Pu)O ₂ pellets ^(b)	Fully enriched (U,Pu)O ₂ shards ^(b)
Pu/(Pu+U), wt %	20.20 ± 0.04	20.10 ± 0.15
²³⁵ U content, wt %	92.68 ^(c)	92.68 ^(c)
O/M, atom ratio	1.983 ± 0.003	1.980 ± 0.005
Moisture, ppm oxide	2.4 ± 1.0	42.5 ± 12.9
Sorbed gas, cc/g	0.0053	0.045
Density, % theor	93.0 ± 2.0	91.0
Nitrogen, ppm metal	23.8 ± 2.7	35.2 ± 9.2
Carbon, ppm metal	17.2 ± 6.6	70.5 ± 11.5
Chloride, ppm metal	16.2 ± 3.7	12.7 ± 0.6
Fluoride, ppm metal	13.0 ± 4.3	6.6 ± 1.9

^(a) Second lot.

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

^(c) ²³⁵U content determined by AVCO.

Table 6-8. Trace Metallic Impurities in EBR-II Fuel Pellets and Shards^(a)

Element	Fully enriched (U,Pu)O ₂ pellets	Fully enriched (U,Pu)O ₂ shards
Aluminum	300	330
Beryllium	<0.5	<0.5
Bismuth	<1	<1
Boron	<0.3	<0.3
Cadmium	<0.5	<0.5
Calcium	≈120	≈120
Chromium	12	18
Copper	75	19
Iron	65	80
Lead	<2	<2
Lithium	<1	<1
Magnesium	6	15
Manganese	<4	<4
Nickel	<10	<10
Silicon	150	≈450
Silver	<0.5	<0.5
Sodium	<10	<10
Tin	<2	<2
Titanium	9	27
Vanadium	<2	<2
Zinc	<50	<50

^(a) Second lot, values given are in ppm of metal and are the mean of three determinations.

Table 6-9. EBR-II Fuel Microsphere Characterization

Analysis	93% ²³⁵ U-enriched (U, Pu)O ₂ microspheres ^(a)	
	420-590 μ	<44 μ
Pu/(Pu + U), wt %	20.51 ± 0.06	19.16 ± 0.06
²³⁵ U content, wt %	92.68 ^(b)	92.68 ^(b)
O/M, atom ratio	1.992 ± 0.006	1.993 ± 0.004
Moisture, ppm oxide	53.5 ± 9.2	88.2 ± 2.0
Sorbed gas, cc/g	0.033 ^(c)	0.033 ^(c)
Density, % theor	91.5	NM
Nitrogen, ppm metal	10.5 ± 5.8	6.5 ± 0.9
Carbon, ppm metal	6.8 ± 0.8	14.0 ± 3.9
Chloride, ppm metal	2.6 ± 0.8	4.7 ± 0.7
Fluoride, ppm metal	0.9 ± 0.2	2.6 ± 0.6

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

(b) ²³⁵U content determined by AVCO.

(c) Sorbed gas analysis was performed on a mixture of 73 wt % coarse and 27 wt % fine microspheres in order to simulate the fuel composition in a pin.

Table 6-10. Trace Metallic Impurities In
EBR-II Microspheres^(a)

	93% ²³⁵ U-enriched (U,Pu)O ₂ microspheres	
	420-590 μ	<25 μ
Aluminum	320	320
Beryllium	<0.5	<0.5
Bismuth	<0.5	<0.5
Boron	1.0	<0.5
Cadmium	3	<0.5
Calcium	50	22
Chromium	45	$\approx$ 330
Copper	10	<5
Iron	260	140
Lead	2	1
Lithium	1	1
Magnesium	9	16
Manganese	8	32
Molybdenum	NM	NM
Nickel	<50	<50
Silicon	125	230
Silver	<0.5	<0.5
Sodium	$\approx$ 80	$\approx$ 320
Tin	<2	<2
Titanium	6	17
Vanadium	<5	<5
Zinc	<30	<30

(a) Values given are in ppm of metal and are the mean of three determinations.

