Argonne National Laboratory

**THE MANUFACTURE OF THE
GRAPHITE-URANIA FUEL MATRIX
FOR TREAT**

by

J. H. Handwerk and R. C. Lied

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THE MANUFACTURE OF THE GRAPHITE-URANIA FUEL MATRIX FOR TREAT

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ABSTRACT

A study⁽¹⁾ of the methods of dispersing uranium in graphite indicated that the most feasible method of forming a dispersion would be to fabricate a mixture of graphite, carbon and U_3O_8 . A similar procedure for manufacturing graphite had, through independent research, been developed by Great Lakes Carbon Corporation, and this procedure was found to be readily adaptable for forming the graphite-urania material for the TREAT fuel. This fabrication was, therefore, undertaken as a joint enterprise of Argonne National Laboratory and Great Lakes Carbon Corporation.

INTRODUCTION

The Transient Reactor Test (TREAT) was designed to produce short term bursts of thermal neutrons of sufficient intensity to permit melt down studies to be made of fast reactor fuels. To generate this large thermal neutron flux with the greatest margin of safety, a nonmetallic fuel system operating on a low loading was required. In the light of existing technology a dispersion of highly enriched uranium oxide in a matrix of graphite appeared to be the most suitable type of fuel.

In this type of fuel the graphite, in addition to being a moderator, would serve as a heat sink to dissipate the heat generated by the particles of uranium oxide. On heating, the graphite would also raise the energy of the thermal neutrons, and increase the probability of the neutrons leaking from the reactor. This would result in the reactor having a rather large negative temperature coefficient.

The reactor was estimated to have an average operating temperature of 260°C , and it was further estimated that hot spots in the fuel could be as high as 400°C . As the reactor was to be air-cooled, it was necessary to protect the graphite-urania fuel from oxidation. Therefore, the graphite-urania was encased in a cladding of Zircaloy-3, which, in addition to protecting the graphite from oxidation, also served to contain fission products. However, since this report is concerned only with the manufacture of the fuel matrix, a description of the fuel-can fabrication and the loading of the fuel⁽²⁾ is not included.

PRIOR DEVELOPMENT

Graphite-urania mixtures have been formed by two methods primarily. The first method is to impregnate a graphite shape with a solution containing a soluble uranium compound, while the second is to fabricate a mixture of uranium dioxide and carbon or graphite powder. These methods of forming a dispersion of uranium in graphite were evaluated⁽¹⁾ and the results of these investigations were summarized as follows:

1. Uniform dispersions of uranium in graphite can be prepared by impregnating the graphite with a solution containing a soluble uranium compound. However, it would be difficult to maintain a uniform uranium dispersion throughout a large number of graphite pieces due to the variation of pore size and structure in the graphite.
2. Uranium dioxide in finely divided form has a strong tendency to agglomerate, and it is difficult to prepare and fabricate mixtures of UO_2 and graphite or carbon powder without having some rather large-size agglomerates of UO_2 . U_3O_8 appears to have a more discrete particle size. Thus mixtures of U_3O_8 and graphite or carbon powder are more easily prepared without agglomerates of the uranium oxide.
3. U_3O_8 can be mixed with carbon powder, graphite powder or a combination of these materials. These mixtures can be bonded with a thermosetting resin, and these mixtures can be molded into ware having fairly high densities and fairly close dimensional tolerances. Thermoplastic resins are also suitable for bonding the mixtures of carbon and graphite, but high densities were more difficult to achieve and dimensional tolerances were harder to hold when these resins were used.
4. Both the thermosetting and the thermoplastic resin-bonded ware was found to have good mechanical strength after the resin was dissociated by heating to temperatures of 900 to 1000°C.
5. Ware formed from a mixture of U_3O_8 , carbon powder and a resin consisted of a uranium oxide dispersion in carbon, following the heat treatment to dissociate the resin. This ware required an additional heat treatment at a temperature of 2300°C or above to graphitize the carbon.
6. Ware formed from a mixture of U_3O_8 , graphite powder, and a resin consisted essentially of a uranium oxide-graphite mixture bonded by a carbon matrix, following the heat treatment to dissociate the resin. This ware was found to have properties intermediate between carbon and graphite, and appeared to be a suitable fuel material without the need for graphitizing the carbon matrix. Thus the potential hazard of uranium volatilization at high temperatures would be eliminated.

