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FAST REACTOR MIXED-CARBIDE FUEL ELEMENT
DEVELOPMENT PROGRAM

Seventh Quarterly Progress Report
January through March 1969

July 7, 1969

Work Performed on UNC Project 2335
Contract AT(30-1)-3415 with the
New York Operations Office of the
United States Atomic Energy Commission

RESEARCH AND ENGINEERING CENTER / GRASSLANDS ROAD, ELMSFORD, NEW YORK 10523

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1. INTRODUCTION

The Fast Reactor Mixed-Carbide Fuel Element Development Program is aimed at the establishment of mixed carbides as a competitive LMFBR fuel. The major objective of the program is to develop fast breeder reactor fuel rods of (UPu)C clad with stainless steel or higher strength alloys that will perform satisfactorily to a minimum of 50,000 Mwd/T and a maximum of 100,000 Mwd/T at power levels of 10 to 40 kw/ft.

The program is organized by tasks in the following manner:

Task 1200 – High-Density Pellet Evaluation in EBR-II (10 Rods)

Task 1300 – Annular and Low-Density Pellet Evaluation in EBR-II
(24 Rods)

Task 1500 – Unrestrained Swelling and Fission Gas Release

Task 1800 – Sodium-Bonded Fuel Evaluation

Task 1900 – Fabrication Development

1930 – High-Power Rods in EBR-II

1950 – Low-Power Subassembly in EBR-II (19 Rods)

1960 – High-Power Subassembly in EBR-II (19 Rods)

Task 2400 – Uranium-Plutonium Dioxide Pellet Fabrication (Previously
Task 1970).

Progress during the period January 1, 1969 to March 31, 1969 on the currently active tasks of this program is described in this report.

This is the seventh in a series of quarterly progress reports written in partial fulfillment of Contract AT(30-1)-3415 between the United States Atomic Energy Commission and the United Nuclear Corporation.

Prior reports include the following:

Topicals

- UNC-5137 Cihi, J., Hurwitz, S., Rosh, D., and Strasser, A.: The Design and Fabrication of Three (UPu)C Pilot Experiments for EBR-II (Oct. 25, 1965).
- UNC-5170 Andersen, J., Cherry, B., Cihi, J., Powers, R., and Stahl, D.: EBR-II Design and Fabrication Reports of Rods 81 through 90 (Mar. 1967).
- UNC-5198 Stahl, D. and Strasser, A.: Post-Irradiation Examination of High-Density (UPu)C Pellet-Fueled EBR-II Rods Irradiated to 30,000 Mwd/T (Jan. 1968).
- UNC-5229 Powers, R. and Strasser, A.: Compatibility of Several Cladding Materials with Uranium-Plutonium Carbides in Sodium (Nov. 1968).

Progress Reports

Fast Reactor Mixed-Carbide Fuel Element Development Program

- UNC-5193 First Quarterly Progress Report, July through September 1967 (Nov. 15, 1967).
- UNC-5201 Second Quarterly Progress Report, October through December 1967 (Feb. 10, 1968).

- UNC-5217 Third Quarterly Progress Report, January through March 1968
(July 10, 1968).
- UNC-5224 Fourth Quarterly Progress Report, April through June 1968
(Sept. 26, 1968).
- UNC-5233 Fifth Quarterly Progress Report, July through September 1968
(Dec. 12, 1968).
- UNC-5238 Sixth Quarterly Progress Report, October through Decem-
ber 1968 (April 1, 1969).

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2. HIGHLIGHTS OF THE CURRENT QUARTER

Analysis of the clad profilometry data from three rods containing high-density helium-bonded mixed carbide pellets irradiated to 40,000 Mwd/T at 28 kw/ft indicates that the fuel swells in the range of <2.0 to 2.5% $\Delta v/v$ per 10,000 Mwd/T. This is similar to previous rates.

The capsule trains to test unrestrained swelling and fission gas release of mixed carbides started up in the test reactor. The novel, complex instrumented assemblies are measuring fuel temperatures up to 1600 °C. The experiments are operating as designed.

The second all-mixed carbide subassembly was inserted and started up in EBR-II on schedule. The experiment is operating satisfactorily. The first one, also designed and fabricated by UNC, has attained over 27,000 Mwd/T burnup.

Mixed-oxide pellets made from fully enriched ADU source UO_2 and oxalate source PuO_2 were sintered with essentially no hourglassing and diametral variations of less than ± 0.0015 in. The plutonium homogeneity was excellent and 100% solid solution was obtained.

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3. PAST RESULTS AND CURRENT PROGRESS

FAST REACTOR IRRADIATION

3.1 TASK 1200 — HIGH-DENSITY PELLET EVALUATION IN EBR-II (10 RODS)

Introduction and Past Results

The objective of the task is to evaluate the performance of high-density (96% of theoretical, minimum) uranium-plutonium carbide pellets with varying size helium annuli between fuel and clad to provide void space for fuel swelling. The experiments are designed to determine the fuel-clad gap and clad strength combination to give satisfactory performance.

Ten doubly encapsulated fuel rods (Rods UNC 81 through 90) were designed, and nine were inserted for irradiation in EBR-II at powers of 20 to 30 kw/ft to burnups of 50,000 to 100,000 Mwd/T. The fuel is high-density, helium-bonded (UPu)C which was prepared by carbothermic reduction of the mixed dioxides followed by cold pressing and sintering of pellets. The cladding is Type 316 stainless steel and Incoloy -800. Eight of the capsules contain full-length prototypic fuel rods, while two capsules contain four segmented experiments each. A detailed description of these experiments is given in Table 1 and UNC-5170.

Rods UNC 87, 89, 90 (28 kw/ft) were discharged at the end of EBR-II Run 29 (August 1968) with a peak exposure of 40,000 Mwd/T. Neutron radiographs of the capsule showed that all three were in good condition.