Table 6-11. EBR-II Insulator Pellet Characterization(a)

Analysis	Firing 1	Firing 2	Firing 3
²³⁵ U content, wt %	<0.7 ^(b)	<0.7 ^(b)	<0.7 ^(b)
O/M, atom ratio	2.007 ± 0.000	2.007 ± 0.000	2.007 ± 0.000
Moisture, ppm oxide	10.3 ± 2.2	3.2 ± 0.7	1.1 ± 0.6
Sorbed gas, cc/g	0.004	0.005	0.005
Nitrogen, ppm metal	24.0 ± 11.7	23.8 ± 4.4	23.7 ± 3.9
Carbon, ppm metal	13.8 ± 11.4	12.2 ± 9.1	10.5 ± 5.3
Chloride, ppm metal	2.6 ± 0.9	6.3 ± 2.0	6.8 ± 2.4
Fluoride, ppm metal	0.5 ± 0.3	0.7 ± 0.4	2.6 ± 1.2

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

(b) ²³⁵U content was not determined but should be less than normal.

Table 6-12. Trace Metallic Impurities in EBR-II Insulator Pellets(a)

Element	Firing 1	Firing 2	Firing 3
Aluminum	<15	<15	<15
Bismuth	<2	<2	<2
Boron	0.5	0.4	0.3
Cadmium	<0.3	<0.3	<0.3
Calcium	55	40	40
Chromium	100	47	26
Copper	4	3	<2
Iron	130	150	67
Lead	<2	<2	<2
Lithium	<2	<2	<2
Magnesium	<3	<3	7
Manganese	<6	<6	<6
Molybdenum	21	16	<3
Nickel	<10	<10	<10
Silicon	15	16	26
Silver	<0.25	<0.25	<0.25
Sodium	15	19	59
Tin	22	15	6
Vanadium	<3	<3	<3
Zinc	<50	<50	<50

(a) Values given are in ppm of metal and are the mean of four determinations.

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P.O. Box 999
Richland, Washington 99352
Attn: DeHalas, DR (2)
Eschbech, EA
Evans, EA
Roake, WE
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16. Atomic Power Development Associates, Inc.
1911 First Street
Detroit, Michigan 48226
Attn: Shoudy, AA
-
17. Atomics International
P.O. Box 309
Canoga Park, California 91304
Attn: Pearlman, H
-
18. Battelle Memorial Institute (3)
505 King Avenue
Columbus, Ohio 43201
Attn: Dayton, RW
Keller, DL
Paprocki, S
-
19. The Carborundum Company
P.O. Box 37
Niagara Falls, New York
Attn: Taylor, KM
-
20. Combustion Engineering, Inc.
Prospect Hill Road
Windsor, Connecticut 06095
Attn: Chernock, WP
-
21. The Dow Chemical Company
Rocky Flats Division
P.O. Box 938
Golden, Colorado 80402
Attn: Forest, R
-
22. Gulf General Atomic, Inc.
P.O. Box 1111
San Diego, California 92112
Attn: Turner, RF
-

23. General Electric Company (2)
Atomic Power Equipment Department
P.O. Box 1131
San Jose, California 95108
Attn: Bupp, L
Zebroski, EL
-
24. Nuclear Fuel Services, Inc.
Erwin, Tennessee 37650
Attn: Pedersen, Erik
-
25. Nuclear Materials & Equipment Corporation
609 North Warren Avenue
Apollo, Pennsylvania 15613
Attn: Caldwell, C
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26. United Nuclear Corporation
5 New Street
White Plains, New York 10601
Attn: Strasser, A
-
27. Westinghouse Electric Corporation (2)
Atomic Power Division
P.O. Box 355
Pittsburgh, Pennsylvania 15230
Attn: Allio, R
Murray, P
-
28. J. E. Herbert
ANL-ID
P.O. Box 2108
Idaho Falls, Idaho 83401
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29. P. G. Holsted
Pacific Northwest Laboratory
P.O. Box 550
Richland, Washington 99352
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30. M. E. Jackson
Argonne National Laboratory
9800 South Cass Avenue
Argonne, Illinois 60439
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31. J. V. Levy
General Electric
310 DeGuigne Drive
Sunnyvale, California 94086
-

32. R. L. Morgan
Canoga Park Area Office
P.O. Box 951
Canoga Park, California 91305

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

34. Babcock & Wilcox (40)

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