From this study it appeared feasible to prepare a uniform dispersion of uranium oxide in graphite by the fabrication of a mixture of U_3O_8 and graphite powder. A direct process such as this for fabricating graphite had been developed by Great Lakes Carbon Corporation through independent research. This process for manufacturing graphite appeared to be readily adaptable for making a U_3O_8 -graphite dispersion.

In order to evaluate this process, samples, in which 1 weight percent U_3O_8 was dispersed in each of two mixtures formulated by Great Lakes Carbon Corporation, were manufactured by Great Lakes Carbon Corporation (GLC).⁽³⁾ The first mixture was a dispersion of 1 w/o U_3O_8 in a calcined coke-base formulation. Ware formed from this mixture was baked to dissociate the resin binder, and samples were graphitized at each of the following temperatures: 1800, 2150, and 2500°C. These coke-base samples were found to have an average density of 1.74 g/cc when graphitized at 1800°C, 1.75 g/cc when graphitized at 2150°C, and 1.75 g/cc when graphitized at 2500°C. These specimens were all mechanically sound, and no agglomerates of uranium oxide were detectable by radiography.

The second mixture was a dispersion of 1 w/o U_3O_8 in a graphite powder-base formulation. Ware formed from this mixture was baked to dissociate the resin binder, but was not graphitized. These graphite-base samples were also found to be mechanically strong and free of agglomerates of uranium oxide, as indicated by radiographs of the samples. These specimens were found to have densities which ranged from 1.68 to 1.73 g/cc, with an average density of 1.70 g/cc.

Examination of the graphite-urania mixtures fabricated by Great Lakes Carbon Corporation indicated that either of the two methods would produce a suitable fuel material. However, the high temperature graphitization necessary for the coke-base formulation could conceivably result in the volatilization of some urania from the graphite-urania mixtures. Therefore, the graphite powder-base formulation was selected for the fuel matrix. This manufacturing process as developed by Great Lakes Carbon Corporation was readily adaptable to the fabrication of a graphite-urania dispersion, and, though thoroughly familiar with the fabrication of the graphite, they were unfamiliar with certain problems involved in processing enriched uranium oxide, such as accountability procedures, criticality, and health hazards. Therefore, the fabrication of the TREAT fuel was undertaken as a joint enterprise of Argonne National Laboratory and Great Lakes Carbon Corporation.

MATRIX FABRICATION

A TREAT fuel rod assembly is shown in Figure 1. The graphite-uranium matrix section is essentially four inches by four inches by four feet. To reduce development time at Great Lakes Carbon Corporation

Figure 1. TREAT Fuel Element

FUEL MATERIAL: URANIUM OXIDE IN GRAPHITE

and to expedite the fuel fabrication, fuel specifications (Table 1) were generous. These specifications were based on the production experience of Great Lakes Carbon Corporation as well as the results of the evaluation study,⁽²⁾ and were designed to minimize the machining of the formed fuel.

Table 1

SPECIFICATIONS FOR THE GRAPHITE-URANIA TREAT FUEL MATRIX

Minimum Apparent Density, g/cc	1.72
Cross Section, in.	3.800 ^{+0.020} -0.000
Length of Blocks, in.	8 ± 0.125
Boron Content, ppm	1
Iron Content, w/o	0.1
Uranium Content, w/o	0.248
Uranium Enrichment, w/o	93.1
Baked Stock (Not Graphitized)	
Dispersion: -325 mesh U ₃ O ₈ (44 microns)	
with no detectable agglomerates	
as indicated by radiographs.	

