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SPECIAL

CARBIDE FUEL DEVELOPMENT PROGRESS REPORT

Period of
April 1, 1963 to June 30, 1963

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September 30, 1963

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FOREWORD

The Carbide Fuel Development project is sponsored by the USAEC Division of Reactor Development. The prime contractor is the United Nuclear Corporation, Development Division, and the subcontractor is The Carborundum Company. The United Nuclear Corporation is performing the conceptual design, fuel evaluation, and fuel irradiation. The Carborundum Company is fabricating the fuel. Both companies are operating plutonium handling facilities.

The report summarizes progress from April 1, 1963 to June 30, 1963.

Previous progress was reported in:

NDA 2140-2, Carbide Fuel Development – Phase I Report (Oct. 15, 1959)
NDA 2145-1, Carbide Fuel Development – Progress Report (Mar. 11, 1960)
NDA 2145-4, Carbide Fuel Development – Progress Report (June 13, 1960)
NDA 2145-5, Carbide Fuel Development – Progress Report (Aug. 30, 1960)
NDA 2145-6, Carbide Fuel Development – Phase II Report (Nov. 6, 1960)
NDA 2162-1, Carbide Fuel Development – Progress Report (Feb. 28, 1961)
NDA 2162-3, Carbide Fuel Development – Progress Report (June 1, 1961)
NDA 2162-5, Carbide Fuel Development – Phase III Report (Sept. 30, 1961)
UNC-5003, Carbide Fuel Development – Progress Report (Feb. 9, 1962)
UNC-5013, Carbide Fuel Development – Progress Report (May 28, 1962)
UNC-5030, Carbide Fuel Development – Progress Report (Aug. 10, 1962)
UNC-5055, Carbide Fuel Development – Phase IV Report (Mar. 31, 1963)
UNC-5056, Carbide Fuel Development – Progress Report (May 1, 1963)

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1. INTRODUCTION

The Carbide Fuel Development Program is concerned with the technology of the entire UC-PuC fuel cycle. The major goal of the program is to produce (UPu)C and to obtain data on its irradiation behavior for long burnups and at high power generation rates. In addition, other areas of the fuel cycle are being explored to discover possible problems.

Fuel made of a combination of UC and PuC has a potential of reducing the fuel cycle cost of existing fast breeder reactors. This is because of the increased burnup and increased power generation capability of (UPu)C compared to presently available metallic fuels. The effect of high burnup and high power generation rate on the stability of (UPu)C is not known. However, irradiation tests to 5×10^{20} fissions/cm^3 show that (UPu)C is dimensionally more stable than metallic fuel. Because of the high melting point and good thermal conductivity of UC and (UPu)C, there is further expectation that carbide fuels will be capable of high power generation rates. The program was initiated in May 1959 and as originally outlined covers a period of about four and one-half years. Program objectives are outlined below.

Conceptual Design

1. An analytical study of the effect of substitution of (UPu)C on heat transfer, physics, and cost of existing fast breeder reactors.

2. Conceptual design of rod-type, fuel element configurations which can be substituted directly in existing reactors.

The above was completed and reported in NDA 2140-2.

Facility Design and Fabrication

1. Design and construction of a facility for carbide fabrication at The Carborundum Company.
2. Design and construction of a facility for carbide evaluation at United Nuclear.

The above was completed and reported in NDA 2145-6.

Fuel Fabrication and Evaluation

1. Explore various methods for preparation of fuel from powders and fabrication of the fuel into cylindrical pellets. High density (up to 13.2 g/cm^3) UC and (UPu)C pellets have been made. The preparation of sesquicarbides and carbon stabilized monoxides is being studied.
2. Evaluate pellets by density measurement, chemical analysis, x-ray diffraction, metallography; perform fuel-cladding compatibility studies; measure physical constants. This is complete for UC and in process for (UPu)C. The (UPu)C melting point is being measured and the coefficient of expansion measurements are complete. The majority of the compatibility tests is complete.