TABLE 1 — SUMMARY OF CURRENT UNITED NUCLEAR MIXED-CARBIDE IRRADIATION EXPERIMENTS

Task No.	UNC Experiment No.	Description of Experiment	Design Parameters								Operating Conditions				Status
			Pellet Diameter, in.	Fuel Length, in.	Density, % T.D.		a/o Pu	Cladding Material	Maximum Linear Power (Design), kw /ft	Target Burnup, Mwd/T $\times 10^{-3}$	Facility	Operating Maximum Linear Power, kw /ft	Accumulated Burnup, Mwd/T $\times 10^{-3}$		
					Pellet (Type)	Smeared									
1200	81-88	EBR-II rods, helium-bonded, (8 rods)	0.248 to 0.252	14	98 (solid)	81 to 86	20	316 SS and Inco-loy-800	20 to 30	50 to 100	EBR-II	19 to 27	42,000	Rods 81-86 being irradiated, Rod 88 not yet inserted. Rods 87, 89, 90 irradiation complete.	
	89-90	EBR-II multiple specimen rods, helium-bonded (2 rods)	0.249 to 0.252	4	98 (solid)	82 to 90	20	316 SS and Inco-loy-800	24 to 30	40	EBR-II	22 to 28	40,000		
1300	91-114	EBR-II rods, helium-bonded (24 rods)	0.232 to 0.269	14	75 to 84 (solid), 99 (cored)	70 to 85	15	316 SS and Inco-loy-800	20	50 to 100	EBR-II	17 to 23	27,700	19-rod assembly being irradiated.	
1500	115-119	Unrestrained swelling specimens	0.06	0.5	75 to 90 (solid)	—	15	—	Low	20	Hanford	—	—	Irradiation in progress	
	120-124	Fission gas release specimens	0.375 OD, 0.255 ID	0.500	75 to 98 (annular)	—	15	—	Low	20	Hanford	—	—		
1800	125-128	Sodium-bonded rods (4 rods)	0.240	14	93 to 98 (solid)	83 to 87	15	304 SS and Inco-loy-800	25	50	GETR	25	25,000	Rods being irradiated.	
1950	129-147	19-rod low-power subassembly, helium and sodium bonds	0.240 to 0.246	14	75 to 98	70 to 87	15	316 SS, 304 SS, and Inco-loy-800	15	100	EBR-II	15	1,500	Irradiation in progress	
1930	185-190	High-power rods (6 rods)	0.249 to 0.253	14	84 to 96 (solid)	80 to 88	15	316 SS and Inco-loy-800	30	50 to 100	EBR-II	—	—	Capsules awaiting insertion at EBR-II	
1960	191-209	19-rod high-power subassembly, helium and sodium bonds	—	14	84 to 96	—	15	316 SS, 304 SS, and Inco-loy-800	30	30 to 100	EBR-II	—	—	Design completed.	

The fuel rods were removed from their respective capsules. All the rods were intact with the exception of one specimen in UNC 89, the Type 316 clad multispecimen rod. The specimen with 0.002-in. cold diametral gap and 96.6% smear density had three thin, brittle, longitudinal cracks in the fueled segment. The three cracks were 1-in., 1/2-in., and 1/4-in. long. In comparison, the identical specimen clad with Incoloy-800 was in excellent condition.

Current Results

The calculated burnup of the nine rods as of December 31, 1968, in EBR-II is as follows:

1. Rods UNC 81 through 83 (19 kw/ft) – 40,000 Mwd/T
2. Rods UNC 84 through 86 (19 kw/ft) – 42,200 Mwd/T
3. Rod UNC 88 is scheduled for insertion in May 1969.

Evaluation of the nondestructive examination data from LASL on UNC 87, 89, and 90 was completed. A summary of the design, irradiation conditions, and profilometry data is shown in Tables 2, 3, and 4. Diameter increases in the order of 0.003 in. were observed in both the Type 316 SS and Incoloy-800 clads. The significant result is that with a diameter increase of ~0.003 in., the Type 316 SS had a failure, while under almost identical conditions, the Incoloy-800 did not fail. The predicted clad strain in both cases was in the order of 0.5% while the actual strain was ~1%.

The following tentative conclusions were drawn from the data:

1. The swelling $\Delta v/v$ of (UPu)C is similar to the previously determined value. It ranges from <2.0% to 2.5% per 10,000 Mwd/T.
2. Incoloy-800 has a higher ductility, but may swell more than Type 316 SS.
3. The effect of power on clad ΔD may be greater than the effect of initial fuel-to-clad gap.

TABLE 2 — SUMMARY OF IRRADIATION CONDITIONS — UNC 87, 89, 90

Fuel: 98% dense, $(U_{0.8}Pu_{0.2})C + 10 \text{ v/o } (UPu)_2C_3$ pellets, 1/4-in. diamPeak burnup, Mwd/T: 40,000; fission/cm³: 1.4×10^{21} Peak clad fluence, nvt: 1.1×10^{22}

	UNC 87	UNC 89				UNC 90			
		A	B	C	D	A	B	C	D
Clad	Incoloy -800	← Type 316 stainless steel →				← Incoloy -800 →			
Fueled Length, in.	14	2	2	2	2	2	2	2	2
Fuel-Clad Gap, in. (diametral, cold at startup)	0.006	0.006	0.002	0.006	0.009	0.007	0.003	0.006	0.009
Smear Density, % T.D.	94.2	93.8	96.6	94.2	92.1	93.9	96.7	94.3	92.2
Clad Thickness, in.	0.022	← 0.024 →				← 0.022 →			
Calculated Maximum Fuel Temp at Startup, °C	2065	2090	2115	1815	2055	2090	2115	1815	2055
Calculated Clad Surface Temp, °C	660	585	590	590	535	585	590	590	535
Calculated (Predicted) Maximum Clad Strain, %	0.0	0.0	0.7	0.0	0.0	0.0	0.5	0.0	0.0
Maximum Linear Power, w/cm	820	820	875	780	565	475	605	605	525
Maximum Clad Surface Heat Flux, w/cm ²	295	295	315	280	200	170	215	215	190
Clad Condition after Irradiation	Intact	Intact	Cracked	Intact	Intact	Intact	Intact	Intact	Intact

