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FINAL REPORT ON THE TESTING OF GRAPHITE-TYPE NUCLEAR ROCKET (ROVER) FUEL IN A PULSED REACTOR (TREAT)

(Title Unclassified)

by

C. R. Simmons, W. R. Yario,
and R. O. Ivins

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C. R. Simmons,* W. R. Yario,*
and R. O. Ivins

Chemical Engineering Division

*On loan from Westinghouse Astronuclear Laboratory

May 1964

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I. INTRODUCTION

The use of a nuclear reactor as an energy source in spacecraft for either propulsion, or as an onboard power supply, presents several unique safety problems. If such a machine must be returned to the earth during its flight, the decision of bringing it back intact or destroying it must be made. If it is desired to destroy the fissionable device, the mechanism used to achieve its destruction must ensure sufficient fragmentation to allow very efficient distribution of the radioactive materials in the atmosphere so as to reduce the level of radioactivity to a negligible biological hazard.

This study was initiated to determine whether or not Kiwi-type fuel could be reduced by a reactor transient to particles small enough to be sufficiently consumed by burnup on re-entry to the earth's atmosphere to satisfy the safety requirements. If a severe reactor transient could reliably reduce the core of a nuclear rocket to very small particles, the safe return of the radioactive core to earth in an aborted mission could be ensured.

To determine the susceptibility of the Kiwi fuel to fragmentation by a nuclear transient, autoclaved samples were subjected to transient pulses of TREAT (Transient Reactor Test Facility, NRTS, Idaho). As the experiments progressed it became apparent that other information, valuable to the design of a nuclear rocket, was obtained. Information such as how rapidly the power level (neutron flux) could be increased without the loss of integrity of the core material was obtained. This is valuable information in considering the reactor startup in the design requirements. In addition, the maximum power level that could be withstood by the core material during transient irradiations yielded information important in the consideration of the steady-state operating level of such a machine.

Motion-picture studies of the material during irradiation, metallographic examination of the materials before and after transient irradiation, fission-product analysis of the gaseous environment of the fuel, and other analyses provided considerable information of general interest in the program.

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This is the final report covering the work done in the Chemical Engineering Division of Argonne National Laboratory by personnel of the Westinghouse Astronuclear Laboratory and Argonne. It covers the experiments with graphite matrix fuel of the Rover (Kiwi) type carried out between July 30, 1962, and September 30, 1963. The work done before that time was published in ANL-6522⁽¹⁾ and ANL-6572⁽²⁾, the first and second status reports on these experiments. These reports covered the testing of the Kiwi fuel with a uranium carbide dispersion in a graphite matrix. This document describes the subsequent testing of the Kiwi fuel with pyrolytic carbon-coated beads of uranium carbide in a graphite matrix.

II. DESCRIPTION OF EXPERIMENTS

A. Fuel Material

The fueled specimens studied contained beads of pyrocarbon-coated, enriched uranium carbide (UC_2), dispersed in a reactor-grade graphite matrix. The coated beads were nominally 150 microns in diameter, with a laminar-type pyrocarbon coating approximately 25 microns thick. The samples tested had either a cylindrical or hexagonal geometric configuration with longitudinal coolant channels (cylindrical holes). The samples were formed by extrusion and graphitized at 1900°C.

In addition, experiments were performed on loose batches of different-sized, pyrocarbon-coated beads. The fuel samples, categorized by geometry, are discussed in the following paragraphs.

1. Cylindrical Stock

The cylindrical test samples were cut from 1/2-inch-diameter extruded rods, which were received from Los Alamos Scientific Laboratory in the as-manufactured state. The rods contained seven unlined longitudinal coolant channels (cylindrical holes) each approximately 90 mils in diameter. The channels were spaced in the rods with about 170 mils between centers as shown in Figure 1. The characteristics of the batches tested are as follows:

<u>Batch No</u>	<u>w/o Uranium</u>	<u>U^{235} Density, mg/cc</u>	<u>Fabrication History</u>
2-2721	20	400	Seven longitudinal holes, drilled in solid extruded rods
2-2771	15 5	358	Extruded as full 19-hole hex, machined to 1/2-inch-diameter rod
2-2820	27 5	633	Extruded as solid 1/2-inch-diameter rod

The microstructure of as-manufactured cylindrical fuel stock is shown in Figure 2.

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Figure 1
TYPICAL CYLINDRICAL AND
HEXAGONAL GRAPHITE SAMPLES
USED IN TREAT EXPERIMENTS

108-6112

Figure 2
MICROSTRUCTURE OF AS-MANUFACTURED, CYLINDRICAL FUEL STOCK, SHOWING
PYROCARBON-COATED FUEL BEADS AND GRAPHITIZED MATRIX

As Polished

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2. Hexagonal Stock

Hexagonal test samples were cut from extruded hexagonal rods nominally $3/4$ inch across the flats. The rods contained 19 longitudinal coolant channels (cylindrical holes, 100 mils in diameter). In all but one of the batches tested, the coolant channels were lined with vapor-deposited niobium carbide (NbC). The linings varied from 1 to 2.5 mils in thickness. A photograph of a typical NbC-lined hexagonal sample is shown in Figure 1. The typical microstructure of as-manufactured hexagonal fuel stock, showing the character of the NbC lining, is shown in Figure 3. Fuel was leached from the surfaces of the coolant channels by chlorine treatment before the deposition of NbC.

Figure 3
MICROSTRUCTURE OF
NbC-LINED,
HEXAGONAL FUEL STOCK

150X

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Some of the hexagonal fuel tested had been heated and reacted with water vapor to convert part of the UC_2 to UO_2 . This material is referred to as back-reacted fuel. The back-reacted fuel stock showed varying degrees of fuel pellet break-up and fuel migration. The microstructure of a sample containing 22% of the uranium as UO_2 is shown in Figure 4.

Figure 4

MICROSTRUCTURE OF 22% BACK-REACTED, HEXAGONAL FUEL STOCK SHOWING PELLET BREAKUP, FUEL MIGRATION, AND LINING

As Polished

100X

The hexagonal fuel material tested is as follows:

Batch No.	w/o Uranium	U^{235} Density, mg/cc	Bulk Density, g/cc	State
2-2765	15.5	358	2.31	As-manufactured, lined
2-2802	15.9	386	2.41	As-manufactured, lined
2-2806	15.9	386	2.41	As-manufactured, lined
2-2764	15.5	358	2.31	Back-reacted, 3%, lined
2-2767	16.6	381	2.46	Back-reacted, 22%, lined
2-2800	19.8	448	2.24	As-manufactured, unlined

3. Bead Material

The fuel beads were pellets of pyrocarbon-coated UC_2 . Five batches of these pellets were tested, each batch having a different average pellet diameter and uranium loading as follows:

<u>Batch No.</u>	<u>Screen Fraction</u>	<u>Average Pellet Diameter</u>	<u>w/o Uranium</u>
B6945	-210 to +177	193 μ	76.29
B6949	-177 to +149	163 μ	71.41
B6952	-149 to +125	137 μ	69.31
B6955	-125 to +105	115 μ	68.53
B6958	-105 to + 88	96 μ	64.57

Representative photomicrographs of the largest- and smallest-diameter pellets are shown in Figures 5 and 6. The pyrocarbon coatings averaged about 25 microns in thickness. The uranium fuel used in all batches was approximately 93 w/o enriched.

Figure 5

PYROCARBON-COATED UC_2 FUEL BEADS;
96-micron AVERAGE BEAD DIAMETER

As Polished

250X

Figure 6
PYROCARBON-COATED UC₂ FUEL BEADS;
193-micron AVERAGE BEAD DIAMETER

As Polished

250X

B. TREAT Facility

The experiments were carried out in Argonne National Laboratory's TREAT reactor at the National Reactor Testing Station in Idaho. TREAT is a homogeneous, graphite-moderated and graphite-reflected, transient nuclear reactor. Further details of design and operation of the reactor are contained in various reports on TREAT.⁽³⁻⁷⁾

An isometric drawing of the reactor is shown in Figure 7. The core is composed of graphite-urania matrix fuel elements encased in Zircaloy cans. The nominal size of the core is 5 ft x 5 ft x 4 ft. The reactor core is capable of sustaining transient thermal-flux integrals of approximately 3.5×10^{15} nvt. The maximum energy release of the reactor is 1000 MW-sec, with a period of 40 msec. The overall dimensions of each fuel element assembly, which includes reflector graphite at the top and bottom, are 4 in. x 4 in. x 8 ft. The reactor fuel has a carbon:uranium-235 atomic ratio

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of 10,000:1. The large amount of carbon in the reactor core serves three purposes. First, it acts as a moderator (thermalizes the neutrons). Second, it forms a large heat sink which absorbs fission energy from the dispersed urania particles. The total heat capacity is such that this reactor can undergo pulses of total energy release far greater than those sufficient to destroy or cause meltdown in most other reactor cores. Third, the reactor fuel has a negative temperature coefficient of reactivity, which is sufficient to make an excursion self-limiting.

Figure 7

PERSPECTIVE VIEW OF TREAT

112-552

The reactor is initially set at a nominal steady-state power level of 10 W. After the transient is initiated, the total energy of the pulse (integrated power) is temperature-limited, as described above, or the pulse can be clipped by rapidly inserting control rods which contain boron carbide poison. The period of a particular pulse can be controlled by the addition of the proper excess reactivity. Thus, control can be exercised over both

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the rate of energy input (period) and the total energy input (integrated power). However, the peak power depends on the period and limits the selection of period and total energy.

C. Experimental Assemblies and Techniques

Several types of experimental assemblies were used to conduct the transient irradiations in TREAT. Generally, the assemblies can be classified into three categories: opaque autoclaves, transparent capsules, and special-purpose assemblies. The experimental assemblies are described using this classification.

1. Opaque Autoclaves (Experiments CEN-104 to -108, CEN-126 to -129)

The autoclaves used in these experiments were made of stainless steel and designed for a working pressure of 4000 psi at 300°F. These autoclaves have been described in detail in the earlier status reports.^(1,2) For testing, the autoclave assemblies were placed in dummy elements, which were then inserted into the center of the TREAT reactor.

Two inner graphite crucible arrangements were used to support cylindrical fuel specimens during autoclave testing. Figure 8 shows cutaway drawings of the two crucible arrangements. Some of the specimens were placed in the graphite crucibles without constraint; others were wrapped in graphite cloth and mounted in a graphite annulus so as to be constrained laterally with an estimated pressure of 100 psi.

The specimens tested in this type of experiment were 3/4 in. long, 1/2 in. in diameter, and contained seven unlined coolant channels. The specimens in CEN-104 and -108 were instrumented with a W-5% rhenium versus W-26% rhenium sheathed thermocouple, which was positioned in the center coolant channel. These thermocouples were 1/16 in. in diameter with a tantalum sheath and were insulated with beryllia.

All the experiments in this series, except CEN-107, used a 20-psia helium environment; CEN-107 was conducted in vacuum.

2. Transparent Capsules (Experiments CEN T-3 to T-6, T-8, T-9, and T-10 through T-18)

The transparent capsules used in these studies were described in detail in the second status report.⁽²⁾ Each capsule consisted of a Zircaloy rectangular capsule with a high-purity, fused-silica window in one end to permit high-speed photography of the fuel specimen during irradiation. A photograph of an unassembled transparent capsule is shown in Figure 9. Motion-picture records were obtained using a high-speed Wollensak Fastax,

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Figure 8

OPAQUE AUTOCLAVE ASSEMBLIES FOR TESTING GRAPHITE FUEL IN TREAT

INTERNAL ASSEMBLIES OF GRAPHITE CRUCIBLES
FOR NON-CONSTRAINED TESTS

VIEW OF COMPONENTS

INTERNAL ASSEMBLIES OF GRAPHITE CRUCIBLES
FOR PROVIDING FUEL CONSTRAINT ~~CONFIDENTIAL~~

Figure 9
TRANSPARENT CAPSULE

WF-14 camera (with 16-mm film) which had a maximum speed of 4000 frames per second. The lens was a 6-in. (152-mm), f/4.5 Raptar Telephoto. The film was Kodak Ektachrome commercial color safety film, type 7255.

Both hexagonal and cylindrical specimens were tested in the transparent assemblies. Generally, one specimen was irradiated at a time, but in two experiments (CEN T-5 and T-6) the capsules were modified to hold two cylindrical specimens. Most of the specimens were pin-supported, as shown in Figure 10, but the samples in two tests (CEN T-4 and T-6) were constrained in a graphite annulus.