Fuel Irradiation

1. Irradiate clad fuel samples with burnup and maximum fuel temperature as the major variables.
2. A minimum 2% burnup of all fuel atoms by fission and a minimum 650°C (1200°F) central fuel temperature were selected to establish the economic advantages of the fuel.

The UC samples have been irradiated and examined (NDA 2162-5, UNC-5055). Eight (UPu)C samples are being irradiated, and the irradiation of four samples has been completed. Preparations for their post-irradiation examination are in progress.

2. SUMMARY

2.1 FUEL FABRICATION AND EVALUATION

Experiments were performed to synthesize carbon stabilized (UPu)O. The products were mixtures of dioxide- and monoxide-type phases, and contained about 1 w/o carbon.

Measurements of lattice parameters vs time indicated a change with $(U_{0.8}Pu_{0.2})C_{0.95}$ which was not evident with $(U_{0.95}Pu_{0.05})C_{0.98}$.

Microprobe analyses of $(U_{0.8}Pu_{0.2})C_{0.95}$ + 0.1% Ni concluded that the second-phase sesquicarbide had a higher Pu/U ratio than the monocarbide matrix.

Compatibility tests between $(U_{0.95}Pu_{0.05})C_{0.98}$ and type 316 stainless steel, niobium and niobium-1% zirconium alloy for 1000 hr at 816°C showed no reaction. Zircaloy-2 is believed to have reacted with the fuel at a temperature as low as 593°C, after 1000 hr.

Examination of coefficient of expansion specimens after testing showed no change in chemical analysis or microstructure.

2.2 IRRADIATION TESTS

All 12, scheduled, $(U_{0.8}Pu_{0.2})C_{0.95}$ specimens operated in-pile, close to design conditions. Four specimens reached their target burnups of 4.9×10^{20} fissions/cm^3, and were removed from the reactor for post-irradiation examination.

Fabrication and setup of equipment for post-irradiation examination was nearly completed.

3. FUEL FABRICATION AND EVALUATION

3.1 SYNTHESIS OF CARBON STABILILIZED $(U_{0.8}Pu_{0.2})O$

Several experiments were performed in an attempt to synthesize $(U_{0.8}Pu_{0.2})O$. The existence and stability of the pure, mixed monoxide phase is unknown. However, the existence of a carbon stabilized (UPu)O is very likely. The identification of (UPu)(OC) type compounds is important, since there are significant quantities of oxygen present in the (UPu)C produced by the oxide-carbon reaction. In addition, such a compound could have desirable engineering properties: the oxidation resistance of an oxide, and the thermal conductivity of a monocarbide.

For reaction purposes, PuO_2, UO_2, and C in stoichiometric proportions to give $(U_{0.8}Pu_{0.2})O$ were intimately blended and pressed, without binder, into 0.2-in. diameter pellets. The forming pressure was 30,000 psi.

For synthesis, the temperatures investigated were 1650°C, 1750°C, 1850°C, 1950°C, and 2000°C. The results of this study are shown in Table 1.

Metallographic examination of the product from Experiment 8 showed a two-phase structure, a gray dioxide-type phase, and a light monoxide-type phase, each about 50% in volume. (See Fig. 1.)

The method of synthesis produces a product of about 1 w/o carbon containing a mixture of dioxide- and monoxide-type phases. The carbon distribution between the two phases is unknown at this time.