TABLE 3 — SUMMARY OF CLAD DIAMETER CHANGES — UNC 87, 89, AND 90 —
IRRADIATED TO A PEAK BURNUP OF 40,000 MWD/T

	<u>Clad</u>	<u>Fuel- to-Clad Gap, in.</u>	<u>Value</u>	<u>Pre - Irradiation, in.</u>	<u>Post - Irradiation, in.</u>	<u>$\Delta D(\pm 0.0002),$ in.</u>
UNC -87	Incoloy -800	0.006	Average	0.3020	0.3032	0.0015
			Peak	0.3021	0.3051	0.0032
UNC -89						
A	Type 316 SS	0.006	Average	0.3059	0.3059	0.0000
			Peak	0.3060	0.3060	0.0001
B	Type 316 SS	0.003	Average	0.3058	0.3073	0.0014
			Peak	0.3059	0.3092	0.0035
C	Type 316 SS	0.006	Average	0.3058	0.3067	0.0009
			Peak	0.3060	0.3076	0.0018
D	Type 316 SS	0.009	Average	0.3058	0.3060	0.0002
			Peak	0.3059	0.3060	0.0004
UNC -90						
A	Incoloy -800	0.006	Average	0.3017	0.3020	0.0004
			Peak	0.3018	0.3022	0.0006
B	Incoloy -800	0.003	Average	0.3018	0.3036	0.0018
			Peak	0.3018	0.3048	0.0030
C	Incoloy -800	0.006	Average	0.3017	0.3035	0.0018
			Peak	0.3018	0.3046	0.0029
D	Incoloy -800	0.009	Average	0.3016	0.3025	0.0009
			Peak	0.3019	0.3027	0.0012

TABLE 4 — FUEL ROD SWELLING DATA — UNC 87, 89, AND 90 —
IRRADIATED TO A PEAK BURNUP OF 40,000 MWD/T

	Total Fuel Rod $\Delta v/v$ per 10,000 Mwd/T, <u>%</u>	Estimated Fuel $\Delta v/v$ per 10,000 Mwd/T, <u>%</u>	Total* $\Delta D/D$, <u>%</u>	Percent† Strain
UNC-87	0.82	2.5	1.06	0.91
UNC-89				
A	0.09	<2.0	0.03	0.0
B	0.87	2.0	1.14	1.04
C	0.49	2.5	0.59	0.52
D	0.09	2.3	0.13	0.12
UNC-90				
A	0.09	2.5	0.20	0.12
B	0.73	2.5	0.99	0.89
C	0.64	2.3	0.96	0.89
D	0.18	2.3	0.40	0.89

*per 40,000 Mwd/T.

†Total $\Delta D/D$ - clad $\Delta D/D$ per 40,000 Mwd/T.

4. Both the 6- and 3-mil gap Incoloy -800 specimens show a similar clad OD change (ΔD), whereas in Type 316 SS the ΔD is different for the same specimens.
5. No measurable clad OD change occurred in the Type 316 SS specimens with a 9-mil gap.
6. At the peak exposure for these fuel rods the clad swelling is small and well within measurement error, therefore, does not contribute appreciably to the total swelling.

The experiments also have shown that in the Incoloy -800 specimens, the 2-in. fuel specimen gives data which are comparable to the 14-in. fuel specimens. This indicates that the multispecimen design could be used for screening a number of design variables in a minimum of reactor space.

The schedule for the destructive examination has not been defined because of higher priority work at the LASL hot cells.

3.2 TASK 1300 — ANNULAR AND LOW-DENSITY PELLET EVALUATION IN EBR-II

Introduction and Past Results

The objective of the task is to evaluate various methods of void deployment in helium-bonded uranium-plutonium carbide pellets to provide void space for fuel swelling. Specifically, high-density annular (97% of theoretical) and low-density pellets (75 and 84% of theoretical) with various smear densities are being evaluated to determine the best fuel rod design.

Rods UNC 91 through 144 were designed for irradiation in EBR-II at powers of 20 kw/ft to burnups of 50,000 to 100,000 Mwd/T. Design variables include power, burnup, clad material and thickness, fuel structure, and fuel smear density. Design details are shown in Table 1.

Nineteen rods for an EBR-II subassembly were fabricated. The 19 rods were accepted by ANL-Idaho, then assembled into subassembly X033, and inserted in the reactor. This represents the first mixed-carbide subassembly ever to be irradiated.

Current Results

The 19-rod subassembly (X033) is operating in the EBR-II at about 20 kw/ft, and has accumulated an exposure of 27,700 Mwd/T.

3.3 TASK 1930 — HIGH-POWER RODS IN EBR-II

Introduction and Past Results

The objective of the high-power rod experiments is to extend the investigation of the irradiation behavior of helium-bonded (UPu)C fuel to higher linear power. Fission gas release and restrained swelling data are to be obtained for a total of six rods in an EBR-II subassembly with a maximum linear power of 30 kw/ft. The design variables include Incoloy-800 and Type 316 stainless steel cladding, high- and low-density fuel pellets, and fuel burnups of 50,000 to 100,000 Mwd/T.

The design was completed and the test parameters for the capsules are given in Table 1.

The six rods, UNC 185 through 190, were fabricated, encapsulated in the standard EBR-II capsules, sodium-bonded, and shipped to the EBR-II for irradiation.

Current Results

The insertion of the six rods into the EBR-II has been scheduled for May 1969.

3.4 TASK 1950 — LOW-POWER SUBASSEMBLY IN EBR-II (19 RODS)

Introduction and Past Results

The objective of the experiment is to obtain performance data on uranium-plutonium carbide fuel rods at low powers. Fuel swelling data are of particular interest, because a large portion of an LMFBR will be operating at less than the 20 to 30 kw/ft peak power range at which the carbides are expected to operate.

The design was completed and the test variables are given in Table 1. A program was completed to develop an end-plug design for use with an automatic welder.

The preparation of all fuel rod components has been completed.

Fuel fabrication of the first ($U_{0.85}Pu_{0.15}$)C subassembly to be fabricated in the pilot facility was completed. Rods UNC 129 through 147 were loaded and prepared for encapsulation in standard EBR-II capsules.

Current Results

Fuel rods UNC 129 through 147 were encapsulated into standard EBR-II capsules and shipped to the reactor.

The subassembly X055 containing Capsules UNC 129 through 147 was assembled by ANL and placed in the EBR-II at 10 to 15 kw/ft. The accumulated burnup is 0.53×10^{20} fiss/cm³ (1500 Mwd/T).

This is the second all mixed-carbide subassembly ever to be irradiated.

3.5 TASK 1960 — HIGH-POWER SUBASSEMBLY IN EBR-II (19 RODS)

Introduction

The objective of the 19-rod experiment is to extend the current irradiation study to higher powers (30 to 35 kw/ft).