The four-foot length of the fuel matrix, for convenience, was divided into six sections, making the fuel block increments eight inches in length. The uranium-to-carbon atom ratio was established as approximately 1 to 10,000 and, based on the Great Lakes Carbon Corporation batch mixture, was calculated to be 0.248 w/o U₃O₈.

A portion of one building at Great Lakes Carbon Corporation research laboratory, located at Morton Grove, Illinois, was allocated to the TREAT project. In order to contain the enriched uranium oxide and to maintain an adequate, properly filtered flow of air for all operations, a polyethylene-sheeted room was constructed in this building. An exterior view of this room is shown in Figure 2. The entrance shown at the right was normally closed, and was used only to move the baking crucibles to and from this area. The entrance shown near the center was used to move batch materials to the area and as a personnel entrance. The hydraulic pump for the press was excluded from the fabrication room to minimize the possibility of contamination of this unit. The ventilation duct work is shown at the top of Figure 2. These ducts carried the exhaust air to the filters, shown in Figure 3, where it was exhausted through the building wall to the outside.

Figure 2. Exterior View of Fabricating Room

Figure 3. Filter Boxes and Exhaust Fan

The enriched U_3O_8 was weighed in batch quantities at ANL. These tared containers were transported to GLC and, while sealed, were reweighed. The remaining raw materials, graphite, carbon and resin, were weighed outside the fabrication area in tared containers (Figure 4).

Figure 4. Weighing of Raw Materials

The flow sheet of the manufacturing process is shown in Figure 5. A batch consisting of 17.5 kg of graphite powder, carbon and resin was charged into the mixer and hot mixed for a short period of time (see Figure 6). The mixer was shut off, the U_3O_8 (approximately 43 grams) was added (Figure 7), and the batch was mixed for 40 min. The batch was discharged into a stainless steel batch can by means of a bottom discharge door and cooled to solidify the resin. The mixed material was crushed in a hammermill and weighed into approximately five increments of 3400 grams each on a balance (Figure 8). Each increment of material, sufficient for one fuel block, was discharged from the balance pan into a small batch can. These cans with the weighed material were placed in a preheated oven ($100^{\circ}C$) for one to two hours before pressing.

Figure 5. Flow Sheet of Block Fabrication

Figure 6. Covered Muller-type Mixer

Figure 7. Addition of U_3O_8 to Mixer

Figure 8
Balance for Weighing Material
for Fuel Blocks

One can at a time was transferred to the press, and the material emptied from the can into the press cavity (Figure 9). The graphite mixture was pressed at 100°C under a pressure of 5000 psi. The molded block was ejected from the die cavity (Figure 10), deburred, weighed, and measured for length (Figure 11). After being numbered, the blocks were placed in stainless steel crucibles (Figure 12) and packed in graphite.

Figure 9. View of Press with Molding Die

Figure 10. Ejection of the Molded Fuel Block

These "green" blocks contained approximately 1.5 w/o hydrogen due to the pitchbinder and, surrounded with graphite packing, represented a better moderated mass than the final fuel element. Therefore, criticality considerations limited the amount of fuel charge in each container to 1000 pounds, and the containers were divided into quadrants by means of thermally black boron-steel dividers.

Two crucibles (Figure 13) were set side by side in the baking furnace, and the temperature was slowly raised to 900°C. During this operation the resin binder was dissociated to carbon, and the U_3O_8 was presumably reduced to UO_2 . The furnace was allowed to cool to room temperature and the crucibles were removed. This baking cycle required two weeks. After cooling to room temperature, the blocks were removed

Figure 11. Inspection of the Molded Fuel Block

Figure 12. Fuel Blocks Packed prior to Baking

Figure 13. Loading Crucibles in Baking Furnace

from the crucibles and were brushed to remove any adhering graphite packing material. The fuel blocks were gauged for length, weighed, inserted in polyethylene bags, and packed in shipping crates for removal to ANL. These shipping crates, shown in Figure 14, were designed to hold sixteen fuel blocks per crate and insured at least one foot of separation between each lot of fuel blocks.