Table 1 — Results of Experiments to Synthesize $(U_{0.8}Pu_{0.2})O$

Experiment No.	Reaction Temp, °C and Hold Time	Batch Size, g	Total Carbon, %	X-Ray Analysis (Diffractometer)
1	1650 – 5 hr	5	1.09	Major 2-phase $(UPu)O_2$ type $a_0 = 5.482 \pm 0.002$ Å $a_0 = 5.4732 \pm 0.0009$ Å Moderate/Strong (UPu)O or (UPu)C type $a_0 = 4.9565 \pm 0.0002$ Å Indication of UC_2
2	1750 – 6 hr	5	0.96	Major, 3 distinct $(UPu)O_2$ type phases $a_0 = 5.4938 \pm 0.0007$ Å $a_0 = 5.4841 \pm 0.0009$ Å $a_0 = 5.475 \pm 0.001$ Å Moderate (UPu)O or (UPu)C type $a_0 = 4.9540 \pm 0.0007$ Å
3	1850 – 3½ hr	5	0.98	Major, 2 distinct $(UPu)O_2$ type phases $a_0 = 5.4959 \pm 0.0009$ Å $a_0 = 5.4864 \pm 0.0003$ Å Weak (UPu)O or (UPu)C type $a_0 = 4.945 \pm 0.003$ Å
4	Product from Exp. 3 crushed, pressed, and reheated to 2000 – 2 hr		0.65	Major, several $(UPu)O_2$ type phases Avg. $a_0 = 5.473$ Å Moderate (UPu)O or (UPu)C type $a_0 = 4.9453 \pm 0.0008$ Å Indication of UC_2
5	1850 – 3 hr	30	1.11	Not determined
6	Product from Exp. 5 crushed, pressed, and reheated to 1850 – 1 hr		1.00	Not determined
7	Product from Exp. 6 crushed, pressed, and reheated to 1850 – 2 hr		1.07	Major $(UPu)O_2$ type $a_0 = 5.4908 \pm 0.0005$ Å Moderate (UPu)O or (UPu)C type $a_0 = 4.9456 \pm 0.0006$ Å
8	Product from Exp. 7 crushed, pressed, and reheated to 2000 – 1 hr		0.90	Major, several $(UPu)O_2$ type phases Avg. $a_0 = 5.4929 \pm 0.0006$ Å Moderate/Strong, several (UPu)O or (UPu)C type phases* Avg. $a_0 = 4.9429 \pm 0.0007$ Å
9	1950 – 3 hr	5	0.75	Not determined
10	Pellets from Exp. 9 reheated to 2000 – 2 hr		0.82	Not determined
11	2000	5.	0.98	Major, several $(UPu)O_2$ type phases Avg. $a_0 = 5.486$ to 5.494 Å Moderate/Strong, several (UPu)O or (UPu)C type phases Avg. $a_0 = 4.943$ to 4.956 Å

*Metallography and etchants identified the face-centered cubic phase as monoxide rather than monocarbide.

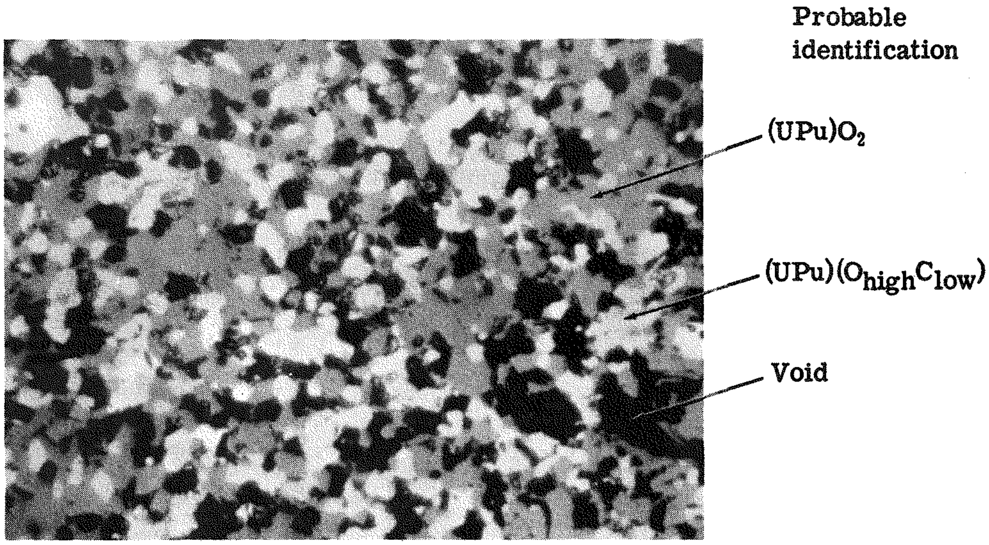


Fig. 1 — Reaction product from (UPu)O synthesis. 600×. Unetched. (Experiment 8, Table 1)