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Figure 10

HEXAGONAL SAMPLE HOLDER FOR TRANSPARENT CAPSULE EXPERIMENTS

A. Assembled

B. Components

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The specimens in four experiments (CEN T-8, T-9, T-10, and T-17) were not instrumented. In the rest of the transparent tests, sample temperatures were measured by one or two tungsten/rhenium thermocouples of various types. The types of thermocouples used and their placement in the samples were as follows:

Test No.	Thermocouples per Specimen	Location	Type
CEN T-3, -4, -5, -6	1	Center	Tantalum sheathed
CEN T-11, -12, -13, -14, -18	1	Center	Bare junction (see Figure 10)
CEN T-15, -16	2	Center, apex	Sheathed (see Figure 11)

Sample surface temperatures were measured in experiments CEN T-11 through T-18 by a fast-response optical pyrometer. During testing, a flat of the hexagonal sample was viewed by the pyrometer through an optical path provided by a system of mirrors. This optical path was also used for high-speed photography. A Milletron color-ratio optical pyrometer was used which had a response time of 11 msec at temperatures above 1600°C.

In experiments CEN T-3 to T-6, the capsules were supplied with a helium flow of 0.5 liter/min at a small positive pressure. The helium entered at the rear of the capsule above the specimen and exited at the front of the capsule at the bottom of the viewing window. Under these conditions of flow, vaporized carbon, which formed in tests at moderate and high energies, condensed on the inner capsule window and filled the interior with a heavy black smoke during the latter part of each transient. These effects obscured the specimen during a good portion of the photographic record because of the condensation of carbon on the viewing window. Therefore, in later experiments, capsules CEN T-8 and T-9 were modified to direct the inlet helium flow directly on the window just above the camera viewing area. This modification, along with increasing the helium flow to 4.5 liters/min, essentially eliminated the deposition of material on the window. However, the clouds of carbon still obscured the photographic view.

In an attempt to eliminate the clouding, capsules CEN T-10, -11, -12, -13, and -14 were fitted with a steel-wool filter, as shown in Figure 12. It was believed that a large portion of the vapor rising from the specimen would strike the steel wool and condense. The motion-picture records from these experiments showed that the cloud of carbon was not rising far enough to reach the filter. In the last photographic experiments run (CEN T-15 through T-18), a sight tube of Zircaloy was installed in the capsule to prevent the blockage of the camera's view of the sample. A helium flow was directed over the sample to carry away the vaporized material. A photograph of this arrangement is shown in Figure 13.

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Figure 11

HEXAGONAL SAMPLE WITH TWO SHEATHED THERMOCOUPLES FOR TRANSPARENT EXPERIMENTS

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Figure 12

SCHEMATIC DIAGRAM OF MODIFIED TRANSPARENT CAPSULE

108-6222

Figure 13

SIGHT TUBE ASSEMBLY

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This arrangement improved the photographic records of all but the highest-energy experiment (CEN T-17).

3. Special-purpose Assemblies

The assemblies included in this group were designed to obtain unique or specific information. They are discussed in the following paragraphs according to the purpose of the individual experiment.

a. Relative Reactivity of Loose Fuel Particles (Test CEN T-7).

The objective of this experiment was to determine the kinetics of particle ejection and the character of damage induced in fuel beads, as a function of bead diameter and transient energy. The experiment was performed in a transparent capsule. A special Zircaloy sample holder was designed with five graphite trays horizontally arrayed 1.5 in. apart. Five different particle-size fractions of pyrocarbon-coated uranium dicarbide fuel beads were placed loosely in the trays. The beads have been described in detail in Section II-A-3 of this report (page 18). A motion-picture record was made to record the behavior of the beads during three transients of increasing energy. The helium flow and camera arrangements were identical with those used for experiments CEN T-3 through T-9. No attempt was made to measure sample temperatures.

b. Fission Product Release Experiments (CEN-132, -146, -153, -154, -155, -156, -160, -161, and -162. The objective of these tests was to characterize fission-product release (notably iodine and tellurium) from beaded-type graphite fuel in various test environments and at various transient energy levels. The experiments were performed in the opaque autoclaves described previously. With the exception of tests CEN-160, -161, and -162, all the experiments were conducted on hexagonally shaped, NbC-lined specimens. Preirradiated, fueled, graphite, 1/4-in.-OD by 1/8-in.-long cylinders were tested in CEN-160, -161, and -162.

Two general types of inner hardware were used to support the specimens. The first type (used in CEN-132, -146, and -156) is shown in Figures 14 and 15. In tests with this type of hardware, the entire autoclave interior and hardware were leached after irradiation to recover released nuclides for radiochemical analysis.

The second type of inner hardware used in the remaining tests in this series included an inner stainless steel capsule, as shown schematically in Figure 16. The capsule was constructed of 1/16-in.-thick Type 304 stainless steel and was sealed on the ends by butt-welded plates. The inner capsules were evacuated through a manually-operated valve at the top before being tested. Only the inner capsules and accompanying hardware were leached chemically after being tested.

Figure 14
EXPLODED VIEW OF INNER
HARDWARE USED IN
FISSION-PRODUCT TESTS

108-6139

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Figure 15
ASSEMBLED HARDWARE USED IN
FISSION-PRODUCT TESTS

108-6135

Figure 16

SCHEMATIC OF INNER HARDWARE
AND INNER STAINLESS STEEL
CAPSULES FOR FISSION-PRODUCT
TESTS CEN-153, -154, -155,
-160, -161, AND -162

The fission-product tests were performed in various environments and with both as-manufactured and back-reacted fuel specimens. They were conducted in the following environments:

Test No.	Environment	Sample History and Shape
CEN-132	20-psia helium	As-manufactured, hexagonal
CEN-146	14.6-psia air	22% back-reacted, hexagonal
CEN-156	Distilled water	22% back-reacted, hexagonal
CEN-153, -154, -155	Vacuum	As-manufactured, hexagonal
CEN-160, -161, -162	Vacuum	As-manufactured, pre-irradiated, small cylinder

In three of the fission-product tests (CEN-132, -146, and -156), an effluent system was specially designed to obtain gas samples remotely from the autoclave at various times after being irradiated. The system was used also to introduce water to the sample in CEN-156. Stainless steel bulbs were connected to the autoclave through Hoke two-way solenoid valves and 1/8-in. stainless tubing, as shown in Figure 17. In tests CEN-132 and -146, the two metal bulbs were evacuated before the transient, and the autoclaves and the effluent system up to the solenoids were pressurized with

Figure 17
EFFLUENT SYSTEM USED IN
FISSION-PRODUCT AND
WATER-IMMERSION
EXPERIMENTS

108-6168

the appropriate gas environment. A gas sample was taken in each bulb at about 10 sec and about 30 min after the transient was initiated. In test CEN-157, the autoclave and one bulb were evacuated while the other bulb was filled with 60 ml of water under a 70-psia helium pressure. The water was injected into the autoclave 20 sec before the transient. The second bulb was opened to collect a gas sample from the autoclave at 10 sec after the transient.

None of the fission-product release experiments described above was instrumented. However, a temperature-calibration test, CEN-152, was conducted in which a hexagonal sample about .03 in. long was tested in helium at four transient energy levels in an inner capsule hardware arrangement similar to that shown in Figure 16. In the test, the sample was instrumented with a tantalum-sheathed tungsten-rhenium thermocouple, and the inner capsule was not sealed.

c. Water Immersion, CEN-131, -147. Two specimens of lined, hexagon-shaped fuel were irradiated while immersed in sea water. One of the specimens was as-manufactured (CEN-131); the other was 22% back-reacted (CEN-147). The objective of these tests was to determine the composition of the reaction product-gases and the fragmentability of the fuel under these conditions. The autoclave, inner hardware, and effluent arrangement were similar to those used in CEN-156 (see Figures 14 and 15). Two stainless steel bulbs of 150-ml capacity were connected to the autoclave via remotely-operated solenoid valves. One bulb was filled with 70 cc of sea water and pressurized to 70 psia with helium. The autoclave and the second bulb were evacuated to 2 microns mercury pressure. A pressure transducer was attached at the end of a 3-ft tube leading from the bottom of the autoclave. The fuel samples in both tests were not instrumented with thermocouples.

Twenty sec before the transient, water was injected around the specimen, producing an initial pressure of 20 psia in the autoclave. The second bulb was opened remotely 5 min after the transient and held open for 25 sec to take a representative sample of the gaseous reaction products.

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d. Long-channel Experiments, CEN-133 and -139. The objective of these experiments was to determine if the material that vaporizes from a fueled specimen at high temperature will redeposit in adjacent coolant channels that are at or near ambient temperature. The specimens used in this study were hexagonal, NbC-lined, and 1-1/2 in. long. The fueled specimen was placed between two sections of 19-hole, unfueled, reactor-grade graphite, and in this arrangement, the cooler surfaces were provided by the coolant channels in the unfueled pieces. A schematic sketch of the specimen and hardware arrangement is shown in Figure 18. Since the specimens were considerably larger than the others tested, a somewhat elongated opaque autoclave with 300 cc free volume was designed. The long-channel autoclaves were fabricated from 1/2-in. thick stainless steel, providing a rating of 8200 psi at 800°F. The inner diameter of the autoclave was 1-1/2 in. which allowed only 5/16 in. clearance between the specimen and the autoclave wall. As shown in Figure 18, the fueled specimen was separated from the autoclave by a cylinder of pyrolytic graphite 200 mils thick. The layers of pyrolytic graphite in the cylinder were aligned so that the thermal conductivity through the wall of the cylinder was only about 1/200 the conductivity in a direction parallel to the major cylinder axis. Therefore, the heat from the specimen was conducted away from the specimen along the cylinder axis to the relatively large heat sinks on the top and

Figure 18

SCHEMATIC OF
LONG-CHANNEL SPECIMEN
ARRANGEMENT

108-5832

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bottom of the specimen without raising the autoclave wall temperature appreciably. A pressure transducer was attached to the bottom of the autoclave, and two thermocouples were used as shown in Figure 18. In the interest of safety, these tests were performed at a pressure of 5 psia helium. Photographs of the partially-assembled, long-channel hardware and the autoclave are shown in Figures 19 and 20.

Figure 19

GRAPHITE ASSEMBLY FOR LONG-CHANNEL EXPERIMENT

108-5855

Figure 20

PHOTOGRAPH OF ASSEMBLED LONG-CHANNEL AUTOCLAVE

108-5856

e. Radial Thermal-gradient Test. This experiment (CEN-148) was performed in the long-channel autoclave using the same type of specimens described in the previous section. However, to reduce longitudinal cooling effects, graphite sample holders were designed to reduce conductive heat losses from the specimens. The hardware, specimen, and thermocouple are shown in Figure 21. Two shielded-type tungsten/rhenium thermocouples

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were used to measure temperatures in the sample, one in the center coolant channel, and one in an apex coolant channel. The experiment was performed in helium at 20 psia.

Figure 21

HARDWARE USED IN THERMAL-GRADIENT TEST

A. Assembled

108-6134

B. Components

108-6138

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f. Thin-walled Autoclaves. Two high-energy tests on solid fuel (CEN-158) and on fuel beads (CEN-159) were performed in specially-designed, stainless steel, opaque autoclave of thin-wall construction to reduce flux perturbations. Schematic sketches of the specimen and hardware arrangements in both tests are shown in Figures 22 and 23. Photographs of the assemblies are shown in Figures 24 and 25. Graphite cloth was fastened over the sample cavity in the fuel-bead test to retain the beads during assembly and shipment to TREAT. No thermocouples were included in these tests. The autoclaves were evacuated to approximately 5 microns mercury before testing.

The thin-walled autoclaves used in these tests were the ones used in tests performed by the Reactor Engineering Division at ANL.⁽⁸⁾ The top cap was welded to the body of the autoclave, and a manually-operated valve was attached to the exit tubes after the interior hardware was in place. The assemblies were pressure- and leak-tested at 250 psia.