High linear and specific powers at high burnup are necessary in fast reactor fuels to reduce fuel cycle costs significantly. The most critical problem of high-power, high-burnup fuels is the accommodation of fuel swelling without rupturing the clad.

The classical solution is a controlled helium gap between the fuel and clad. As an alternative approach, accommodation for swelling can be provided within the fuel. This can be in the form of a central void (cored pellets) or as uniformly distributed porosity. The third approach is the use of sodium fuel-to-clad bond. The sodium provides expansion space, eliminates clad strain, and reduces fuel temperatures.

The design of the 19-rod subassembly is complete and test variables are given in Table 1.

Current Results

The cladding has been ordered and fabrication of metal components for fuel rods UNC 191 through 199 completed.

Re-evaluation of the power output for the subassembly, based on the latest fission rates for the EBR-II, has been completed. The subassembly will operate between 26 and 29 kw/ft rather than 30 to 34 kw/ft. The reduced power will be caused by an enlarged EBR-II core which has depressed the flux near the core center.

Fabrication of the fuel for the 19-rod subassembly was initiated.

MC + M₂C₃ for Na-Bonded Rods

A 1300-g batch of (UPu)C was synthesized by the standard method. Chemical analysis of the product was satisfactory and is given below.

Carbon, w/o	4.77 ± 0.02
Plutonium, w/o	14.34 ± 0.04
Oxygen, ppm	970 ± 160

Pellets for test sinters of the two-phase high-density pellets with 0.1% nickel sintering aid were fabricated by the standard method. Problems were encountered in the initial test sinters by: first, surface oxidation of the pellets from impurities on the heat shields, and second, by the formation of some continuous networks of M₂C₃ near the pellet surface. The latter is undesirable because it may promote carbon transfer to the clad in sodium-bonded rods. A subsequent test sinter with clean heat shields and under a high vacuum (<10 μ Hg) eliminated both the oxidation and the traces of continuous M₂C₃ networks.

Based on the test sinters, pellets for loading of UNC rods 194, 195, 197, and 198 were compacted and sintered at 1500°C-1 hr under a high vacuum. Characterization results are shown in Tables 5A and 5B and indicate they met specifications.

Metallographic examination of some pellets revealed a continuous layer of sesquicarbide approximately 5-microns thick at the pellet surface preceded by a trace amount of dicarbide that extended to a depth of about 1 mil. The pellets will be surface ground to remove 1 mil from the surface prior to rod loading.

Pellets for Rods UNC 196 and 199 were fabricated by the modified procedure. The pellets for rod UNC 196 were surface ground to remove 0.009 in.

TABLE 5A — CHARACTERIZATION OF THE HIGH-DENSITY MC + M₂C₃ PELLETS
FOR LOADING OF UNC RODS 194, 195, 197, AND 198

<u>UNC Rod No.</u>	<u>Cold Formed Density, g/cm³</u>	<u>Intended Sintered Diameter, in.</u>	<u>Sintered Diameter, in.</u>	<u>Sintered Density, g/cm³</u>	<u>% T.D.</u>
194, 195	8.15 ± 0.01	0.241 ± 0.001	0.2403 ± 0.0001	13.12 ± 0.03	98
197, 198	8.12 ± 0.02	0.241 ± 0.001	0.2404 ± 0.0002	13.10 ± 0.02	98

TABLE 5B — CHARACTERIZATION OF THE HIGH-DENSITY MC + M₂C₃ PELLETS FOR
LOADING OF UNC RODS 194, 195, 197, AND 198 (TASK 1962)

<u>UNC Rod No.</u>	<u>C, w/o</u>	<u>Pu, w/o</u>	<u>O₂, ppm</u>	<u>N₂, ppm</u>	<u>Equivalent Carbon* w/o</u>	<u>X-Ray Diffraction Lattice Parameter (Å) and Phases</u>
194, 195	4.78 ± 0.07	14.39 ± 0.04	2405 ± 190	660 ± 120	5.02	93 v/o (UPu)C a ₀ = 4.9651 ± 0.0003 7 v/o (UPu) ₂ C ₃ a ₀ = 8.0940 ± 0.0023
197, 198	4.71 ± 0.05	14.45 ± 0.04	2505 ± 155	90 ± 15	4.91	

*Carbon equivalent (w/o C + 12/16 w/o O₂ + 12/14 w/o N₂).

from the pellet OD to remove the normal single-phase MC surface layer and to expose the representative MC + M₂C₃ structure to the Na bond.

Characterization of the pellets for loading of rod UNC 196 is given in Table 6. The pellets for Rod UNC 199 have been sintered and are awaiting grind-

TABLE 6 — CHARACTERIZATION OF THE HIGH-DENSITY
MC + M₂C₃ PELLETS FOR LOADING OF ROD UNC 196

Surface Ground Diameter,* <u>in.</u>	Sintered Density, <u>g/cm³</u>	C, <u>w/o</u>	Pu, <u>w/o</u>	O ₂ , <u>ppm</u>	N ₂ , <u>ppm</u>
0.2410 ± 0.0003	13.17 ± 0.02	4.78 ± 0.04	14.32 ± 0.01	2925 ± 780	280 ± 230

*Pellets were surface ground to remove the 0.009-in. single-phase MC layer.

ing and characterization.

Single-Phase MC for Na-Bonded Rods

A 650-g batch of (UPu)C was synthesized by the standard method for fuel rods UNC 191 through 193. Chemical analysis on the product showed it met specification:

Carbon, w/o	4.64 ± 0.03
Plutonium, w/o	14.46 ± 0.06
Oxygen, ppm	3185 ± 250

3.6 TASK 1500 — UNRESTRAINED SWELLING AND FISSION GAS RELEASE

Introduction and Past Results

The objective of the experiments is to investigate the unrestrained swelling and fission gas release characteristics of several types of (UPu)C fuel as a function of irradiation exposure and temperature. Since temperature measurement is essential to obtain meaningful data, the experiments have to be carried out in instrumented capsules; this is feasible in thermal reactors only. The problem of flux depression and resulting large ΔT 's, which are not representative of fast reactors, is resolved by the use of small cross-section specimens operating at essentially isothermal conditions.