3.2 EFFECT OF AGING ON THE LATTICE PARAMETER OF (UPu)C

X-ray analyses by the Debye-Sherrer camera method for phase and unit cell determinations continued, to determine the effect of aging on the lattice parameter of the $(U_{0.95}Pu_{0.05})C_{0.98}$ specimen prepared in December 1962. The results of the analyses collected are shown in Table 2. A definite trend was not noted during the first four months of analyses, and the measurement was discontinued.

Table 2 — Effect of Aging on Lattice Parameter of
$(U_{0.95}Pu_{0.05})C_{0.98}$ Powder (Camera Method)

Date	Phase Present	Unit Cell Size
12/15/62	Single-phase (UPu)C	$a_0 = 4.9607 \pm 0.0003$ Å
1/15/63	Single-phase (UPu)C	$a_0 = 4.9603 \pm 0.0004$ Å
2/15/63	Single-phase (UPu)C	$a_0 = 4.9612 \pm 0.0005$ Å
3/15/63	Single-phase (UPu)C	$a_0 = 4.9613 \pm 0.0009$ Å
4/15/63	Single-phase (UPu)C	$a_0 = 4.960 \pm 0.001$ Å

During May, a specimen containing a higher Pu to U ratio, $(U_{0.8}Pu_{0.02})C_{0.95}$, was prepared for x-ray analyses by the diffractometer method. Self-damage due to alpha radiation should be more pronounced with the higher plutonium composition.

The initial results are shown in Table 3.

This experiment will continue for several months to confirm the trend indicated from the data.

3.3 MICROPROBE ANALYSES

The microprobe analyses of $(U_{0.8}Pu_{0.2})C_{0.95}$ and $(U_{0.8}Pu_{0.2})C_{0.95}$ + 0.1% Ni were completed by the Monsanto Research Corporation's Mound Laboratories. The sesquicarbide phase has a higher Pu/U ratio than the monocarbide phase as reported previously. Assuming the monocarbide phase has a Pu/U ratio of 2/8, the

Table 3 — Effect of Aging on the Lattice Parameter of $(U_{0.8}Pu_{0.2})C_{0.95}$ Powder (Diffractometer Method)

Date	Phases Present	Unit Cell Size
4/15/63	Major (UPu)C Weak $(UPu)C_2$ Faint $(UPu)_2C_3$ Indication of $(UPu)O_2$	$a_0 = 4.9648 \pm 0.0005$ Å
5/15/63	Major (UPu)C Faint $(UPu)_2C_3$ and $(UPu)C_2$	$a_0 = 4.9661 \pm 0.0003$ Å
6/23/63	Major (UPu)C Faint $(UPu)_2C_3$ $(UPu)C_2$, and $(UPu)O_2$	$a_0 = 4.9667 \pm 0.0007$ Å

sesquicarbide phase would have a Pu/U ratio of 3.63/6.37. Absolute plutonium and uranium analyses were hindered by surface film formations on the highly oxidizable carbides.

3.4 FUEL-CLAD COMPATIBILITY TESTS

All tests were made as described on pp. 23-25 of the previous progress report, UNC-5056. The results are summarized below.

3.4.1 593°C (1100°F), 1000-hr Tests

The examination of the only test, that between $(U_{0.95}Pu_{0.05})C_{0.98}$ and Zircaloy-2, was completed.

Metallographic examination showed a second phase distributed homogeneously through the thickness of the Zircaloy-2 disc (similar to Fig. 4). The second phase appeared at the grain boundaries most of the time. It has not been identified, but it is believed to be a result of reaction between (UPu) and Zircaloy-2.