Figure 22

HIGH-ENERGY, EXPERIMENTAL ASSEMBLY, HEXAGONAL SPECIMEN

108-6408

Figure 23

SCHEMATIC DIAGRAM OF EQUIPMENT USED IN HIGH-ENERGY EXPERIMENT; CEN-159 FUEL PELLETS

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Figure 24

PHOTOGRAPH OF
HIGH-ENERGY-ASSEMBLY;
HEXAGONAL SPECIMEN

108-6443

Figure 25

PHOTOGRAPH OF
HIGH-ENERGY-ASSEMBLY;
FUEL PELLETS

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g. Combined Chemical-transient Destruct Experiments (CEN-149, -150, and -151). These experiments were performed on hexagonal, lined fuel which had been pretreated with fluorine. The purpose of these experiments was to determine if the fragmentation threshold of beaded-type fuel could be lowered by the presence of fluorine in the graphite structure. The tests were performed on samples approximately 1/2 in. long in opaque autoclaves with inner hardware arrangements similar to those described for the fission-product test CEN-132 (see Figures 14 and 15). Sample temperatures were not measured.

4. Metallographic Techniques

With a few exceptions, all the specimens irradiated in this study were subjected to metallographic examination. Both transverse and longitudinal sections were taken in several cases. In some samples, special sections were taken in order to investigate some specific effect.

Most of the specimens were mounted in Lucite by standard procedures. Because of the brittleness and high porosity of many of the irradiated specimens, the Lucite was heated slightly above 180°C and high pressure was not applied until this temperature was reached. To minimize contamination from radioactive samples, all specimens were sectioned in a ventilated hood. They were cut by hand using a hacksaw with a fine-tooth blade. The mounted specimens were carefully wet-ground through a series of papers down to 600 grit. Thereupon, they were polished using diamond abrasives of 6 μ and 1 μ particle size with kerosene as a lubricant. Most of the specimens examined were etched with a 30 v/o nitric acid-water solution.

Some problems were encountered in the polishing of samples irradiated at high-energy levels. These problems were principally due to the high porosity of the samples and the "pull-out" of the fuel beads. In many cases, the "pull-out" of the beads caused elongated gouges to be formed on the surfaces to be examined.

5. Analytical Techniques

Uranium was determined in fuel samples chosen from the fuel stock to represent the specimens that were actually tested. The samples, both with and without NbC lining, were ignited in air in platinum crucibles at about 1100°C. The residue was dissolved in dilute HNO₃ solution in the case of unlined samples, and in a dilute mixture of HNO₃ and HF in the case of lined specimens. Uranium contents were determined by standard titrimetric and colorimetric techniques.

III. RESULTS AND DISCUSSION

Forty-seven experiments were performed in TREAT on graphite samples containing pyrocarbon-coated fuel beads. Detailed transient test data and some of the post-test findings are presented in the Appendix. These data are discussed in the subsequent paragraphs in terms of the nuclear and thermodynamic test characteristics which were developed, and the general behavior of the fueled material under these conditions.

A. Nuclear and Thermodynamic Test Characteristics

The behavior of the fueled material can be best discussed as a function of the nuclear energy input and peak temperatures produced in the samples. For the purpose of this report, the nuclear energy deposited in the samples has been termed "sample heat generation" and is expressed in calories per gram of gross sample weight.

Burn-up analyses were performed on natural uranium foils and on the fueled graphite samples in several of the experiments. These analyses provided data for the four main types of test assemblies used (opaque autoclave, transparent capsule, long-channel autoclave, and thin-walled autoclave). Based on these analyses, and on the assumption of 182 MeV per fission, heat generation parameters were obtained that are believed to be representative of the flux perturbations occurring in the various types of assemblies used for testing. These parameters are listed below:

<u>Assembly</u>	<u>Fission/gm U²³⁵-MW sec</u>	<u>cal/gm U²³⁵-MW sec</u>
Opaque autoclave	2.80×10^{12}	19.40
Opaque autoclave (inner capsule)	2.41×10^{12}	16.70
Transparent capsule	4.67×10^{12}	32.36
Long-channel autoclave	2.38×10^{12}	16.49
Thin-walled autoclave	5.36×10^{12}	37.14

Specific sample heat-generation levels were determined using the appropriate heat generation parameter, the integrated power, the sample fuel loading, the total weight of the sample, and the perturbation factor due to the self-shielding of the fuel in each experiment.

A few of the tests were instrumented with tungsten-rhenium thermocouples that were placed in each sample to monitor the temperature developed in the fuel samples during the transient. The thermocouple data from experiments equipped with the tantalum-sheathed thermocouples were limited to peak sample temperatures of about 2500°C. Above these temperatures, the thermocouples failed or were erratic. Post-test examination of thermocouples of the type that had failed indicated that the

BeO insulation had melted (the melting point of BeO is 2570°C). The thermocouple data from tests instrumented with the bare-junction thermocouples indicated that peak sample temperatures as high as 2950°C could be measured successfully by this means. With comparable sample heat-generation levels, the indicated peak sample temperatures were essentially the same for both the tantalum-sheathed and bare-junction types of thermocouples. However, the response of the bare-junction thermocouples was observed to be somewhat faster than that of the tantalum-sheathed thermocouples.

In two of the opaque autoclave tests (CEN-108 and -148), a "flat-top" type transient was performed in an attempt to prolong the time at which a sample could be held at or near peak temperature. In this type of transient, the shut-down mechanism, which in TREAT is the negative reactivity coefficient of temperature, was compensated for by the continuous addition of reactivity. This, in effect, changed the transient power input to a short-term, steady-state input. The sample temperature data for the "flat-top" performed on CEN-108 at an initial reactor period of 450 msec is plotted against time in Figure 26.

Figure 26

MEASURED SAMPLE TEMPERATURE VS TIME
FOR FLAT-TOP EXPERIMENT CEN-108;
REACTOR POWER CURVES INCLUDED

Measured peak sample temperatures, as a function of sample heat generation, were described for earlier TREAT tests in the second status report.⁽²⁾ The data compiled for the current series from both opaque autoclave and transparent tests are shown in Figure 27, in which they are compared with the calculated adiabatic sample temperature. The peak sample temperature data obtained in autoclave experiments on constrained

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samples during this series of tests were in good agreement with the earlier autoclave data on dispersion-type fuels. However, somewhat higher temperatures were obtained in the photographic and autoclave tests on unconstrained samples. This difference is due to the lower conductive heat losses in the latter experiments.

Figure 27

MAXIMUM MEASURED TEMPERATURES VS
NUCLEAR ENERGY INPUT FOR
BEADED-TYPE GRAPHITE FUEL SAMPLES

Optical pyrometer data are given for the unconstrained samples tested at high-energy levels since, with one exception (bare-junction thermocouple, 2950°C), thermocouple behavior was erratic and unreliable above 2500°C. Since the optical pyrometer records the samples surface temperature, the maximum central core temperature was somewhat higher than that indicated by the pyrometer. An example of the radial temperature gradient is shown in Figure 28. On the basis of these data, the maximum core temperature may be from 200 to 300°C higher than the optical readings. At the higher energy levels (above 1500 cal/gm), the reported maximum center temperatures were based on the optical readings and are probably low.

B. Macrostructural Changes

As a result of TREAT transients, various physical changes took place in the beaded-type graphite fuel specimens. Data were obtained during these experiments relating to the structural integrity of the fuel elements, damage to the NbC coolant channel lining, dimensional stability, carbon vaporization, and weight loss. In addition, data were obtained on

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the fragmentation characteristics of chemically-treated fuel. These data are discussed in the following paragraphs.

Figure 28
TEMPERATURE-VS-TIME DATA
FROM CEN T-15

1. Structural Integrity of As-manufactured Fuel

As reported in the second status report,⁽²⁾ the fragmentation threshold for fully carbided dispersed-type graphite fuel tested in helium was 2900 cal/gm (2900°C). In the beaded-type, as-manufactured fuel, true fragmentation* was not observed except in cylindrical specimens at very high energies. The structural damage actually observed was limited to cracks generated in the webbing between coolant channels. In some samples, these cracks caused the material to split longitudinally into relatively large fragments. The crack damage and the energy level of crack occurrence were different for cylindrical specimens than for the hexagonal type. The following paragraphs describe this damage in terms of heat generation level and peak sample temperature.

Figures 29-34 are photographs of the cylindrical specimens tested without constraint. These figures illustrate the tendency of the fuel to crack in the webbing at intermediate energy levels, between 1300 and 2200 cal/gm (see Figures 30 to 32). The most serious damage of this type occurred in test CEN-128

(1900 cal/gm, 2960°C), while somewhat less damage occurred in specimen CEN-129 tested at a higher temperature (2170 cal/gm, 3100°C). The specimen in CEN T-3, tested without constraint at 2320 cal/gm (3160°C), was not damaged, but the specimens in CEN T-5 (two samples were used) were broken into large longitudinal pieces at energy inputs greater than 3500 cal/gm. This rather unusual crack behavior of unconstrained samples may be due to the different fabrication histories of the specimens used in

*Fragmentation is described here as the reduction of a sample to relatively small particles that have no preferential orientation.

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the experiments CEN-126 to -129, as opposed to those in tests CEN T-3 and T-5 (see Section II-A). In tests CEN-126 to -129, the specimens had been machined from the extruded hexagonal stock, while in tests CEN T-3 and T-5, the specimens were tested in the extruded graphitized state. Differences in orientation may have existed in the two types of fuel stock; i.e., the fuel stock used in CEN T-3 might have suffered crack damage if it had been tested, unconstrained, at the lower levels.

Figure 29

PHOTOGRAPH OF CYLINDRICAL FUEL SPECIMEN
IN EXPERIMENT CEN-126; SAMPLE HEAT
GENERATION, 1130 cal/gm

108-5894

Figure 30

PHOTOGRAPH OF CYLINDRICAL FUEL SAMPLE IN
EXPERIMENT CEN-127; SAMPLE HEAT
GENERATION, 1320 cal/gm

108-5895

Figure 31

PHOTOGRAPH OF CYLINDRICAL FUEL SAMPLE IN EXPERIMENT
CEN-128; SAMPLE HEAT GENERATION, 1900 cal/gm

108-5896

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Figure 32

PHOTOGRAPH OF CYLINDRICAL FUEL SPECIMEN
IN EXPERIMENT CEN-129; SAMPLE HEAT
GENERATION, 2170 cal/gm

108-5897

Figure 33

PHOTOGRAPH OF CYLINDRICAL FUEL SAMPLE
IN EXPERIMENT CEN T-3; SAMPLE HEAT
GENERATION, 2320 cal/gm

108-5542

Figure 34

PHOTOGRAPH OF TWO CYLINDRICAL FUEL SAMPLES IN EXPERIMENT CEN T-5;
SAMPLE HEAT GENERATION, 3970 AND 3660 cal/gm

108-5540

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Photographs of cylindrical specimens tested while under constraint are shown in Figures 35 through 39. In these specimens, the cracking at intermediate energy (CEN-106, 1500 cal/gm, 2200°C) occurred again, but was confined to a general deterioration of the ends of the sample. The most interesting feature in the constrained series of tests was the structural stability of the fuel samples at the higher energy levels (CEN T-4 and T-6, 3700 and 3670-3550 cal/gm, respectively). Considering the damage to CEN T-5 (Figure 34), the constraint was sufficient to hold the fuel together. A solid cylindrical sample (no longitudinal coolant channels), tested under constraint, did fragment at 4360 cal/gm, as shown in Figure 39. The particle size distribution obtained in this test is shown in Figure 40. The Sauter mean particle diameter (d_{sv})* was 27.5 mils, but 39% of the material was in fragments larger than 250 mils in diameter.

Figure 35

PHOTOGRAPH OF CONSTRAINED
CYLINDRICAL FUEL SAMPLE IN
EXPERIMENT CEN-106; SAMPLE
HEAT GENERATION, 1500 cal/gm

108-5449

Figure 36

PHOTOGRAPH OF CONSTRAINED
CYLINDRICAL FUEL SAMPLE IN
EXPERIMENT CEN-105; SAMPLE
HEAT GENERATION, 2340 cal/gm

108-5448

* d_{sv} - The Sauter diameter is the diameter of a particle having the same surface-area-to-volume ratio as the bulk.