Design of the two capsule trains was completed and a description of the capsule trains was given in the third quarterly report, UNC-5217.

A capsule to measure the neutron flux in the experimental facility was irradiated. Evaluation of the flux monitor data showed good agreement between the measured and calculated neutron flux in the fuel.

The gas control systems and control consoles have been fabricated by Douglas-United Nuclear at the reactor experiment site.

Fabrication of the various U-Pu carbide and mixed-oxide fuels for the fission gas release and unrestrained swelling studies is complete. Subsequently, the capsule trains were assembled and shipped to Douglas-United Nuclear for irradiation.

A thermal analysis of the mixed-oxide unrestrained swelling pins has shown that they can be operated in the capsule designed to operate at 1300°C for mixed carbide and be controlled to 1600°C.

Current Results

Both the unrestrained swelling and fission gas release experiments were inserted in the reactor and are operating as designed. The intended and actual fuel temperatures are summarized in Table 7. The temperatures are being controlled within $\pm 25^{\circ}\text{C}$.

UNC 124A and B is at 80% of intended temperature because it is being shadowed by a control rod. An attempt will be made to relocate the control rod. The capsule contains 98% and 84% mixed-carbide unrestrained swelling specimens.

UNC 118A is at 85% of intended temperature while its companion capsule, UNC 118B – controlled by the same gas mixture – is at the intended temperature. The pins in UNC 118A are probably no longer intact and have fallen to one side of the specimen holder. The UNC 118 capsule contains mixed-oxide unrestrained swelling specimens.

TABLE 7 — SUMMARY OF CAPSULE TRAIN
OPERATING CONDITIONS

Unrestrained Swelling Experiment

<u>UNC Capsule</u>	<u>Fuel</u>	<u>% T.D.</u>	<u>Fuel Temp, °C*</u>	
			<u>Design</u>	<u>Actual</u>
116A	MC + M ₂ C ₃	98	1300	1289
116B	MC	84	1300	1340
117A	MC	93	1300	1325
117B	M(OC)	93	1300	1360
118A	MO ₂	>90	1600	1366
118B	MO ₂	>90	1600	1578
119A	MC + M ₂ C ₃	98	1600	†
119B	MC	84	1600	1604

Fission Gas Release Experiment

120A	MC + M ₂ C ₃	98	1000	1003
120B	MC	84	1000	978
121A	MC + M ₂ C ₃	98	1300	1275
121B	MC	84	1300	1375
122A	MC	93	1300	1308
122B	MC	75	1300	1375
123A	M ₂ C ₃	93	1300	1288
123B	M(OC)	93	1300	1398
124A	MC + M ₂ C ₃	98	1600	1298
124B	MC	84	1600	1320

*The "A" specimen is the control specimen and is operated at $\pm 25^{\circ}\text{C}$ of the design temperature.

†Thermocouple not operating. Control is on the "B" side and temperature is monitored with the peripheral thermocouples.

3.7 TASK 1800 — SODIUM-BONDED FUEL EVALUATION

Introduction and Past Results

The objective of the irradiations is to evaluate the performance of sodium-bonded, uranium-plutonium carbide rods. The test parameters for the capsules are given in Table 1.

Fabrication of the fuel experiments is complete. This represents the first time a long stack (14-in.) of uranium-plutonium carbide fuel pellets has been bonded with sodium.

The four instrumented capsules, UNC 125 through 128, were shipped to GETR, inserted, and are operating according to design.

Current Results

Capsules UNC 125, 126, 127, and 128 are operating at 25 kw/ft in the GETR. Each capsule contains one sodium-bonded fuel rod with a 14-in. long fueled length. All systems are operating according to design and without any thermocouple failures. The accumulated burnup for the capsules is as follows:

<u>Capsule</u>	<u>Burnup,</u>	
	<u>fiss/cm³</u>	<u>Mwd/T</u>
UNC 125	8.8×10^{20}	25,000
UNC 126	7.7×10^{20}	22,000
UNC 127	5.5×10^{20}	15,600
UNC 128	8.8×10^{20}	25,000

3.8 TASK 2400 — URANIUM-PLUTONIUM DIOXIDE PELLET FABRICATION

Introduction and Past Results

The purpose of this task is to develop methods for producing well characterized (UPu)O₂ pellets and fuel rods of the LMFBR/FFTF type. The fuel produced will be subjected to a high level of quality control. The quality control methods themselves will be established and improved to the levels required by the specification.

A. Fabrication

Two processes are being evaluated for preparation of press feed in the pilot plant process equipment. The two processes were described in the fourth quarterly report, UNC-5224.

Pressing and sintering studies, with UNC depleted UO₂ and burned PuO₂, to evaluate the effect of variations in cold-formed density showed that a 0.1 g/cm³ change in cold-formed density results in a 0.001-in. change in sintered pellet diameter. The importance of control of press feed characteristics and press performance in obtaining diametral reproducibility is apparent. The density and diametral reproducibility for fabrication of sintered dioxide pellets was ± 0.04 g/cm³ and ± 0.001 in., respectively. The oxygen-to-metal ratio was 1.95, and X-ray diffraction analysis established that complete solid solution was not obtained.

UO₂-25 w/o PuO₂ press feed containing 0.25 w/o PVA and 0.7 w/o zinc stearate was milled in an attempt to reduce particle size and improve homogeneity. Pellets were cold pressed directly from the milled powder and sintered. X-ray diffraction and O/M analysis showed complete solid solution was not obtained and the O/M was 1.94.

B. Characterization

A Perkin-Elmer Shell Sorptometer is being adapted to a glove box to measure surface area of fuel powders.

Development continued of the instrument for measuring the open and closed porosity of fuel pellets by comparison of geometric and helium displacement volumes.

Current Results

A. Fabrication

Starting materials designated for FFTF pellet fabrication are PuO_2 produced by the oxalate route and natural UO_2 . Characterization of these materials used in fabrication studies is shown in Table 8. During the initial analytical determination, the PuO_2 was observed to be very hygroscopic. The rapid sorption of moisture from the air atmosphere of the glove box prevented accurate gravimetric determinations. A series of tests demonstrated that when PuO_2 was exposed to the glove-box atmosphere, the weight gain due to moisture remained constant for a period of 4 hr. The exposure of oxalate source PuO_2 to the glove box atmosphere for 4 hr has been adopted as a standard procedure prior to weighing for batching of mixed oxides. The as-received UO_2 powder was found to be relatively stable and did not require prior treatment to obtain an accurate O/M value.