3.4.2 816°C (1500°F), 1000-hr Tests

The examination of the tests was completed. The results are given in Table 4. Representative photomicrographs are shown in Figs. 2 through 4.

3.4.3 593°C, 4000-hr Tests

The tests are in progress, and logged 2050 hr as of 7/30/63.

3.4.4 816°C, 4000-hr tests

The tests were completed in early June, and examination started. None of the fuel and clad samples were bonded to each other.

$(U_{0.95}Pu_{0.05})C_{0.98}$ and $(U_{0.95}Pu_{0.05})C_{0.98}$ + 0.1% Ni were each tested against type 316 stainless steel, niobium, and niobium-1% zirconium alloy. In addition, $(U_{0.95}Pu_{0.05})C_{0.98}$ was tested against 2¼% Cr-1% Mo steel. The metallographic examination of all the fuel halves of the specimens was completed. Significant microstructural changes were not noted.

3.5 COEFFICIENT OF EXPANSION MEASUREMENTS

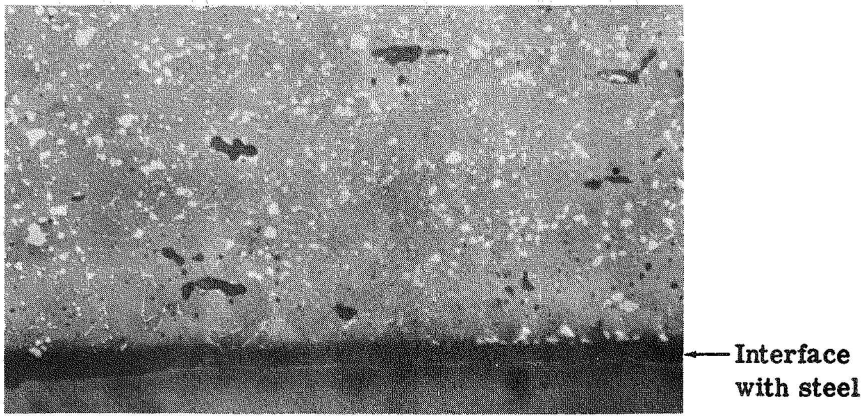
Chemical analyses and metallographic examination of the tested coefficient of expansion bars were completed and compared to the untested bars. Results are given in Table 5. There were no significant changes due to testing. The tests cycled the specimens to 1400°C several times.

Table 4 — Summary of 816°C, 1000-hr Compatibility Tests

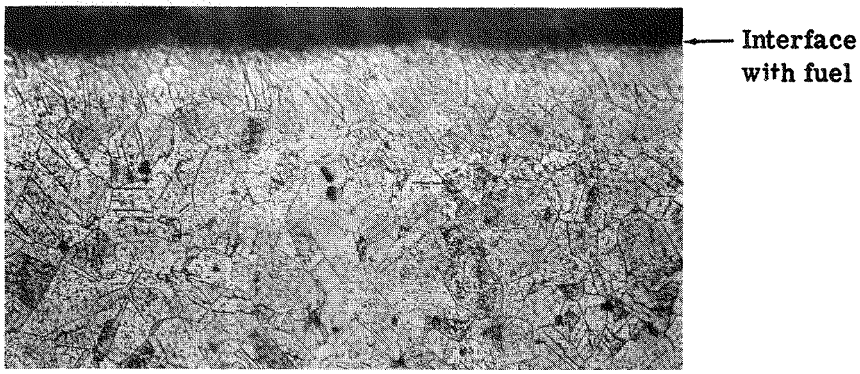
Clad	Fuel	Metallographic Examination Results	Remarks
Type 316	$(U_{0.95}Pu_{0.05})C_{0.98}$	No reaction	Similar to Fig. 2
Type 316	$(U_{0.95}Pu_{0.05})C_{0.98}$	No reaction	Fig. 2
Niobium	$(U_{0.95}Pu_{0.05})C_{0.98}$	No reaction	Similar to Fig. 3
Niobium-1% Zr	$(U_{0.95}Pu_{0.05})C_{0.98}$	No reaction	Fig. 3
Zircaloy-2	$(U_{0.95}Pu_{0.05})C_{0.98}$	Fuel bonded to one Zircaloy disc. Interface crumbled during polishing. A second phase appeared at the grain boundaries as in the 593°C, 1000-hr test, and this is believed to be the product of a reaction.	Fig. 4