108-5541

Figure 37

PHOTOGRAPH OF CONSTRAINED CYLINDRICAL FUEL SAMPLE IN
EXPERIMENT CEN T-4; SAMPLE HEAT GENERATION, 3700 cal/gm

Figure 38
PHOTOGRAPH OF THE TWO
CONSTRAINED CYLINDRICAL
FUEL SAMPLES IN EXPERIMENT
CEN T-6; SAMPLE HEAT
GENERATION, 3670 AND
3550 cal/gm

108-5539

108-6584

Figure 39

PHOTOGRAPH OF CONSTRAINED,
SOLID, CYLINDRICAL FUEL SAMPLE
IN EXPERIMENT CEN-157; SAMPLE
HEAT GENERATION, 4360 cal/gm

Figure 40
PARTICLE SIZE DISTRIBUTION
FOR CEN-157

The hexagonal specimens were all tested without constraint. Figures 41 through 52 are photographs of a series of specimens that were tested in both autoclaves and transparent capsules. None of the hexagonal specimens showed structural damage up to a heat generation level of 2490 cal/gm. Cracks were noted in the webbing of the fuel material in specimens CEN T-9, T-17, and CEN-158, which were tested at 2490, 2660, and 5580 cal/gm respectively. A more detailed view of the webbing cracks in test CEN T-17 is shown in Figure 53. Reference to the temperature data of Figure 27 shows that crack damage in the webbing of as-manufactured hexagonal fuel occurs at peak sample temperatures above 3200°C.

Figure 41
PHOTOGRAPH OF HEXAGONAL,
NbC-LINED FUEL SAMPLE IN
CEN-133; SAMPLE HEAT
GENERATION, 800 cal/gm

108-5899

Figure 42
PHOTOGRAPH OF HEXAGONAL, NbC-
LINED FUEL SAMPLE IN CEN-154;
SAMPLE HEAT GENERATION,
890 cal/gm

ANL-ID-103-7319

Figure 43

PHOTOGRAPH OF HEXAGONAL,
NbC-LINED FUEL SAMPLE IN
CEN T-13, SAMPLE HEAT
GENERATION, 1140 cal/gm

108-6404

Figure 44

PHOTOGRAPH OF HEXAGONAL,
NbC-LINED FUEL SAMPLE IN
CEN T-15; SAMPLE HEAT
GENERATION, 1280 cal/gm

108-6571

Figure 45

PHOTOGRAPH OF HEXAGONAL,
NbC-LINED FUEL SAMPLE IN
CEN T-16; SAMPLE HEAT
GENERATION, 1570 cal/gm

108-6572

Figure 46

PHOTOGRAPH OF HEXAGONAL,
NbC-LINED FUEL SAMPLE IN
CEN T-14; SAMPLE HEAT
GENERATION, 1720 cal/gm

108-6405

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Figure 47

PHOTOGRAPH OF HEXAGONAL, NbC-LINED FUEL
SAMPLE IN CEN T-12; SAMPLE HEAT GENERATION,
2060 cal/gm

108-6403

Figure 48

PHOTOGRAPH OF HEXAGONAL, NbC-LINED FUEL
SAMPLE IN CEN-155; SAMPLE HEAT GENERATION,
1870 cal/gm

ANL-ID-103-7322

Figure 49

PHOTOGRAPH OF HEXAGONAL, NbC-LINED FUEL
SAMPLE IN CEN T-18; SAMPLE HEAT GENERATION,
2160 cal/gm

108-6575

Figure 50

PHOTOGRAPH OF HEXAGONAL, NbC-LINED FUEL
SAMPLE IN CEN T-9; SAMPLE HEAT GENERATION,
2490 cal/gm

108-5818

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Figure 51

PHOTOGRAPH OF HEXAGONAL,
NbC-LINED FUEL SAMPLE IN
CEN T-17; SAMPLE HEAT
GENERATION, 2660 cal/gm

108-6574

Figure 52

PHOTOGRAPH OF HEXAGONAL, NbC-LINED FUEL SAMPLE IN
CEN-158; SAMPLE HEAT GENERATION, 5580 cal/gm

108-6579

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Figure 53
PHOTOMICROGRAPH OF SECTION OF SAMPLE IN
CEN T-17 SHOWING CRACKS IN WEBBING

10X

Based on this series of tests, it is evident that the as-manufactured, hexagonal, beaded-type fuel has an extraordinary ability to withstand total fragmentation at very high transient energies and reactor periods as low as 39 milliseconds. Hexagonal samples absorbed as much as 5580 cal/gm without fragmenting.

2. Structural Integrity of Back-reacted Fuel

Earlier TREAT tests on back-reacted, dispersed, carbide graphite fuel indicated that fragmentation in helium occurred at heat generation levels as low as 1200 cal/gm (1900°C). Three samples of beaded-type graphite fuel that had been back-reacted to give 3% (CEN T-8) and 22% (CEN T-10, CEN-146) conversion of UC_2 to UO_2 were tested. The samples in the CEN T-8 and T-10 photographic experiments were tested unconstrained in helium at 2530 and 2750 cal/gm, respectively; CEN-146 (autoclave experiment) was tested in air at 1850 cal/gm. No fragmentation occurred in the CEN T-8 sample, while the samples in CEN T-10 and CEN-146 fragmented. Photographs of the fragmented samples are shown in Figures 54 and 55. Included are plots of particle size distribution for each test. The mean particle size diameter was determined to be 49.3 and 137 mils for tests CEN T-10 and CEN-146, respectively. The fragments from these two tests were considerably coarser than those produced from back-reacted, dispersed, carbide fuel tested at comparable energy levels [compare to tests CEN-74, -75, and -76 in Status Report II⁽²⁾].

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Figure 54

HEXAGONAL, NbC-LINED FUEL SAMPLE IN CEN T-10;
SAMPLE HEAT GENERATION, 2750 cal/gm

A. Fragmented Sample

108-5976

B. Detail of Fragments

108-5957

2.5X

C. Particle-size Distribution

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Figure 55

HEXAGONAL, NbC-LINED FUEL SAMPLE IN CEN-146;
SAMPLE HEAT GENERATION, 2150 cal/gm

A. Fragmented Sample

108-6292

UNCLASSIFIED

B. Detail of Fragments

2.5X

C. Particle-size Distribution

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3. Damage to NbC Lining

The NbC lining in the coolant channels of the hexagonal specimens melted on specimens tested at sample heat-generation levels above 1720 cal/gm (peak specimen temperature $>2800^{\circ}\text{C}$). The melted lining characteristically formed ball-like masses on the ends of the samples at the orifices of the coolant channels. This phenomenon is shown in the photograph of the sample from test CEN T-8 (2530 cal/gm, 3200°C) in Figure 56. Figure 57 is a photograph of a series of lined specimens tested at heat generation levels from 1190 cal/gm (CEN T-15, 2240°C) to 2160 cal/gm (CEN T-18, 3120°C). The lining was not melted on CEN T-14 (1720 cal/gm, 2800°C), but it was melted on CEN T-12 (2060 cal/gm, 3040°C). In some cases, as in test CEN T-18, the ball-like melts of the lining essentially blocked the coolant channels. The lining damage noted in all of the experiments is summarized in Table I.

Figure 56

PHOTOGRAPH OF HEXAGONAL,
NbC-LINED FUEL SAMPLE IN
CEN T-8; SAMPLE HEAT
GENERATION, 1850 cal/gm

108-5817

UNCLASSIFIED

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Figure 57

LINING DAMAGE AS A FUNCTION OF INCREASING TRANSIENT HEAT-GENERATION LEVEL AND PEAK SAMPLE TEMPERATURE

Series of Test Samples Showing Relative Lining Damage

T-15

T-14

T-12

T-18

Table I

NbC LINING DAMAGE AT VARIOUS LEVELS
OF SAMPLE HEAT GENERATION

Test No.	Heat Generation, cal/gm	Lining Damage
CEN T-13	1140	Not melted
CEN T-15	1190	Not melted
CEN T-16	1570	Not melted
CEN T-14	1720	Not melted
CEN-13	1730	Partially melted
CEN-132	1760	Melted
CEN-155	1870	Melted
CEN T-12	2060	Melted
CEN T-18	2160	Melted

The melting point of NbC is reported to be about 3500°C. It can be lowered by adding uranium and carbon. The ternary phase relationships have not been fully established, but a binary eutectic (NbC + C) has been reported at 3250°C, and a ternary eutectic (NbC + UC₂ + C) at 2350°C. Figures 58 through 65 are photomicrographs of the lining areas on a series of samples tested at increasing levels of heat generation (and peak sample temperature). The onset of melting appears to be preceded by the formation of a second phase, as shown in the lining on the sample from test CEN T-14 (Figure 60). The threshold of interaction of the lining with the matrix and/or fuel appears to be about 1190 cal/gm, based on the small amount of second-phase material noted at the lining interface on test CEN T-15 (Figure 59, 2260°C). The etched structure of the melted lining on samples tested at peak sample temperatures above 2800°C has a eutectic-like appearance with a precipitate of carbon needles.

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Figure 58

PHOTOMICROGRAPH OF UNIRRADIATED, STANDARD
FUEL SAMPLE SHOWING NbC LINING

As Polished

200X

Figure 59

PHOTOMICROGRAPH OF SAMPLE IN CEN T-15 AT
1280 cal/gm (2350°C), SHOWING NbC LINING

As Polished

200X

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Figure 60

PHOTOMICROGRAPH OF SAMPLE IN CEN T-14 AT
1720 cal/gm (2800°C), SHOWING NbC LINING

As Polished

200X

Figure 61

PHOTOMICROGRAPH OF SAMPLE IN CEN-132 AT
1760 cal/gm (2820°C), SHOWING NbC LINING

80X

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Figure 62

PHOTOMICROGRAPH OF SAMPLE IN CEN T-12 AT 2060 cal/gm
(3040°C), SHOWING A PORTION OF MELTED NbC LINING

As Polished

~190X

Figure 63

PHOTOMICROGRAPH OF SAMPLE IN CEN T-18 AT
2160 cal/gm (3100°C)

As Polished

75X

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Figure 64

PHOTOMICROGRAPH OF SAMPLE IN CEN T-9 AT
2490 cal/gm (3200°C), SHOWING NbC LINING

As Polished

75X

Figure 65

PHOTOMICROGRAPH OF SAMPLE IN CEN T-17 AT
2660 cal/gm (3200°C), SHOWING NbC LINING

As Polished

75X

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4. Fragmentation of Fluorinated Fuel

The objective of autoclave experiments CEN-149 through -151 was to determine the fragmentation characteristics of beaded graphite fuel pretreated with fluorine gas. The pretreatment consisted of subjecting half-inch-long specimens of NbC-lined hexagonal fuel to a flow of fluorine gas for 15 min at 300°C. This treatment resulted in a 15% weight loss in the specimen, and about half the NbC lining was destroyed. A thermal decomposition analysis indicated that the specimens contained about one w/o fluorine before the transient irradiations. Pretest metallographic examinations of a control specimen revealed that the fluorine was not uniformly distributed but appeared to be concentrated at the surfaces of the specimen and the coolant channels. Erosion had occurred on the surfaces of the specimen directly exposed to the fluorine gas flow, as can be seen in the photograph of a control specimen shown in Figure 66.

Figure 66

SIDE VIEW OF AS-RECEIVED SAMPLE USED IN CEN-149,
SHOWING EFFECT OF FLUORINE TREATMENT
(Fragment of Fuel on Right Shows Partial Disintegration
of NbC Lining by Fluorine Attack)

108-6296

The results of the transient tests on the three samples are detailed in Table II. These data indicate that the threshold of partial fragmentation of the beaded-type fuel can be lowered to 670 cal/gm (CEN-149, 1400°C) by chemical pretreatment with fluorine. Each specimen fragmented into four or five relatively large pieces and many small fragments. Figures 67 to 69 are photographs of the specimens after testing and include particle size distributions calculated for all the fragments of each sample and neglecting the larger fragments. The large fragments were fitted together to

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reconstruct the specimen (see Figures 67 to 69) in an attempt to characterize the mode of fragmentation. All the specimens appeared to have fractured longitudinally through the coolant channels and not in the webbing. This behavior seems to indicate that the fragmentation was initiated on those areas where fluorine attack was concentrated, i.e., near the surfaces of the coolant channels. Samples of the large fragments from the three experiments were metallographically examined and appeared quite similar to samples not chemically treated but tested at comparable energy levels. This is further evidence that only the chemically treated portion of the specimen fragmented.

Table II

RESULTS OF TREAT TRANSIENTS ON LINED, HEXAGONAL, BEADED-TYPE, FUELED GRAPHITE SPECIMENS, PRETREATED WITH FLUORINE

CEN No	MW-sec	Period (msec)	cal/gm	w/o ^a Large Fragments	d _{sv} ^c All Fragments (mil)	d _{sv} ^c Only Small Fragments (mil)	Pressure ^b Developed (psia)
149	275	59	670	70.0	92.9	28.9	3.8
150	417	42	1010	72.0	72.8	21.4	5.6
151	820	42	1990	42.2	95.5	49.9	14.1

^aBased on initial sample weight

^bPressure measured in autoclave after test Autoclaves were initially evacuated

The results of a mass spectrographic analysis of gas samples from all three tests are shown in Table III. There was no evidence of carbon-fluorine compounds in these samples. However, gaseous products were found corresponding to a carbon-water reaction.