A 3-kg batch of UO_2 -24 w/o PuO_2 was prepared according to the flow diagram shown in Fig. 1. Characterization of the UO_2 -24 w/o PuO_2 powder after the ball-milling operation is summarized in Tables 9A and 9B. After granulation, the bulk density was 2.36 g/cm^3 .

Pellets were pressed by a hydraulic automatic press with green densities between 5.0 and 6.5 g/cm^3 . Sintering of 10 pellets at each of four green densities

TABLE 8 — CHARACTERIZATION OF AS-RECEIVED NATURAL
ELDORADO UO_2 AND OXALATE PRECIPITATED PuO_2

	<u>Eldorado UO_2</u>	<u>Oxalate PuO_2</u>
Bulk Density, Scott Volumeter, g/cm^3	0.77	1.40
Particle Size, microns	100% <3.5	100% <5.5
Surface Area, m^2/g	9.3	47.5
O/M	2.215	2.012
Lattice Parameter, Å	Single-phase $a_0 = 5.4020$	Single-phase $a_0 = 5.4650$

TABLE 9A — CHARACTERIZATION OF
 UO₂-24 W/O PuO₂ POWDER AFTER
 BALL MILLING

	<u>Analysis</u>
Carbon, %*	1120 to 1300
Uranium, %	66.74 ± 0.08
Plutonium, %	20.81 ± 0.07
Powder Bulk Density, g/cm ³	1.07

*Carbon content of as-received PuO₂ was
 1625 ± 55 ppm. Analysis determined at
 UNC.

TABLE 9B — SPECTROCHEMICAL ANALYSIS OF UO₂-24 W/O
 PuO₂ POWDER AFTER BALL MILLING FOR 12 HR

<u>Element</u>	<u>ppm</u>	<u>Element</u>	<u>ppm</u>
Ag	<0.1, <0.1	Mn	2.8, 2.7
Al	<5, <5	Mo	ND <2, ND <2
As	ND <10, ND <10	Na	<1, <1
Au	ND <0.5, ND <0.5	Nb	ND <5, ND <5
B	ND <0.1, ND <0.1	Ni	8.5, 7.0
Ba	<10, <10	P	ND <25, ND <25
Be	ND <2, ND <2	Pb	2.9, 2.5
Bi	ND <2, ND <2	Rb	ND <5, ND <5
Ca	<10, <10	Ru	ND <3, ND <3
Cd	ND <0.5, ND <0.5	Sb	ND <3, ND <3
Co	ND <1, ND <1	Si	17, 19
Cr	15, 11	Sn	<2, <2
Cs	ND <5, ND <5	Sr	ND <50, ND <50
Cu	1.6, 1.6	Ta	ND <100, ND <100
Fe	33, 27	Te	ND <10, ND <10
Ga	ND <10, ND <10	Ti	1.0, 1.1
Ge	ND <2, ND <2	Tl	ND <10, ND <10
Hg	ND <10, ND <10	V	ND <5, ND <5
In	ND <10, ND <10	W	ND <50, ND <50
K	<1, <1	Zn	<10, <10
Li	<1, <1	Zr	ND <5, ND <5
Mg	9.2, 10.4	C	*

*Carbon content of as-received Pu was 1625 ± 55 ppm.

Fig. 1 — Flow Diagram for UO₂-PuO₂ Press Feed Preparation

was performed in a batch-type furnace with a tungsten element. The sintering atmosphere was a moist He-2% H₂ atmosphere at 1600°C for 4 hr. The density of all sintered pellets was within the range of $93 \pm 2\%$ of theoretical. All sintered pellets had a taper which increased toward the bottom. Pellet characterization data are summarized in Table 10A.

A second sinter was performed with 10 pellets each of four green densities. Sintering was at 1650°C-1/2 hr and 1700°C-2 1/2 hr. There was no appreciable change in the sintered pellet density or diameter from that of Sinter 1. The excessive amount of pellet taper is attributed to insufficient travel of the lower platen during the pressing cycle.

A third sinter was performed to produce pellets to meet the specification using the existing larger diameter die. The pellets were to be centerless ground to 0.2120 ± 0.0015 in. From data generated by the first sinter, and allowing for hourglassing associated with the high sinter shrinkage, a green density of 5.8 ± 0.05 g/cm³ was selected to produce a target sintered diameter of 0.215 in., sintered length of 0.250 ± 0.010 , and sintered density of 10.3 ± 0.10 g/cm³. Seventy-five pellets were pressed to the green density specification above and sintered. The average sintered pellet density was 10.35 ± 0.05 g/cm³ ($93.4 \pm 0.5\%$) and the average diameter was 0.2151 in. Sintered pellet data are summarized in Table 10B. The hourglassing ranged from 0.4 to 3.3 mils. Six pellets out of the 75 failed to meet the sintered length specification of 0.250 ± 0.010 in. Three pellets were too long, range 6.7 to 8.6 mils, and three were short, range 0.5 to 5.2 mils. Ten pellets were measured for perpendicularity and all met a specification of ± 1 mil. The pellet diametral tolerance range was -0.9 to +0.7 mils.

Irradiation experiments are planned using enriched UO₂ and the sintering characteristics of enriched UO₂+25 w/o PuO₂ mixtures are not known. Sintering characteristics for these starting materials are required to determine the die bore size and number of die sets needed to fabricate the pellets. Fully enriched UO₂, on

TABLE 10A — CHARACTERIZATION OF UO₂-24 W/O PuO₂ PELLETS FROM THE FIRST SINTER BATCH*

Compaction Pressure, psi	Cold-Formed Pellet Density, g/cm ³	Sintered Pellet Data — 1600°C-4 hr				Sintered Pellet Analytical Data		
		Density		Diameter, in.	Taper, mils	Pu, %	O/M	Solid Solution, %
		g/cm ³	% T.D.					
—	5.0	10.38 ± 0.03	93.7	0.1898 ± 0.0003	2.3 ± 0.7	21.29	2.00	65 ± 5
54,000	5.5	10.43 ± 0.02	94.1	0.1974 ± 0.0019	2.0 ± 0.4	—	—	—
78,000	6.0	10.47 ± 0.07	94.2	0.2021 ± 0.0010	1.0 ± 0.5	—	—	—
132,000	6.5	10.43 ± 0.06	94.1	0.2079 ± 0.0004	1.4 ± 0.4	—	—	—

*Sintered in He-2% H₂ atmosphere moisture in exit gas 200 to 1200 ppm.