Table 5 — Effect of Testing on the Chemical Composition of Thermal Expansion Bars

	w/o C			w/o Pu	
	Before Testing		After Testing	Before Testing	After Testing
	United Nuclear	Carbo-rundum			
$(U_{0.95}Pu_{0.05})C_{0.98}$	4.69	4.67	4.67	4.99	5.01
$(U_{0.95}Pu_{0.05})C_{0.98}$ + 0.1 Ni	4.81	4.77	4.83	5.10	4.95



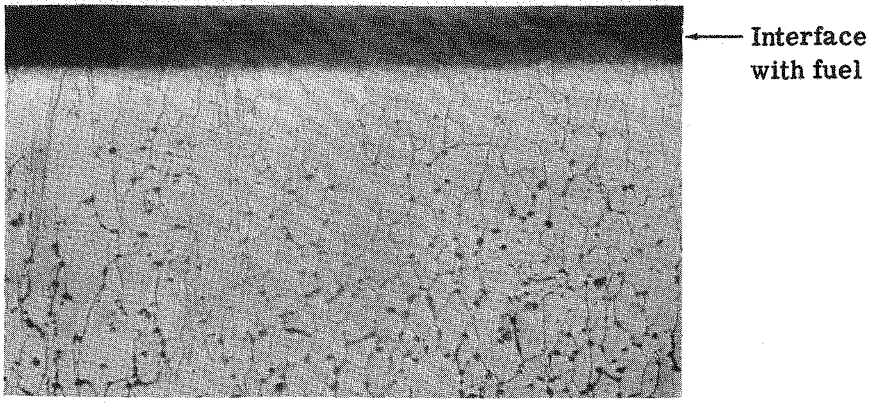
(a) $(U_{0.95}Pu_{0.05})C_{0.98}$ + 0.1% Ni. 150×. Etchant: nitric acid-acetic acid-water. Structure: monocarbide matrix (gray), sesquicarbide second phase (white).



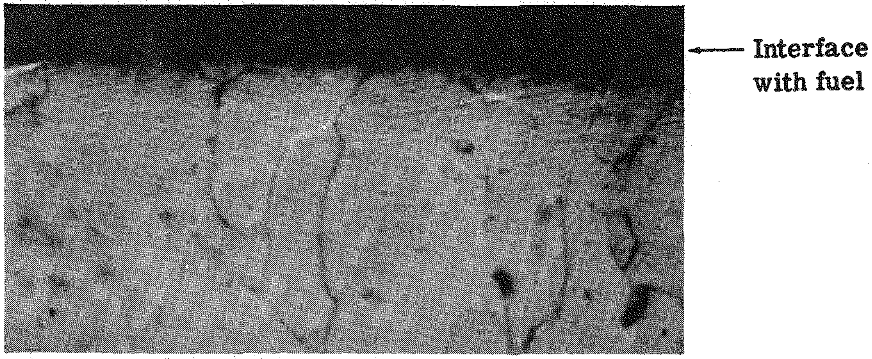
(b) Type 316 stainless steel. 150×

(c) Type 316 stainless steel. 600×. Etchant: Marble's. Structure: austenitic matrix with carbides and sigma. Homogeneous precipitation of carbides and sigma is a thermal effect.