In an attempt to determine the decomposition products that are formed on heating this material, a small sample was heated in an evacuated quartz tube, first to 460°C, and then to 1085°C. Gas pressures were measured as a function of temperature and, after cool-down, gas samples were taken for mass spectrographic analysis at the two temperatures. The composition of the gas formed at the two lower temperatures is shown in Table III. The gas pressure developed in the system (43 cc) after sample cool-down corresponded to 1.3 psia (1.57×10^{-4} moles) for the 460°C condition, and approximately 3.5 psia (4.22×10^{-4} moles) for the 1085°C condition. On the basis of the results in Table III, water was present in the sample and furnishes an explanation for the presence of carbon-water reaction products in the TREAT tests. While the identity of mass 47, 81, and 77 components cannot be firmly established, the absence of well-known carbon-fluorine compounds, and the presence of SiF₄ in the 460°C test, indicate that the fluorine in the material was not firmly held in the graphite structure. It apparently was evolved either as fluorine or as HF. The absence of these two species in the gas phase can be assumed to be due to chemical reactivity, i.e., with SiO₂ in the case of quartz, or with stainless steel in the case of the TREAT autoclaves.

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Figure 67

HEXAGONAL, NbC-LINED FUEL SAMPLE IN CEN-149;
SAMPLE HEAT GENERATION, 670 cal/gm

A. Fragmented Sample

108-6348

B. Large Fragments

108-6347

C. Complete Particle Size Distribution

D. Particle Size Distribution, Less Large Particles

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HEXAGONAL, NbC-LINED FUEL SAMPLE IN CEN-150;
SAMPLE HEAT GENERATION, 1010 cal/gm

B. Large Fragments

108-6346

108-6345

C. Complete Particle Size Distribution

D. Particle Size Distribution, Less Large Particles

Figure 69

HEXAGONAL, NbC-LINED FUEL SAMPLE IN CEN-151;
SAMPLE HEAT GENERATION, 1990 cal/gm

A. Fragmented Sample

108-6344

B. Large Fragments

108-6342

C. Complete Particle Size Distribution

D. Particle Size Distribution, Less Large Particles

Table III

MASS SPECTROGRAPHIC ANALYSIS OF GAS SAMPLES:
TREAT TESTS CEN-149, -150, AND -151;
BENCH TESTS 460-AF AND 1085-BF

Test	Temp., °C	Gas Composition, v/o										
		H ₂	H ₂ O	CO	CO ₂	HC's	O ₂	N ₂	SiF ₄	47	77	81
CEN-149	1400(b)	33.1	0.2	57.4	6.8	1.7	0.2	0.6	-	-	-	-
CEN-150	2000(b)	34.8	4.0	54.7	3.0	2.2	0.1	1.1	-	-	-	-
CEN-151	3000(b)	53.3	0.3	41.3	1.5	1.8	<0.1	1.5	-	-	-	-
460-AF(a)	460(c)	23.8	3.4	6.8	30.3	2.1	-	-	33.3	0.1	0.3	0.1
1085-BF(a)	1085(c)	42.7	2.1	24.0	6.7	9.5	-	-	0.7	2.5	1.5	10

(a) Sample weighed 1.9437 gm before test, cracked into three pieces plus small particles during test.

(b) Estimated temperatures.

(c) Temperatures measured by Chromel-Alumel thermocouple.

5. Carbon Vaporization and Weight Loss

A black cloud of vaporized material was noticed in the motion-picture records of transparent capsule tests. The cloud formation appeared to be most pronounced at heat-generation levels above 2000 cal/gm (<3000°C). In addition, deposits of carbon were noted on the surfaces of some of the specimens tested in both transparent capsules and opaque autoclaves.

A typical example of the type of carbon deposit noted in transparent capsule tests is shown in the photographs of the CEN T-9 and T-8 samples after test (see Figures 50 and 56). The long needlelike deposit, which is shown in more detail in Figure 70, appeared to have grown from the top surface and was quite flexible. The structure consisted of "piled-up" shiny black spheres which are believed to be a form of pyrocarbon formed by redeposition of vaporized carbon on the cooler sample surfaces.

Figure 70

PHOTOGRAPH OF NEEDLE-LIKE
CARBON DEPOSIT WHICH GREW
FROM TOP SURFACE OF
SAMPLE IN CEN T-8

4X

A slightly different type of deposit was noted in autoclave samples that were tested in contact with graphite hardware. A typical example is the long-channel experiment, CEN-139, in which the fuel elements are in contact on both ends with unfueled, 19-hole, hexagonal rods. The test was designed to study the deposition of material evolved from the sample onto the cooler hardware surfaces. The sample in the test was observed to have on its ends a deposit of carbon which grew up into the holes of the unfueled rods, as shown in Figure 71. The carbon deposits did not block the coolant channels of the unfueled pieces but formed as shells extending up into the channels. Similar behavior was noted on autoclave tests CEN-132 and -155. A metallographic examination of the end surfaces of the CEN-139 sample indicated that the deposits were a form of pyrocarbon, as shown in Figure 72.

Figure 71

PHOTOGRAPH OF SAMPLE IN CEN-139, SHOWING CARBON DEPOSITS ON SAMPLE AND GRAPHITE CHANNEL PLUGS

108-5975

A most unusual and interesting demonstration of carbon vaporization and redeposition was noted in a high-energy test performed on hexagonal fuel (CEN-158, see Figure 52). In this test, a shell of pyrocarbon about 10 to 20 mils thick formed circumferentially around the sample (Figure 73), and thin deposits of pyrocarbon were formed on the inner surfaces of the top hollow graphite sample holder at a distance 5 in. from the fuel sample (see Figure 74). A photomicrograph of the shell formed around the sample is shown in Figure 75, which indicates that the shell was actually two layers of pyrocarbon. Uranium was found in the circumferential shell and in the thin deposits on the top sample holder. The distribution of fuel in the deposits and in the sample, compared with the initial sample loading, was as follows:

Initial w/o uranium in fuel sample = 19.87 (2.19 gm)
w/o uranium in fuel sample, after test = 8.77
Uranium in shell around sample = 0.399 gm
Uranium found in sample holder deposit, 5 in. from sample =
0.09 gm

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Figure 72

PHOTOMICROGRAPH OF SAMPLE IN CEN-139 AT
1730 cal/gm (2800°C), SHOWING GRAPHITE DEPOSIT
OVER A BEAD OF MELTED NbC LINING

As Polished

200X

Figure 73

PHOTOGRAPH OF GRAPHITE SHELL AROUND SAMPLE IN CEN-158

108-6586

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Figure 74

PHOTOGRAPH OF TOP SAMPLE HOLDER, SPLIT
LONGITUDINALLY TO SHOW DEPOSIT

108-6588

Figure 75

PHOTOMICROGRAPH OF DEPOSITS ON SAMPLE IN CEN-158

As Polished

200X

The threshold of carbon vaporization was difficult to ascertain from the motion-picture records and post-test visual examinations. To determine this parameter, weight losses were determined on several specimens. Although some uranium was undoubtedly lost from the samples, it was believed that a major contributor to sample weight loss was carbon vaporization. In Figure 76 the weight-loss data obtained from several tests are plotted as a function of heat generation. Although there is considerable scatter in the data, the weight loss increases rapidly at transient levels above 2000 cal/gm (3000°C). On the basis of visual evidence and the weight-loss data, the threshold of carbon vaporization in a 20-psia helium environment under transient test conditions may be somewhat lower than this level.

Figure 76
SAMPLE WEIGHT LOSS AS A
FUNCTION OF SAMPLE HEAT
GENERATION FOR BEADED-
TYPE FUEL

6. Dimensional Changes

Several hexagonal specimens tested in photographic capsules were measured before and after irradiation to record dimensional changes. As shown in Figure 77, the percent increase in axial and radial dimensions generally increased with increasing heat generation. Although there is considerable scatter in the data, the threshold of dimensional change appeared to be at about 1600 cal/gm. The growth was anisotropic; i.e., the radial growth was three to six times as great as the axial growth. The swelling can be attributed to thermal stresses plastically deforming the specimens during rapid heating. The anisotropic nature of the growth is related to the preferred orientation that extruded fuel bodies have before test. During extrusion, the basal planes ("a" direction) of hcp graphite are aligned parallel to the longitudinal axis of the fuel. The thermal

expansion coefficient of graphite in the "a" direction parallel to the basal planes is only half the expansion coefficient in the "c" direction (perpendicular to the basal planes). Under this condition, a higher radial deformation might be expected.

Figure 77
DIMENSIONAL INCREASES AS A
FUNCTION OF HEAT GENERATION
FOR BEADED-TYPE FUEL

C. Microstructural Changes

1. Metallographic Studies

As the result of metallographic examination of specimens (tested at increasing energy levels) progressive damage was noted to the fuel beads. This damage involved migration of uranium into the pyrocarbon coatings and general disintegration of the fuel beads. Significant microstructural heterogeneity was noted in given samples. The amount of damage observed on neighboring fuel beads was sometimes different, and the damage was more intensive in beads located in the center portions of the sample due to the presence of thermal gradients. As a result, the discussion which follows is a generalization only and represents the way most of the fuel beads appeared in the center of a particular specimen. A series of photomicrographs of fuel beads examined in several specimens tested at various heat-generation levels is shown in Figures 78 to 86.

The first definite indication of uranium diffusion into the coating was seen in the fuel beads of the sample in experiment CEN T-13 tested at 1140 cal/gm (2260°C). The diffusion occurred in progressively greater amounts in the tests at energy levels above this. The first definite indication of damage to the pyrocarbon coatings was noted in experiment CEN T-16 tested at 1570 cal/gm (2660°C). From the conditions of the fuel beads and coatings in the CEN T-16 test, the threshold for coating deterioration appears to be somewhat below this energy level. The coatings were less and less distinguishable in samples tested at progressively higher energy levels. They were practically indistinguishable in experiments CEN T-14 (1720 cal/gm, 2800°C) and T-18 (2160 cal/gm, 3080°C).

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On samples tested at higher energy levels, the beads were practically destroyed and extensive uranium migration had occurred in the matrices (CEN T-17, 2660 cal/gm, $>3200^{\circ}\text{C}$). With the exception of a very few shell-like remains, no fuel beads were found in the sample examined from CEN-158 (5580 cal/gm). The above observations indicate that damage to the fuel beads during transient testing appears to be preceded by a migration of uranium into the coatings beginning at about 1100-1200 cal/gm ($2100-2250^{\circ}\text{C}$). The damage to the coatings is accelerated when the melting point of UC_2 (reported to be 2450°C) is exceeded; i.e., the threshold of coating damage on this basis is probably about 1400 cal/gm.

Figure 78
PHOTOMICROGRAPH OF TYPICAL FUEL BEADS
IN UNIRRADIATED STANDARD

250X

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Figure 79

PHOTOMICROGRAPH OF FUEL BEADS IN CEN-133 AT 800 cal/gm (1640°C)

200X

Figure 80

PHOTOMICROGRAPH OF FUEL BEADS IN CEN T-13 AT 1140 cal/gm (2260°C)

400X

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Figure 81

PHOTOMICROGRAPH OF FUEL BEADS IN CEN T-15 AT 1280 cal/gm (2360°C)

200X

Figure 82

PHOTOMICROGRAPH OF FUEL BEADS IN CEN T-16 AT 1570 cal/gm (2660°C)

200X

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Figure 83

PHOTOMICROGRAPH OF FUEL BEADS IN CEN T-14 AT 1720 cal/gm (2800°C)

200X

Figure 84

PHOTOMICROGRAPH OF FUEL BEADS IN CEN T-18 AT 2160 cal/gm (3080°C)

200X

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Figure 85

PHOTOMICROGRAPH OF FUEL BEADS IN CEN T-17 AT 2660 cal/gm ($>3200^{\circ}\text{C}$)

200X

Figure 86

PHOTOMICROGRAPH OF SAMPLE IN CEN-158 AT 5580 cal/gm ($>3200^{\circ}\text{C}$)

80X

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2. EPM (Electron Probe Microanalyzer) Studies

Samples taken from the fuel specimens in seven experiments and one control sample were studied via the Cambridge electron probe microanalyzer by the IIT Research Institute. These samples are listed in Table IV. The results of the analyses are described in the following paragraphs.