Figure 14. Packed Fuel Blocks Ready for Shipment

INSPECTION AND EVALUATION OF FUEL BLOCKS

A. Radiographic Inspection

When the blocks were received at ANL a random sample was selected and radiographed. This sampling totaled 23% of the manufactured blocks and included at least one fuel block from each batch of material mixed. These radiographs revealed that 94.0% of the fuel blocks inspected were free of any visible defects or agglomerates. The radiographs of the remaining 6.0% of the blocks revealed some small spots or agglomerates, which were assumed to be small particles of uranium oxide. Of the radiographs which showed these agglomerates, 3.7% of the blocks inspected appeared to have one small agglomerate which was estimated to be less than 0.015 in. in size; 1.6% of the blocks had two of these small agglomerates; 0.1% had three small agglomerates, and the remaining 0.6% had four or more agglomerates of estimated sizes ranging from 0.015 to 0.125 in., as shown by a typical photograph in Figure 15.

Figure 15. Photo of Agglomerates in Fuel Block

Micro 25074

IX

The blocks in which the radiographs indicated three or more agglomerates were found to be manufactured from three consecutive batches. These batches were mixed at a time when the drive belt on the mixer was found to be worn and slipping. It was also found that, during the initial manufacturing runs, the U_3O_8 had been added to the top of the batch and during mixing some dust had a tendency to stick to the mixer lid. The mixer belt was replaced. As an added precaution the U_3O_8 was covered with the graphite mixture before mixing. The blocks from the three questionable batches were rejected.

B. Density and Chemical Analysis

Fifty blocks, picked at random, were measured and the bulk densities calculated. These densities were found to range from 1.71 to 1.76 g/cc, with the average density being 1.73 g/cc. The porosity of these blocks was found to range from 11.5 to 16.1%, with the average porosity being 13.5%. The apparent density (by water immersion) was found to range from 1.98 to 2.03 g/cc, with the average as 2.01 g/cc. Ninety-two of the fuel blocks were cored for the control rod elements, and portions of the cores were analyzed

for uranium and iron contents. The uranium was found to range from $0.205 \pm 0.004\%$ to $0.222 \pm 0.004\%$, with the average being $0.211 \pm 0.004\%$. In these samples the average iron content was found to be 0.0267% .

The raw materials (graphite flour, carbon and resin binder) were analyzed for boron content, and from these analyses the boron content of the manufactured blocks was calculated to be very close to 1 ppm. However, when portions of the above cores were analyzed, it was found that the fuel blocks had widely varying amounts of boron. These analyses indicated that the boron content exceeded the specified 1 ppm and that the fuel blocks actually averaged about 6 ppm boron. This increase in boron content was due to the unexpected diffusion of the boron from the borated steel divider plates in the baking crucibles.

C. Thermal Conductivity and Specific Heat

The thermal conductivity of the graphite-urania fuel was measured at Armour Research Foundation⁽⁴⁾ by a steady-state measurement of the radial heat flow and radial temperature drop in a vertical stack of disks of the fuel material. Radial heat flow through the annular disks was supplied by an electric heater centered in an axial hole of the stacked disks. In order to obtain high temperatures as well as to control the temperature gradient in the test specimens, the column of stacked disks was placed in an electrically heated furnace in which a helium atmosphere was maintained. The radial heat flow through a section of the stacked disks, 3 inches long, $1\frac{1}{2}$ inches above and below the vertical center of the column, was obtained from the measurement of the current and voltage drops along a 3-in. length of the axial heater adjacent to the test section of the stacked disks. The temperature gradient was measured by means of thermocouples inserted in two small vertical holes, one near the axial hole containing the heater, and the other near the outer edge of the disks. The thermal conductivity of the graphite-urania material was calculated under steady-state conditions from the power input, the radial distance of the inner and outer thermocouples from the axis of the disks, and the temperature difference between these points.