Fig. 2 — $(U_{0.95}Pu_{0.05})C_{0.98}$ + 1% Ni vs type 316 compatibility specimens tested for 1000 hr at 816°C in helium

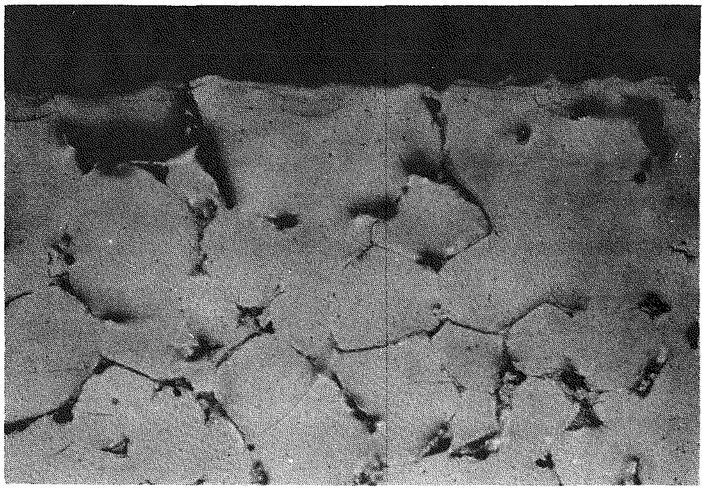


(a) Niobium-1% zirconium. 150×.



(b) Niobium-1% zirconium. 600×. Etchant: lactic-nitric-hydrofluoric acids.

Fig. 3 — $(U_{0.95}Pu_{0.05})C_{0.98}$ vs Nb-1% Zr compatibility specimens tested for 1000 hr at 816°C in helium



Zircaloy-2. 150×. Etchant: hydrofluoric acid-nitric acid-glycerine-water.

Fig. 4 — $(U_{0.95}Pu_{0.05})C_{0.98}$ vs Zircaloy-2 compatibility specimens tested for 1000 hr at 816°C in helium

4. IRRADIATION TESTS

4.1 EXPERIMENT OPERATION

The detailed description of the irradiation tests is in the previous progress report, UNC-5055, Section 4.3. In summary, each of the six irradiation capsules contains two specimens. There is a total of 12 specimens. Each specimen consists of 16 fuel pellets, 0.191-in. diameter, for a 3-in. fueled length, clad in stainless steel or niobium. Both specimens contain $(U_{0.8}Pu_{0.2})C_{0.95}$, one without sintering aid and one with 0.1 w/o nickel sintering aid. The void space is filled with helium. The specimens designed to operate at less than 1040°C (1900°F) central fuel temperatures have a niobium central thermocouple well for temperature measurement, and for this reason are also clad with niobium. The higher temperature specimens do not have provision for fuel center temperature measurement. These specimens are clad with type 316 stainless steel because this is potentially one of the most economical fuel clads for a fast, central station, power plant.

During the reporting period, all 12 specimens operated in-pile, close to design temperatures. Their operating conditions are summarized in Table 6.

Capsule 65, a high power capsule for irradiation in the GETR core, was started up with the April cycle (No. 43). In order to check the heat transfer characteristics of the capsule, initial irradiation was in the pool. The experimentally determined capsule thermal resistances were in good agreement with design values, and the capsule was moved into a core position. The power is still short of the goal of

Table 6 — Summary of Irradiation Specimen Operating Conditions (Startup to 6-30-63)
(Temperatures rounded off to nearest 5)