Table IV

SUMMARY OF TEST PARAMETERS OF SPECIMENS STUDIED VIA EPM

Specimen No.	Original Shape	Sample Heat Generation, cal/gm	Peak Temperature, °C
21	Round	(Control)	-
CEN-108	Round ^a	1960	2400
CEN-127	Round	1320	2400
CEN-139	Hexagonal	1730	2800
CEN-107	Round ^a	2320	2850
CEN-129	Round	2170	3100
CEN T-8	Hexagonal	2530	>3200
CEN T-4	Round ^a	3700	>3200

^aConstrained

a. Control Sample No. 21. Two separate carbide particles were examined. Uranium was distributed in the surrounding pyrolytic graphite coating but not to any significant extent into the graphite matrix. Static analysis of one particle indicated 83.4 w/o uranium in the core, and 14 w/o uranium average in the pyrolytic graphite coating. In the second particle, some uranium diffusion into the coating had occurred. The diffusion contact between particle and coating was poor. There was no apparent difference between the central and outer portions of the fuel element.

b. Sample CEN-108 (2400°C). Extensive diffusion of uranium into the pyrolytic graphite coating had occurred. Spot analysis indicated the concentration in the richer portions of the diffused zone to be approximately 38.1 w/o uranium. One UC₂ particle was analyzed to be 76.6 w/o uranium, clearly indicating outward migration of uranium. There appeared to be no significant migration of the uranium into the matrix.

c. Sample CEN-127 (2400°C). Several fuel particles were found to be intact. The uranium content in particle sites varied between 68.1 and 3.8 w/o. Diffusion of uranium into the coating had occurred. Some migration of uranium to the matrix occurred.

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d. Sample CEN-139 (2800°C). The quantitative analysis performed on this sample is summarized as follows:

	<u>w/o Uranium</u>	<u>w/o Niobium</u>
UC ₂ particle	85.3	-
Pyrolytic carbon coating	3.4-31.2	-
NbC lining, intact	-	83.2-92.8
NbC lining (melted)	3.4	61.7-71.3

The UC₂ particles were deteriorated. Both diffusion of uranium into the pyrocarbon coating and migration of uranium into the matrix had occurred. Uranium was found in portions of the NbC lining that showed incipient melting.

e. Sample CEN-107 (2850°C). The UC₂ particles suffered extensive deterioration. Prior carbide particle sites were still evident as craters or holes.

f. Sample CEN-129 (3100°C). The uranium content of pyrolytic fuel particle coatings at former UC₂ particle sites was between 4.7 and 87.7 w/o. Craters or holes were evident.

g. Sample CEN T-8 (>3200°C). Uranium was found within pyrolytic graphite coatings that had remained intact. Two typical spot analyses were 7.0 and 38.8 w/o uranium. There was some evidence of former particle sites in which high and highly variable concentrations of uranium were found. Gross migration into the graphite matrix had occurred.

Uranium was found to have diffused into the NbC coolant channel lining. Several areas of incipient melting within the carbide lining were analyzed with the following results:

<u>NbC Lining</u>	
w/o Niobium	66.5, 72.9, 79.1, 84.5
w/o Uranium	2.1, 8.0, 37.9

These values indicate the composition ranges encountered. Both the uranium and niobium showed strong microsegregation suggestive of an eutectic distribution.

Pyrolytic carbon redeposits were found on the outer side of the NbC lining. No niobium could be detected in any of the pyrolytic redeposits.

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h. Sample CEN T-4 ($>3200^{\circ}\text{C}$). This sample suffered extensive deterioration. No evidence of prior sites of the carbide particles was found. Gross migration of the uranium into the matrix had occurred.

All of these samples were surveyed from the outer to the inner surfaces at as low a magnification as possible to show any massive concentration gradient in uranium content. None was found.

D. Fission-product Release

The release of fission products from the graphite fuel elements during a reactor transient has significance from the standpoint of safety requirements and criticality experiments. In addition, reactor transients have been considered as a means of reducing the fission-product inventories in the reactor core.

Two types of fission-product release information were obtained from TREAT tests during this period: (1) xenon-133 release to the gas phase, and (2) iodine-131 and tellurium-132 release to the sample environment.

1. Xenon-133 Release

The xenon-133 inventory in a graphite fuel sample after a typical TREAT transient of 10^{15} thermal fissions is plotted in Figure 87 as a function of time after transient. Samples of gas were taken from several autoclave tests at various elapsed times after transient testing.

Figure 87

FISSION-PRODUCT INVENTORY FROM 0.1-minute BURST OF 10^{15} THERMAL FISSIONS OF $^{133}\text{NUCLIDES}$

These samples were analyzed by gamma scintillation spectrometer for xenon-133 activity. The total released activity at the time of sampling (A_s) and the calculated number of thermal fissions for each test are listed in Table V. The xenon-133 sample inventory level during the high-temperature portion of the transients (i.e., at about 6 seconds elapsed time in Figure 87) corresponds to 0.02 microcurie for a nominal 10^{15} thermal fissions. The release data in Table V indicate that more xenon-133 was found in the gas phase than was present in the fuel samples during the high-temperature portion of the transient

tests. This discrepancy can be explained if the majority of the xenon activity in the gas phase is assumed to be due to the decay of ¹³³ chain nuclides that were released during the transient, i.e. antimony-133, tellurium-133, tellurium-133* (nuclear isomer) and iodine-133.

Table V
XENON RELEASE

CEN No	Energy Input, cal/gm	Fissions $\times 10^{15}$	Temperature °C	Xenon-133 Activity at Time of Sampling		Percent Release (A_s/A_t) 100	Test Environment	Sample Condition
				Gas Phase, A_s , μ C	Sample Inventory, A_t , μ C			
160	540	0.031	1200	0.23	64.6	0.35	Helium	Intact
153	390	0.21	1440	0.234	312	0.08	Vacuum	Intact
133	800	2.95	1640	1.2	3020	0.04	Helium	Intact
154	890	0.45	1800	4.76	600	0.7	Vacuum	Intact
152	1041	21.9	2090	80.8	2520	3.2	Helium	Intact
126	1130	0.68	2140	0.3	65.5	0.5	Helium	Intact
106	1500	0.71	2210	155.4	830	18.7	Helium	Cracked
148	1035	11.64	2380	96.5	3316	2.9	Helium	Intact
127	1320	0.84	2400	10.6	92.5	11.5	Helium	Cracked
161	1380	0.079	2460	0.66	136.7	0.48	Helium	Intact
131	1650	1.02	2750	189.8	598	31.8	Sea water	Intact, surface eroded
139	1730	6.40	2800	347.3	6200	5.6	Helium	Intact
132	1760	1.09	2820	106.6	1710	6.3	Helium	Intact
107	2320	1.11	2850	71.0	1320	5.4	Vacuum	Intact
146	1850	1.26	2900	460	1980	24.2	Air	Frag (22% B R *)
105	2340	1.10	2900	62.5	1045	6.0	Helium	Intact
155	1870	1.16	2920	167	1740	9.6	Vacuum	Intact
128	1900	1.10	2940	62.6	121	51.8	Helium	Cracked, 3 places
156	1970	1.45	3000	509	2180	23.2	Dist water	Frag (22% B R *)
104	2780	3.41	3000	253.5	3240	7.8	Helium	Intact
129	2170	1.21	3100	70.2	188	37.4	Helium	Cracked
162	2470	0.144	3200	37.2	234.7	15.83	Helium	Intact
158	5580	10.3	3200	807	3090	26.0	Vacuum	Intact, cracked

*B R = back-reacted

The percent xenon-133 release can be estimated if the activity found in the gas phase at the time of sampling (A_s) is compared with the xenon-133 total sample inventory (A_t) determined at a corresponding elapsed time after the transient. The percent xenon-133 release calculated for the various tests on this basis are given in Table V as a function of heat generation, structural integrity, and estimated peak sample temperature. In general, the release of xenon appears to be low at heat generation levels up to 1000 cal/gm (peak sample temperature, approximately 2000°C). The highest release data were obtained from fuel samples that were structurally damaged by the transient and from the samples tested in water. There were discrepancies in the fractional release data obtained on cracked and fragmented samples, just as were there differences in the release from intact and fragmented samples tested in water. Under conditions of cracking or fragmentation, readsorption of the xenon can occur

during the period after test when the sample is in contact with the gas phase at room temperature. Since several of the tests were not sampled for periods up to 30 days, the xenon release data are somewhat questionable.

The most meaningful xenon release data were obtained during the testing of as-manufactured specimens (CEN-132, -153, -154, -155, -160, -161, and -162) in which the gas samples were taken at 2 to 4 days after test. These data indicate that the release of xenon from the samples is significant at about 1000 cal/gm (~1%) and climbs to about 5% at approximately 1900 cal/gm (CEN-155, 2420°C).

Above 1900 cal/gm, the release increases rapidly from intact specimens, approximately tripling at a level of about 2470 cal/gm (CEN-162, >3200°C).

2. Iodine-131 and Tellurium-132 Release

The 131 and 132 chain nuclide inventories for a sample, given a typical TREAT transient, are plotted as a function of elapsed time in Figures 88 and 89. Autoclave tests were conducted on as-manufactured, lined specimens in TREAT in which the hardware and autoclave interiors were leached to recover released iodine-131 and tellurium-132. Similar experiments were performed on 22% back-reacted fuel in air and in distilled water. Based on the elapsed time after test in which the nuclides were separated from the leach solutions, percent release data were calculated in a manner similar to that just described for xenon-133. These data are shown in Table VI. As in the case of the xenon-133 release (CEN-132, -153, -154, -155, -160, -161, and -162), the iodine-131 release becomes appreciable at about 890 cal/gm (1800°C) and increases slowly to about 2200 cal/gm. The release more than doubles in the range of 2200 to 2300 cal/gm (1800°C to 3200°C).

Figure 88
FISSION-PRODUCT INVENTORY FROM 0.1-minute
BURST OF 10^{15} THERMAL FISSIONS OF 131 NUCLIDES

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Figure 89

FISSION-PRODUCT INVENTORY FROM 0.1-minute
BURST OF 10^{15} THERMAL FISSIONS OF 132 NUCLIDES

Table VI

IODINE-131 AND TELLURIUM-132 RELEASE FROM
BEADED-TYPE GRAPHITE FUEL DURING TREAT TESTING

CEN No	cal/gm (Temp, °C)	Test Environment	Sample Condition	Activity of Time of Sampling, μC				% Release	
				I ¹³¹ Leach, A _s	I ¹³¹ Invent, A _s *	Te ¹³² Leach, A	Te ¹³² Invent, A _s *	I ¹³¹	Te ¹³²
132	1760 (2820)	Helium	Intact	11.1	277	-	-	4.02	-
153	390 (1440)	Vacuum	Intact	0.075	87.5	0.081	177	0.086	0.046
154	890 (1800)	Vacuum	Intact	2.98	254	5.77	430	1.17	1.34
155	1870 (2920)	Vacuum	Intact	67.1	640	52.7	1100	10.5	4.8
146	1850 (2900)	Air	Fragmented	189	292	127	780	64.5	16.0
156	1970 (3000)	H ₂ O	Fragmented	503	636	311	906	79	32.6

Larger fractional releases of iodine-131 than tellurium-132 were observed in most of the tests. This difference is believed to indicate that the release of the tellurium-132 precursors (tin-132 and antimony-132) is somewhat less than that of the major precursors for iodine-131 (tellurium-131 and tellurium-131*). The release of both nuclides was considerably greater from the back-reacted specimens, and the highest release was obtained in water tests on this type material.

3. Water-immersion Testing

In the previous TREAT status reports,^(1,2) fragmentation of the fully carbided fuel in sea-water was reported at a heat-generation level as low as 380 cal/gm (900°C). Two specimens of the beaded-type graphite fuel were tested in a water environment in the tests reported here.

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The first test was performed on an as-manufactured, NbC-lined, hexagonal specimen immersed in sea-water (CEN-131). The sample was not damaged at a heat-generation level of 1650 cal/gm, except that some surface interaction had occurred, as shown in Figure 90. The results of a mass spectrographic analysis of the gas sample taken remotely 25 sec after the transient are presented in Table VII, compared with the results from two fully-carbided fuel samples tested in water at somewhat lower heat-generation levels.