The thermal conductivity measurements, shown in Table 2, were made in the temperature range from 65 to 1066°C . After reaching the peak temperature of 1066°C , the samples were held at this temperature for a period of 36 hours, and the thermal conductivity again measured on the cooling cycle (Table 2). The samples were then heated to 1093°C and the thermal conductivity measured at this temperature. These data are plotted in Figure 16.

The thermal conductivity of the graphite-urania mixture was approximately 5% higher after the samples were held at 1066°C for 36 hours. This could be due to outgassing of the graphite-urania mixture, and perhaps to the decomposition of residual resin binder.

Table 2

THERMAL CONDUCTIVITY OF GRAPHITE-URANIA
MATERIAL FOR THE TREAT FUEL⁽⁴⁾

Mean Temperature, °C	Thermal Conductivity, cal/sec/ cm/°C x 10 ³	Mean Temperature, °C	Thermal Conductivity, cal/sec/ cm/°C x 10 ³
65	53.74	538	42.58
121	51.88	641	41.42
185	49.81	760	40.31
202	50.14	867	39.27
310	46.71	982	39.52
315	46.83	1048	39.81
460	43.61	1066	40.31

After heating for 36 hours at 1066°C:

927	41.34	321	48.98
763	42.58	93	54.57
610	44.44	1093	40.72
413	47.54

The enthalpy of the TREAT graphite-urania dispersion fuel was measured by both Armour Research Foundation⁽⁵⁾ and Battelle Memorial Institute. ⁽⁶⁾ The first determinations were made by Armour Research Foundation using a modified Parr adiabatic calorimeter over the temperature range from 197 to 986°C. The measured enthalpies are shown in Table 3 and are plotted in Figure 17. From these data the specific heat of the graphite-urania dispersion was calculated at various temperatures. These specific heats are shown in Table 4 and are plotted in Figure 18.

Table 3

MEASURED ENTHALPY OF GRAPHITE-URANIA
TREAT FUEL TO A DATUM OF 26.7°C⁽⁵⁾

Temperature, °C	Enthalpy, cal/gm	Temperature, °C	Enthalpy, cal/gm
197	39.7	606	186.3
260	58.4	651	205.2
312	76.0	718	234.6
381	97.1	802	270.4
424	112.0	829	284.5
481	136.5	913	318.8
562	169.3	986	355.5

Table 4

SPECIFIC HEAT OF GRAPHITE-URANIA TREAT FUEL (5)

Temperature, °C	Specific Heat, cal/gm/°C	Temperature, °C	Specific Heat, cal/gm/°C
204	0.304	649	0.412
316	0.331	760	0.439
427	0.358	871	0.466
538	0.385	982	0.493

Figure 18. Specific Heat of Graphite-Urania⁽⁵⁾

Using the modified Parr adiabatic calorimeter it was not possible to determine the enthalpy of the graphite-urania dispersion below 150°C. Therefore, the enthalpy of the dispersion was measured by Battelle Memorial Institute over the temperature range from 82.9 to 297.7°C. These measurements were made in an ice calorimeter similar to the one described by Ginnings and Corruccini.⁽⁷⁾ In this apparatus the heat from the specimen melts ice which is in equilibrium with water in a closed system; the resulting volume change is determined by mercury displacement. The ratio of

heat input to the mass of mercury displaced is a constant which is used as a calibration factor to obtain heat inputs to the calorimeter. There is no change in temperature in the calorimeter since all heat transfer occurs at the ice point.

The graphite-urania specimen was encapsulated and the annulus between the capsule and the specimen was filled with helium. The specimen was heated to various temperatures and dropped from each temperature into the ice calorimeter. The measured enthalpies for the various temperatures are shown in Table 5, and these data are plotted in Figure 19.