	Capsule No. and Startup Date					
	61 (11-24-62)	62 (12-19-62)	63 (1-29-63)	64 (1-29-63)	65 (4-6-63)	66 (3-3-63)
Temperature of Fuel Center, °C (°F)						
Upper specimen*						
Maximum	1305 (2380)	1175 (2150)	1230 (2250)	1315 (2400)	1330 (2425)	1330 (2420)
Average	1030 (1890)	1005 (1840)	1005 (1840)	1125 (2060)	1175 (2145)	1155 (2110)
Lower specimen*						
Maximum	1280 (2340)	980 (1800)	1040 (1900)	1415 (2580)	1320 (2410)	1470 (2680)
Average	1015 (1820)	870 (1600)	845 (1550)	1175 (2150)	1150 (2100)	1150 (2100)
Temperature of Clad Surface, °C (°F)						
Upper specimen*						
Maximum	795 (1460)	715 (1320)	675 (1250)	740 (1360)	445 (835)	720 (1325)
Average	625 (1160)	620 (1150)	560 (1040)	655 (1210)	405 (765)	620 (1150)
Lower specimen*						
Maximum	770 (1420)	645 (1190)	545 (1010)	790 (1460)	530 (990)	705 (1300)
Average	615 (1140)	565 (1050)	480 (900)	690 (1270)	415 (795)	590 (1090)
Specimen Heat Generation Rate, w/cm (kw/ft)						
Upper specimen*						
Maximum	450 (13.7)	410 (12.5)	490 (15.0)	470 (14.4)	690 (21.0)	480 (14.5)
Average	355 (10.9)	340 (11.3)	400 (12.1)	380 (11.5)	550 (16.9)	440 (13.5)
Lower specimen*						
Maximum	440 (13.4)	380 (11.6)	460 (13.9)	510 (15.5)	620 (18.9)	600 (18.2)
Average	350 (10.7)	330 (10.1)	370 (11.4)	400 (12.1)	540 (16.4)	490 (14.9)
Cumulative Burnup						
Fissions/cm^3	6.6×10^{20}	6.1×10^{20}	6.0×10^{20}	4.9×10^{20}	3.8×10^{20}	4.5×10^{20}
(MW-d/tonne)	(21,600)	(20,000)	(19,600)	(16,100)	(12,500)	(14,700)
Target Burnup						
Fissions/cm^3	Irradiation completed	10.4×10^{20}	20.8×10^{20}	Irradiation completed	10.4×10^{20}	20.8×10^{20}
(MW-d/tonne)		(34,000)	(68,000)		(34,000)	(68,000)

*Upper specimen contains $(U_{0.8}Pu_{0.2})C_{0.95}$ + 0.1% Ni. Lower specimen contains $(U_{0.8}Pu_{0.2})C_{0.95}$, except in Capsule 64, where they are reversed.

20 kw/ft. The capsule will be moved to a higher flux core position in Cycle 46. The irradiation positions, and corresponding performance of Capsule 65 are summarized in Table 7.

Table 7 — Irradiation Positions and Operating Conditions for Capsule 65

Month	Cycle No.	Position	Avg. Power, w/cm (kw/ft)	Avg. Fuel Center Temp, °C (°F)	Avg. Clad Surface Temp, °C (°F)
April	43	Pool X-1	440 (13.4)	770 (1420)	310 (590)
May	44	Outer core H3	610 (18.6)	1290 (2350)	505 (940)
June	45	Outer core H3	574 (17.5)	1215 (2220)	475 (890)

Table 6 shows the average operating conditions up to the June shutdown.

Capsules 61 and 64 completed their scheduled irradiation exposure in June. They were removed from the reactor and shipped for post-irradiation examination.

4.2 POST-IRRADIATION EXAMINATION

Battelle Memorial Institute was chosen as the subcontractor for the post-irradiation examination.

The first four specimens, contained in Capsules 61 and 64, were shipped to the hot lab in June.

Work at Battelle has been directed toward preparing equipment for the hot lab examination, and checking out experimental techniques which will be used in the analysis of the fuel.

An airtight polyethylene chamber has been constructed and is being installed in a hot cell to provide a protective nitrogen atmosphere for most operations in which the bare fuel will be handled. These operations include removal of specimen cladding, physical measurements of pellets, metallographic preparation, preparation of x-ray specimens, and removal of chemical analysis specimens.

Construction of specialized jigs for specimen clad cutting and slitting were completed.

Metallographic procedures with cold fuel samples were checked in air and nitrogen to see whether an air atmosphere might be satisfactory. Samples stained in air after 1 to 4 hr, and a 24-hr stain could be removed by repolishing.

An electrochemical sampling method for determining radial plutonium distribution in the fuel was tried, but proved to be unsuccessful. It was decided to rely on mechanical sampling techniques.

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