Figure 90
PHOTOGRAPH OF FUEL SAMPLE FROM
WATER IMMERSION TEST CEN-131

108-5898

Table VII

MASS SPECTROGRAPHIC ANALYSIS RESULTS OF GAS SAMPLES
FROM WATER IMMERSION TESTS

	Beaded Types		Dispersed Type	
	As-manufactured	22% B.R. ^d	Fully Carbided ^a	Air-stored ^d
Test No.	131	156	83, 84	96
cal/gm	1650	1970	399, 1370	3040
Period, msec	44	41.6	141, 74	40
d _{SV} , mils	Intact	49	86, 28	31
Gas composition m/o, CO	37.4	37.2	20, 42.7	38.5
CO ₂	6.0	4.8	0.5, 5.4	7.8
Hydrocarbons	0.4	0.9	84, 2.6	0.6
H ₂	46.4	46.7	71, 48.3	51.3
% reaction of sample ^c				
Carbon with H ₂ O	11.8	15.7	0.5, 8.6	15.3

^a5% UO₂.

^b20% UO₂.

^cBased on amount of carbon formed in gas phase.

^dBack-reacted

The second test was conducted in distilled water at a heat-generation level of 1970 cal/gm on an NbC-lined, hexagonal sample (CEN-156) that had been back-reacted to convert 22% of its UC_2 content to UO_2 . The sample fragmented, as shown in Figure 91. The result of a particle-distribution study is included, which indicates a mean particle diameter of 49 mils. Mass spectrographic data obtained on a gas sample taken at the same time after transient as in CEN-131 are shown in Table VII, compared with results from an air-stored (20% UO_2), dispersed-type fuel specimen tested in CEN-96.

The composition of the gas phase in all the tests is similar, indicating a high-temperature interaction of carbon with the water environment (i.e., $\text{H}_2\text{O} + \text{C} \rightleftharpoons \text{CO} + \text{H}_2$). Since the amount of reaction observed in the unfragmented specimen is the same order of magnitude as that in the fragmented specimen, most of the interaction apparently occurs at the sample surfaces during the early portion of the transients.

E. Fuel Bead Tests

Two tests on fuel beads were performed: CEN T-7, a photographic test; and CEN-159, a thin-walled autoclave test.

The purpose of CEN T-7 was to record photographically the effect of three transient energy levels (150, 240 and 325 MW-sec) on five batches of fuel beads of different sizes. The motion-picture record indicated that all five of the bead fractions exhibited bead ejection to varying extents at the lowest integrated power, 150 MW-sec. At this level, the smallest beads (96 μ average diameter) reacted most violently, and considerably less activity was noted in the largest-diameter beads (193 μ). As the integrated power was increased, bead activity in the form of "ejection" or "popcorn effect" increased in all size fractions. During post-test examination of the graphite tray holder, the fuel beads were found to have melted or been ejected for the most part, as shown in Figure 92.

The thin-walled autoclave test, CEN-159, was performed with the most highly loaded (76.29 w/o uranium), largest-diameter, free beads available. As a result of the test, the beads were found to have been completely ejected from the graphite sample holder. The ejected beads however, appeared to have melted and fused on the surface of hardware in the immediate vicinity of the test cavity, as shown in Figure 93. Approximately 76 mg of uranium were found on the surface of hardware located 8 in. away from the bead samples during the test. This represents approximately 7% of the fuel originally present in the sample.

Figure 91

FRAGMENTED SAMPLE FROM WATER IMMERSION TEST ON
22% BACK-REACTED FUEL, CEN-156

A. 1970 cal/gm

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B. Particle Distribution
from CEN-156

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Figure 92

PHOTOGRAPH OF GRAPHITE TRAYS THAT ORIGINALLY CONTAINED
VARIOUS FUEL BEADS IN PHOTOGRAPHIC TEST CEN T-7

Screen fraction	-210, +177	-177, +149	-149, +125	-126, +105	-105, +88
w/o U	72.1%	66.8%	59.7%	49.6%	36.9%
108-5815					

Figure 93

FUEL BEAD TEST CEN-159

A. Sample holder after test, with
residue of melted fuel beads

108-6583

B. Top and bottom sample holders
after test. Tracks of ejected
fuel beads on the surfaces

108-6580

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IV. DISCUSSION AND SUMMARY

The pyrolytic, carbon-coated bead material exhibited an amazing resistance to breakup during transient nuclear heating. The fuel suffered structural damage at various energy levels dependent on its geometry, manufacture, and constraint. The gross damage is classified in two ways. The term "fragmentation" describes the uniform reduction of the entire sample to relatively small particles, while "cracking" describes the damage that was observed to originate as cracks in the webbing that in some cases caused partial breakup of the fuel sample. In light of these definitions, the behavior of the fuel materials is summarized below.

Table VIII lists the lowest energies at which the two phenomena were observed for the various types and geometries of materials tested. Data are included from the previous status report⁽²⁾ on dispersion-type fuel. The table indicates that the hexagonal, beaded-type, as-manufactured fuel remained relatively intact up to 5580 cal/gm ($>3200^{\circ}\text{C}$); however, cracks were observed at an energy level of 2660 cal/gm ($>3200^{\circ}\text{C}$). The same crack-damage was observed at much lower energy levels in the cylindrical specimens. This occurred in transients in which the fuel attained temperatures of 2200 to 2400°C . However, the hexagonal fuel withstood a transient energy level of 2490 cal/gm (3200°C) in CEN T-9 without any apparent crack formation. Evidently, the geometry and manufacture have a considerable effect on the behavior of these samples. This is also evident with respect to fragmentation, since a cylindrical specimen (CEN-157) fragmented at a lower energy of 4360 cal/gm ($>3200^{\circ}\text{C}$) while true fragmentation, as defined here, was not observed in the as-manufactured hexagonal samples at the highest energy level of the test.

Table VIII

LOWEST ENERGY OF OBSERVATION OF FRAGMENTATION

Type of Fuel	Fragmentation	Cracks
1. Hexagonal-beaded, as-manufactured	None to 5580 cal/gm ($>3200^{\circ}\text{C}$)	2660 cal/gm ($>3200^{\circ}\text{C}$)
2. Cylindrical-beaded, cut from as-manufactured hexagon	Only tested up to 2170 cal/gm (3100°C) none observed	1500 cal/gm (2210°C)
3. Cylindrical-beaded, extruded as cylinder constrained	4360 cal/gm ($>3200^{\circ}\text{C}$)	1500 cal/gm (2210°C)
4. Cylindrical dispersion, as-manufactured (fully carbided)	2900 cal/gm (2900°C)	Not observed
5. Hexagonal-beaded, back-reacted (22% UO_2)	1850 cal/gm (2900°C)	Not observed
6. Cylindrical dispersion, back-reacted (20% UO_2)	1200 cal/gm (1900°C)	Not observed

The data on back-reacted fuel indicate that the formation of UO_2 in the graphite matrix and the destruction of the pyrocarbon bead coatings promote fragmentation.

While the back-reacted, beaded-type fuel was not tested at energy levels as low as those for the back-reacted, cylindrical dispersed type, the particles from the fragmented beaded-type fuel (46 mils) were coarser than those from the dispersed-type fuel (23 mils). This indicates that the dispersal of UO_2 in the graphite matrix is more localized in the beaded-type fuel; i.e., the fragmentation threshold for the back-reacted beaded type is believed to be higher than the 1200 cal/gm observed for the back-reacted dispersed type.

The fragmentation threshold in water for the fully-carbided, dispersed-type fuel had previously been observed to be at about 380 cal/gm (900°C). The hexagonal as-manufactured, beaded-type fuel did not fragment when tested in water at 1650 cal/gm (2750°C).

Pretreatment of the hexagonal, beaded-type fuel with fluorine causes partial fragmentation and breakup. The lowest energy at which this occurred was 670 cal/gm (1420°C). Thus fragmentation occurred in these specimens only in those portions of the fuel in which fluorine resided. On these tests, the fluorine resided in the vicinity of the coolant channels and the flat surfaces.

Carbon vaporization occurred in these tests at an energy level as low as 1730 cal/gm (2800°C). The vaporized carbon typically redeposited on adjacent carbon hardware and/or sample surfaces in the form of pyrocarbon. Accompanying weight losses as high as 20% were observed at peak sample temperatures greater than 3200°C . Anisotropic dimensional changes were observed in sample tests at energies as low as 1600 cal/gm (2700°C). Radial growth was two to three times that of the longitudinal growth.

Uranium diffusion was noted in the pyrocarbon coatings of the fuel beads in samples tested by metallography at energy levels as low as 1140 cal/gm (2260°C). This diffusion preceded damage to the pyrocarbon coatings and the migration of uranium into the graphite matrix. Migration of uranium was noted in the sample tested in CEN-127 at 1320 cal/gm (2400°C) by EPM measurements. The first definite metallographic indication of damage to the pyrocarbon coatings was noted in CEN T-16 tested at 1570 cal/gm (2660°C). Under transient conditions, bead damage and uranium migration are greatly increased when the melting point of uranium dicarbide is exceeded (2475°C).

The NbC lining was observed to have partly melted on a sample tested at an energy level as low as 1730 cal/gm (2800°C). Samples of melted lining were found to contain uranium and carbon; i.e., 2-38 w/o uranium was found by EPM studies, and carbon was noted by metallographic techniques.

The release of iodine and tellurium from the as-manufactured, beaded-type fuel under transient conditions depended on the release of high-inventory precursors formed during the high-temperature portion of the transients. In this respect, the release of iodine-131 (tellurium-131, tin-131, and antimony-131 precursor) varied from about 0.1% at 390 cal/gm (1440°C) to about 10% at 1870 cal/gm (2920°C). The release of tellurium-132 (tin-132, antimony-132 precursor) varied from about 0.05% at 390 cal/gm (1440°C) to about 5% at 1870 cal/gm (2920°C). The results indicate that tellurium and iodine, as species, may be released in greater amounts than are tin and antimony, as species, for the same transient conditions. The release of fission products from back-reacted fragmented samples is five to ten times greater than from intact as-manufactured samples at comparable energy levels. Xenon-133 release data are comparable with those of iodine-131 for intact specimens. While there are some indications of higher xenon releases from samples tested in water and from fragmented samples, the xenon data are not conclusive because of possible readsorption effects.

V. CONCLUDING REMARKS

The TREAT program was primarily oriented toward discovery of destruct conditions by nuclear transients. The results indicate that this is not feasible for the beaded-type fuel at reactor periods of the order of 40 msec. However, the cracking phenomenon noted may be an indication that destruct can be accomplished at high energies on a shorter reactor period.

As a consequence of these tests, some valuable information was obtained pertaining to the stability of the Nerva beaded-type fuel, which indicates that the material should be extremely stable under steady-state, reactor operating conditions. For example, the data indicate that Nerva fuel can be expected to be structurally stable at operating temperature of 3000°C, but carbon vaporization and NbC lining melting can be expected at this temperature. In addition, dimensional changes and bead damage were evident at temperatures of 2700 and 2400-2600°C, respectively. The dimensional change may not be significant under steady-state conditions since some growth is normally expected in reactor core design. For the present fuel, it would appear that the most severe limitation on the temperature of operation results from fuel-bead damage. If further stabilization

of the fuel-bead system is possible, then it can be concluded from the TREAT data that core operation could be extended to a temperature near 3000°C.