TABLE 10B — CHARACTERIZATION OF UO₂-24 W/O PuO₂ PELLETS FROM THE THIRD SINTER BATCH*

Compaction Pressure, psi	Cold Formed Pellet Density, g cm ³	Sintered Pellet Data† - 1600 C-4 hr							Sintered Pellet Analytical Data			
		Density			Diameter, in.	Taper, mils	Hour- glass, mils	Pu, %	O /M	Solid Solution, %		
		g cm ³	% T.D.									
56,000	5.80 0.05	10.35 0.05	93.4	0.2151 + 0.0014 - 0.0010	0.1 - 2.0	0.4 - 3.3	21.29 0.05	1.99	65 ± 5			

*Sintered in He-2% H₂ atmosphere moisture in exit gas 200 to 1200 ppm.

†Data from 28% of sintered lot.

hand, was used for a series of test sinters to evaluate sintering characteristics.

A 300-g batch of UO_2 -25 w/o PuO_2 was prepared from Kerr McGee 93% enriched UO_2 and oxalate prepared PuO_2 . The UO_2 -25 w/o PuO_2 mixture was ball milled in a rubber-lined mill for 16 hr. Press feed was prepared with 0.25 w/o PVA. The dried material was granulated, then lubricated with 0.3 w/o zinc stearate. The press feed was compacted in an automatic hydraulic press to average green densities of 5.06, 5.57, 6.03, and 6.39 g/cm³. The compacting pressure ranged from 34,000 to 100,000 psi for green densities ranging from 5.06 to 6.39 g/cm³.

Sintering operations were performed in a He-2% H_2 atmosphere with moisture content of the exit gas ranging from 2400 to 3700 ppm. Test sinters were performed at 1550, 1625, and 1700 °C with a hold-time of 4 hr at the final temperature. The results of the three sinters are reported in Table 11. The data generated from the series of three sinters indicate that pellets can be fabricated to meet a sintered-density specification of 89 to 95 ± 1½% T.D. with fully enriched UO_2 without calcining the UO_2 . From these data, dies will be designed and ordered to fabricate pellets. Analytical characterization of pellets from the 1550 °C sinter is in process.

B. Characterization

1. Plutonium Dispersion

Development of methods and standards to enable an accurate estimate of plutonium dispersion is in process. Methods under investigation are alpha autoradiography to determine particle size and relative amounts of plutonium contained in discrete particles, and X-ray diffraction to determine the amount of solid solution and secondary phases.

a. Pu Dispersion by Alpha Autoradiography

A program was started to establish a standard alpha autoradiograph-

TABLE 11 — CHARACTERIZATION DATA FOR 93% ENRICHED UO_2 -25 W/O PuO_2 PELLETS*

Data for Pellets Sintered at 1550 C-4 hr						
Compaction Pressure, psi	Cold Formed Density, g/cm ³	Density		Diameter, in	Taper, mils	Hourglass mils
		g/cm ³	% T D			
34 000	5.06	9.71 ± 0.07	88.4 ± 0.6	0.1955 + 0.0006 - 0.0007	0.47 + 0.33 - 0.37	0.52 + 0.28 - 0.22
58 000	5.57	9.93 + 0.07 - 0.05	90.3 + 0.6 - 0.5	0.2005 + 0.0008 - 0.0012	1.0 + 0.20 - 0.50	—
74 000	6.03	10.11 + 0.15 - 0.10	92.0 + 1.4 - 1.0	0.2039 + 0.0009 - 0.0012	0.54 + 0.26 - 0.44	—
100 000	6.39	10.25 + 0.08 - 0.06	93.3 + 0.7 - 0.6	0.2071 + 0.0011 - 0.0009	0.10 + 0.30 - 0.0	—
Data for Pellets Sintered at 1700 C-4 hr						
34 000	5.06	10.38 + 0.10 - 0.08	94.4 + 0.9 - 0.7	0.1910 + 0.0007 - 0.0004	0.8 + 0.6 - 0.2	1.0 + 0.3 - 0.2
58 000	5.57	10.51 + 0.06 - 0.08	95.6 + 0.6 - 0.7	0.1966 + 0.0003 - 0.0005	1.5 + 0.5 - 0.4	—
74 000	6.03	10.52 + 0.08 - 0.06	95.7 + 0.7 - 0.5	0.2016 + 0.0004 - 0.0003	1.2 + 0.1 - 0.2	—
50 000	6.39	10.53 + 0.07 - 0.04	95.8 + 0.6 - 0.4	0.2058 + 0.0008 - 0.0005	0.1 + 0.3 - 0.1	—
Data for Pellets Sintered at 1625 C-4 hr						
34,000	5.06	10.13 + 0.09 - 0.13	92.2 + 0.8 - 1.18	0.1922 + 0.0007 - 0.0005	0.6 + 0.4 - 0.3	0.8 + 0.4 - 0.3
58 000	5.57	10.21 + 0.07 - 0.04	92.9 + 0.6 - 0.4	0.1987 + 0.0004 - 0.0002	1.5 + 0.2 - 0.3	—
74 000	6.03	10.33 + 0.05 - 0.07	94.0 + 0.5 - 0.6	0.2028 + 0.0002 - 0.0008	1.0 + 0.3 - 0.2	—
50 000	6.39	10.43 + 0.07 - 0.14	94.9 + 0.64 - 1.27	0.2063 + 0.0008 - 0.0003	0.6 + 0.2	—

*Sinters were performed in a moist He -2% H_2 atmosphere moisture in exit gas ranged from 2400 to 3700 ppm of water

ic method. The principal objective is to evaluate four types of film based on resolution, reliability, and ease of operation. The effect of Mylar attenuation on film exposure time will be determined.