Table 5

Temperature, °C	Enthalpy, cal/gm	Temperature, °C	Enthalpy, cal/gm
82.9	14.88	166.3	36.13
89.0	16.32	214.0	48.97
90.8	16.53	214.1	49.39
96.4	17.45	216.3	50.66
144.6	30.84	282.8	69.76
145.0	29.69	286.2	70.13
145.3	30.61	299.7	75.27
152.5	32.35

From the slopes of the curve in Figure 19, the specific heats of the graphite-urania, shown in Table 6 and graphically in Figure 20, were calculated at interpolated temperatures.

Table 6

INSTANTANEOUS SPECIFIC HEATS AT INTERPOLATED TEMPERATURES
OF GRAPHITE-URANIA(6) AS CALCULATED FROM DATA IN FIGURE 19

Temperature, °C	Specific Heat, cal/gm/°C	Temperature, °C	Specific Heat, cal/gm/°C
0	0.137	176.7	0.279
37.8	0.175	204.4	0.286
65.5	0.203	232.2	0.291
92.2	0.229	260.0	0.294
121.1	0.253	298.9	0.297
148.9	0.269		

Figure 20. Specific Heat of TREAT Fuel
Specimen as Calculated from
Data as Shown in Figure 19

DIMENSIONAL TOLERANCES

Fifty fuel blocks were picked at random. These blocks were found to vary from $7\frac{7}{8}$ to $8\frac{1}{8}$ in. in length, and 45 of the fuel blocks were found to be within the cross-sectional tolerance of $3.800^{+0.020}_{-0.000}$ in. Three of the blocks were found to be within the $3.800^{+0.020}_{-0.000}$ in. on both ends; however, the center of these blocks was found to be from 0.001 to 0.003 in. under-size. The remaining two blocks were found to be from 0.002 to 0.004 in. undersize on one end. The blocks on a whole conformed quite well to the dimensional specifications as given in Table 1. However, the blocks in all cases were slightly rounded on the ends. This was an inherent defect due to the method of fabrication and was not serious enough to warrant squaring the end by machining off the rounded portion. During the gauging to length two blocks were rejected, one due to warping, and the second due to several small cracks which were visible on the surface of the block.

The six blocks used to form the fuel element shown in Figure 1 were set in a gauging fixture and gauged for overall length. In many instances six blocks were found to be of the correct length and no machining was required. However, as the blocks were in the majority of the cases oversize in length, the end of the sixth block was machined to the correct length. Blocks used in the special fuel elements, such as the thermocouple elements, in which several holes were drilled in the blocks, were machined on both ends to insure correct alignment of drilled holes.

PRODUCTION SUMMARY

A total of 2433 fuel blocks were fabricated by Great Lakes Carbon Corporation. The resumé of the accountability for the material involved is given in Appendix A. Of the 2433 blocks, 16 were defective after pressing. The latter were rejected by Great Lakes Carbon Corporation and sent to ANL in the "green" or pressed state. The remaining 2417 blocks were delivered to ANL. Fourteen of these blocks were rejected due to urania agglomerates, one due to warping, one due to small surface cracks, and one since it was a one-half block. Thus a total of 2400 fuel blocks were suitable for use in the reactor.

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The photographs used in the pictorial description were furnished by Great Lakes Carbon Corporation.

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APPENIDX "A"

MATERIAL ACCOUNTABILITY FOR THE MANUFACTURE OF
THE TREAT MATRIX

Batches Process 475 - Gross Weight 8,324,903.20 grams

Weight of Graphite Materials - 8,305,632.45 grams

Weight of U_3O_8 - 19,270.75 grams

	Gross Weight, gm	U_3O_8 , gm	Percent
Finished Fuel Blocks (2417 to ANL)	8,222,569.0	19,017.96	98.77
Blocks Not Baked - 16	54,180.7	133.04	0.65
2 Batches Not Properly Mixed - Scrap	31,388.0	81.14	0.38
Material Recovered in Cleanup	6,027.0	13.94	0.07
Total Accountable Material	8,314,164.7	19,246.08	
Unaccounted (In Filters & Kleenex) by Difference	10,738.5	24.67	0.13
TOTAL	8,324,903.2	19,270.75	100.00