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APPENDIX
DATA SUMMARY TABLES

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CEN No	Sample Description							Test Environment ⁽²⁾	Reactor Data			Post-Test Data					
	Batch	Shape	Lining	Dimensions, in.	Weight, gm	w/o (1)	State		MW-sec	Period, msec	cal/gm	Peak Sample Temp, °C ⁽³⁾			Weight Loss, %	Lining Melt	Structural Integrity
												Center	Apex	Optical			
104-1	2-2721-1	Round	No	3/4 x 1/2 OD	3,4710	20.0	As-mfg.	A, C, 20 psia, He	151	76	510	995	-	-	-	-	-
-2	"	"	"	"	"	"	"	"	215	"	730	1400	-	-	-	-	-
-3	"	"	"	"	"	"	"	"	325	"	1110	1900	-	-	-	-	-
-4	"	"	"	"	"	"	"	"	493	"	1680	2290	-	-	-	-	-
-5	"	"	"	"	"	"	"	"	815	42	2780	(~3000)	-	-	-	-	Intact, very friable
105	2-2721-2	"	"	"	3,2985	19.2	"	"	715	"	2340	(~2900)	-	-	-	-	PY carbon deposit
106	2-2721-3	"	"	"	3,3195	19.5	"	"	452	58	1500	(2210)	-	-	-	-	Intact surface cracks and ends eroded
107	2-2721-4	"	"	"	3,3732	19.4	"	A, C, vacuum	700	42	2320	(~2850)	-	-	-	-	Intact
108	2-2721-5	"	"	"	3,4145	19.6	"	A, NC, 20 psia, He	588 ⁽⁴⁾	450	1960	2400	-	-	-	-	"
T-3-1	2-2721-6	"	"	"	3,3833	19.9	"	P, NC, He flow	235	60	1330	2300	-	-	-	-	"
-2	"	"	"	"	"	"	"	"	154	"	870	1630	-	-	-	-	Intact PY carbon deposits
-3	"	"	"	"	"	"	"	"	410	48	2320	(3150)	-	-	0.14	-	-
T-4-1	2-2721-13	"	"	"	3,3010	20.5	"	P, C, He flow	160	58	930	1520	-	-	-	-	-
-2	"	"	"	"	"	"	"	"	635	45	3700	(>3200)	-	-	20.1	-	Intact, friable
T-5A	2-2721-8	"	"	"	3,3373	21.8	"	P, NC, He flow	640	"	3970	"	-	-	-	-	Cracked and broken
-5B	2-2721-9	"	"	"	3,4105	20.1	"	"	"	"	3660	"	-	-	-	-	"
T-6A	2-2721-10	"	"	0.500 x 3/4 L	3,2604	20.5	"	P, C, He flowing	630	"	3670	"	-	-	17.1	-	Intact, friable
-6B	2-2721-11	"	"	"	3,3456	19.8	"	"	"	"	3550	"	-	-	16.7	-	"
T-7A-1	2-2766-1	PYC beads	"	193μ diam	0,379	76.3	"	P, N, C, He flowing	150	69	1480	-	-	-	-	-	Melted and fused
-2	"	"	-	"	"	"	"	"	240	77	2370	-	-	-	-	-	"
-3	"	"	-	"	"	"	"	"	325	"	3220	-	-	-	38.5	-	"
T-7B-1	2-2766-2	"	-	163μ diam	0,410	71.4	"	"	150	69	1940	-	-	-	-	-	"
-2	"	"	-	"	"	"	"	"	240	77	3100	-	-	-	-	-	"
-3	"	"	-	"	"	"	"	"	325	"	4200	-	-	-	41.5	-	"
T-7C-1	2-2766-3	"	-	137μ diam	0,451	69.3	"	"	150	69	2010	-	-	-	-	-	"
-2	"	"	-	"	"	"	"	"	240	77	3210	-	-	-	-	-	"
-3	"	"	-	"	"	"	"	"	325	"	4350	-	-	-	36.1	-	"
T-7D-1	2-2766-4	"	-	115μ diam	0,545	68.5	"	"	150	69	2050	-	-	-	-	-	"
-2	"	"	-	"	"	"	"	"	240	77	3270	-	-	-	-	-	"
-3	"	"	-	"	"	"	"	"	325	"	4430	-	-	-	48.6	-	"
T-7E-1	2-2766-5	"	-	96μ diam	0,730	64.6	"	"	150	69	2220	-	-	-	-	-	"
-2	"	"	-	"	"	"	"	"	240	77	3550	-	-	-	-	-	"
-3	"	"	-	"	"	"	"	"	325	"	4810	-	-	-	52.3	-	"
T-8	2-2764	Hex, 19 holes	Yes	0.747 A.F x 0.441 L	5,7720	15.5	"	P, NC, He flowing	625	50	2530	(3200)	-	-	6.0	Yes	Void formation in webbing
T-9	2-2765	Hex, 19 holes	"	0.752 x 491 L	6,3410	"	"	"	616	"	2490	(3200)	-	-	7.7	"	Intact, void formation in webbing
126	2-2771	Round, 7 holes	No	0.499 OD x 0.775 L	4,1028	"	"	A, NC 20 psia He	430	59	1130	(2140)	-	-	0.18	-	Intact
127	"	"	"	0.495 OD x 0.739 L	3,7143	"	"	"	500	50	1320	(2400)	-	-	0.73	-	Large, longitudinal cracks in webbing

CEN No.	Sample Description							Test Environment ⁽²⁾	Reactor Data			Post-Test Data					
	Batch	Shape	Lining	Dimensions, in.	Weight, gm	w/o U(1)	State		MW-sec	Period, msec	cal/gm	Peak Sample Temp, °C ⁽³⁾			Weight Loss, %	Lining Melt	Structural Integrity
												Center	Apex	Optical			
128	2-2771	Round 7 holes	No	0.499 OD x 0.774 L	3.4223	15.5	As mfg.	A, NC, 20 psia He	720	42	1900	(2940)	-	-	-	-	Cracked into three pieces at webbing
129	"	"	"	0.500 OD x 0.740 L	3.8003	"	"	"	820	40	2170	(3100)	-	-	7.45	-	Friable, small, longitudinal crack in webbing
130	"	1/3 round	"	0.275 OD x 0.300 L Irregular	0.5007	"	"	"	815	43	2150	(3080)	-	-	Nil	-	Intact
131	2-2765	Hex, 19 holes	Yes	0.750 x 0.306 L	3.9680	"	"	A ² NC, 20 psia He 605 C, H ₂ O	790	44	1650	(2750)	-	-	-	Disintegrated	Intact, surface eroded
132	"	"	"	0.750 x 0.310 L	4.0315	"	"	A ² , NC, 20 psia He	845	40	1760	(2820)	-	-	-	Yes	Intact, PY carbon deposits
133	"	"	"	0.752 x 1.526 L	20.1910	"	"	LC, NC, 20 psia He	388	44	800	(1640)	-	-	Nil	No	Intact
T-10	2-2767	"	"	0.755 x 0.470 L	6.855	16.6	"	P, NC, He flowing	635	48	2750	(>3200)	-	-	-	"	Fragmented
139	2-2765	"	"	0.752 x 1.5 L	20.2550	15.5	"	LC, NC, 5 psia He	840	41	1730	(2800)	1160 ⁽⁵⁾	-	1.53	Partial melt	Intact
146	2-2767	"	"	0.75 x 0.30 L	4.329	16.6	"	A ² , NC, 14.6 psia air	828	40	1850	(2900)	-	-	-	No	Fragmented
147	"	"	"	0.75 x 0.32 L	4.564	"	"	A ² , NC, 20 psia He 60μ H ₂ O	830	44	1850	"	-	-	-	"	"
148-1	2-2802	"	"	0.752 x 1.5 L	21.211	15.9	"	LC, NC, 20 psia He	600 ⁽⁴⁾	600	1270	1760	1760	-	-	"	Intact
-2	"	"	"	"	"	"	"	"	315	86.7	665	1690	1590	-	-	"	"
-3	"	"	"	"	"	"	"	"	490	57	1035	2380	2200	-	0.04%	"	"
149	"	"	"	0.754 x 0.53 L	6.4006	15.5	Fluorine-treated	A, NC, vacuum	275	59	670	(1420)	-	-	-	"	Five large fragments, lining flaked off
150	"	"	"	0.753 x 0.52 L	6.4385	"	"	"	417	42	1010	(1980)	-	-	-	"	Four large fragments, lining flaked off
151	"	"	"	0.755 x 0.52 L	6.1861	"	"	"	820	42	1990	(3000)	-	-	-	Some melting	Four large fragments, very friable and porous
T-11-1	2-2806	"	"	0.752 x 0.742 L	10.4314	15.8	As mfg.	P, NC, He flowing	247	76	1020	1975	-	1750	-	-	-
-2	"	"	"	"	"	"	"	"	304	75	1250	2230	-	2200	-	-	-
-3	"	"	"	"	"	"	"	"	496	56	2040	(3030)	-	3130	-	-	-
-4	"	"	"	"	"	"	"	"	491	57	2020	2950	-	-	-	-	-
-5	"	"	"	"	"	"	"	"	544	51	2240	(3120)	-	3100-3200	2.8	Yes	PY carbon deposits
T-12	"	"	"	0.752 x 0.744 L	10.3132	16.1	"	"	491	54	2060	(3040)	-	3020	-	"	Intact Zircaloy melted to surface
T-13	"	"	"	0.752 x 0.752 L	10.6585	14.8	"	"	295	88	1140	2300	-	2100	Nil	No	Intact PY carbon deposits
T-14	"	"	"	0.752 x 0.742 L	10.3720	15.8	"	"	418	61	1720	(2800)	-	2710	"	"	Intact
152-1	"	"	"	0.752 x 0.326 L	4.4685	15.5	"	A ² , NC, vacuum	161	62	335	830	-	-	-	"	"
-2	"	"	"	"	"	"	"	"	255	61.9	530	1180	-	-	-	"	-
-3	"	"	"	"	"	"	"	"	374	61	780	1660	-	-	-	"	-
-4	"	"	"	"	"	"	"	"	491	60	1040	2090	-	-	0.2	"	-
153	"	"	"	0.752 x 0.315 L	4.3631	15.6	"	"	186	102	390	(1440)	-	-	Nil	"	Intact
154	"	"	"	0.752 x 0.329 L	4.5598	"	"	"	426	67.7	890	(1800)	-	-	0.1	"	"
155	"	"	"	0.752 x 0.333 L	4.5332	15.7	"	"	885	40	1870	(2920)	-	-	-	Yes	Intact PY carbon deposits
156	2-2767	"	"	"	4.8705	16.6	"	A ² , C, 20 pisa He, 60 cc H ₂ O	881	41.6	1970	(3000)	-	-	-	No	Fragmented

CEN No.	Sample Description							Test Environment ⁽²⁾	Reactor Data			Post-Test Data					
	Batch	Shape	Lining	Dimensions, in.	Weight, gm	w/o U(1)	State		MW-sec	Period, msec	cal/gm	Peak Sample Temp, °C ⁽³⁾			Weight Loss, %	Lining Melt	Structural Integrity
												Center	Apex	Optical			
T-15-1	2-2805	Hex, 19 holes	Yes	0.752 x 0.747 L	10.3834	16.0	As-mfg.	P, NC, He flowing	284	84	1190	2250	1950	2000	-	-	-
-2	"	"	"	"	"	"	"	"	307	76	1280	2350	-	2200	Nil	No	Intact
T-16	"	"	"	0.752 x 0.746 L	10.4726	15.8	"	"	380	70	1570	(2670)	-	2400	"	"	"
T-17	"	"	"	0.75 x 0.743 L	10.2600	16.2	"	"	630	48	2660	(>3200)	-	2400	18.1	Yes	Intact PY carbon deposits, cracks in webbing
T-18	"	"	"	0.75 x 0.738 L	10.1721	"	"	"	510	52	2160	(3100)	-	-	4.0	"	Intact PY carbon deposits
157	2-2820	Round	No	0.5 OD x 0.488 L	3.196	27.5	"	A, C, 20 psia He	929	39	4360	(>3200)	-	-	-	-	Partially fragmented
158	2-2800	Hex, 19 holes	"	0.782 x 0.745 L	11.0600	19.8	"	A ¹ , NC, evacuated to 5μ Hg	941	"	5580	"	-	-	-	-	Intact, very porous and friable, pronounced cracks in webbing
159	2-2766-1	Beads	-	96μ D	1.400	76.29	"	A ¹ , NC, vacuum	932	"	10,580	"	-	-	-	-	Completely dispersed
160	2-2819	Round	No	0.250 OD x 0.125 L (2 pellets)	0.1717 0.1790 (2 pellets)	20	Preirradiated	A ² , NC, vacuum	177	60	540	(1200)	-	-	-	-	Intact
161	"	"	"	"	0.1768 0.1773 (2 pellets)	20	"	"	451	50	1380	(2460)	-	-	-	-	"
162	"	"	"	"	0.1806 0.1816 (2 pellets)	20	"	"	806	40	2470	(3200)	-	-	-	-	Intact, carbon deposits

(1) By analysis.

(2) A = Standard autoclave

A¹ = Thin-wall autoclaveA² = Small capsule inside standard autoclave, 20-psia helium in autoclave

P = Photographic capsule

C = Constrained

NC = Not constrained

LC = Long-channel autoclave.

(3) Indicated temperatures given where possible. Estimated peak center temperatures are given in parentheses.

(4) "Flat-top" transient.

(5) Chromel-Alumel couple located on OD of insulating pyrolytic graphite cylinder at sample position