The standard UNC film pack method was used in which the film rests on a Neoprene pad in a light-tight aluminum holder. The film emulsion faces a window in the holder which is covered with two layers of 0.00025-in. thick aluminized Mylar. Further attenuation of the alpha action particles from the specimen can be obtained, as desired, by increasing the thickness of Mylar or by coating of the sample. A light-tight film pack that is free of contamination is essential, since film exposure is made in a standard hood (controlled area) and film pack loading and development are done in a dark room in an uncontrolled area. The four types of alpha autoradiographic films being evaluated are: (1) Kodak Cellulose Nitrate; (2) Kodak Contrast Ortho N411-006-51; (3) Agfa-Gevaert Scientia 10E56; and (4) Personal Neutron Dosimeter, Type A

The specimen selected for this evaluation was prepared from natural Eldorado UO_2 and oxalate precipitated PuO_2 . The sintered UO_2 -24 w/o PuO_2 pellet contained a desirable distribution of plutonium-containing particles for the purpose of a standard that ranged from less than 20 to 300 microns. The autoradiographic study is nearly complete. The results so far indicate the Kodak Contrast Ortho and Agfa Gevaert films will be more useful than the Personal Neutron Dosimeter Type A and Kodak Cellulose Nitrate.

b. Pu Dispersion by X-ray Diffraction

The feasibility of X-ray diffraction as a method of determining less than 5% of secondary phases (in accordance with the FFTF fuel specification) depends on the method of sample preparation, and on the sensitivity of the instrument to discriminate phases of interest at levels below 5%.

Prior to undertaking an extensive development program, the instrument sensitivity will be evaluated. An adequate number of samples of a solid solution matrix phase with from 1 to 5% of UO_2 and PuO_2 second phase will be prepared and analyzed to obtain statistical data on instrument sensitivity. A detailed work program will be initiated if the results are satisfactory.

A solid solution matrix of $(\text{UPu})\text{O}_2$ was synthesized from previously sintered UO_2 -24 w/o PuO_2 pellets. X-ray diffraction analysis of the pellets indicated a complete single-phase solid solution; secondary phases were not present. The autoradiographic prints appeared uniform in structure and color with no evidence of discrete plutonium particles.

A 95% $(\text{UPu})\text{O}_2$ + 5% UO_2 is being prepared for the X-ray diffraction analysis study.

2. Sintered Pellet Analytical Characterization

a. Plutonium Variability

The variability of plutonium content in sintered UO_2 -24 w/o PuO_2 pellets was determined. The fuel was prepared from oxalate precipitated PuO_2 and Eldorado UO_2 by the standard process of blending, milling, agglomeration, drying, and granulation. Compacts were sintered in flowing $\text{He} + 2\% \text{H}_2 + \text{H}_2\text{O}$ at 1600°C -4hr.

Two types of samples were analyzed: (1) samples taken from whole pellets that were crushed and 0.5-g samples removed; and (2) random pellet samples taken as single chips containing less than 0.2 g. The analysis indicated that the material was within specifications at the 95% confidence level. The results are shown in Table 12.

TABLE 12 — PLUTONIUM ANALYSIS OF SINTERED
 UO_2 -24 w/o PuO_2

Pellet No.	Plutonium, w/o		
	0.5-g Sample from Crushed		
	Whole Pellets	0.2-g Chips	0.2-g Chips
1	21.32	21.25	20.96
2	21.20	21.07	21.04
3	21.33	21.15	21.52
4	—	—	21.17
5	—	—	21.47
$\bar{X} \pm 2s$	21.28 ± 0.16	21.12 ± 0.10	21.23 ± 0.48
Intended value	21.23 ± 0.53	21.23 ± 0.53	21.23 ± 0.53

b. Uranium, Plutonium Analysis

A fusion technique for rapid dissolution of mixed oxides was evaluated. Preliminary data indicate that a pyrosulfate fusion followed by dissolution and titration gave uranium and plutonium values comparable to the nitric-hydrofluoric solvent system. A comparison of the standard nitric-hydrofluoric and fusion assay is given in Table 13.

c. X-Ray Fluorescence for U/Pu Ratio

The evaluation of a rapid method to determine U/Pu ratios by X-ray fluorescence analysis continued. The eutectic pyrosulfate dissolution technique was used for sample preparation. This method provides a clear solid solution of salt and ions indicating complete dissolution. A comparison of the wet chemical and X-ray fluorescence methods is given in Table 14.

3. Powder Characterization

A Perkin-Elmer Shell Sorptometer is being set up to measure surface area of plutonium-containing powders. A standard instrument will be adapted to a glove box by placing the sample tube inside the box and keeping the rest of the unit outside. Adaptations will require filters to prevent contamination outside the glove box and an unusually long length of connecting tubing for apparatus connections. Tests to determine the effect of using the intended filters prototypic sample holder and required connecting lines for glove-box setup were completed.

Design of the modified sample holder to be used for glove-box installation is complete.

C. Pellet Loading Methods

The small fuel-clad gap in the FFTF fuel pellet-rod design presents a weld zone contamination problem during loading of fuel pellets.

TABLE 13 — COMPARISON OF PLUTONIUM
ASSAY USING HNO_3 -HF AND
FUSION METHODS

Sample No.	Nitric - Hydrofluoric Pu, w/o	Fusion Pu, w/o
1	20.86	20.83
2	20.82	20.91
3	20.86	20.88
4	20.82	20.88
$\bar{X} \pm 2s$	20.84 ± 0.04	20.87 ± 0.08

TABLE 14 — COMPARISON OF U/Pu RATIOS BY X-RAY
FLUORESCENCE AND WET ANALYSIS METHODS

Sample No.	Nominal U/Pu	Wet Analysis, U/Pu	X-Ray Fluorescence, U/Pu
1	3.2049	3.197	3.100
2	3.2049	3.214	3.110
3	3.2049	3.223	3.105
$\bar{X} \pm 2s$	—	3.211 ± 0.032	3.105 ± 0.012

A loading sleeve design was completed for assembly of mixed-oxide type pellet rods. The sleeve consists of a molded plastic barrel with an internal guide sleeve. The guide sleeve is designed to locate on the inside surface of the clad while the barrel engages on the outer clad surface. A central hole in the barrel, having the same inside diameter as the sleeve, will permit pellets to be slid into the clad while preventing contamination of the closure weld area.

A prototypic loading sleeve was made. Evaluation of the loading sleeve is in process.

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