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EFFECT OF NEUTRON IRRADIATION
ON UHTREX GRAPHITE AND CARBON

J. W. HELM

FEBRUARY, 1966

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EFFECT OF NEUTRON IRRADIATION
ON UHTREX GRAPHITE AND CARBON

By

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Ceramics and Graphite Research Section
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February, 1966

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EFFECT OF NEUTRON IRRADIATION ON UHTREX GRAPHITE AND CARBON

INTRODUCTION

The Ultra-High Temperature Reactor Experiment (UHTREX) now under construction at the Los Alamos Scientific Laboratory makes use of unclad refractory fuel elements in a helium-cooled, graphite-moderated reactor. The thermal power of the reactor will be 3 MW with a maximum coolant temperature of 2400 °F.⁽¹⁾ The reactor core is a hollow vertical cylinder of graphite, 70 in. OD x 23 in. ID x 39 in. high, serving as both structural material and moderator. Surrounding the core is the 16-in. thick reflector made up of 4 in. of graphite and 12 in. of dense ungraphitized carbon. Between the reflector and the carbon-steel pressure vessel is rigid, low-density carbon insulation. All of the core, as well as parts of the reflector and insulation, can be rotated about the cylindrical axis to allow for fuel charging and discharging. This movable portion is supported on a column of carbon.

It is well established that graphites and carbons undergo dimensional changes when exposed to neutron irradiation.^(2,3) Even though large dimensional changes in the graphite or carbons were not expected at the maximum predicted UHTREX lifetime exposure (5×10^{20} nvt, $E = 0.18$ MeV), a program for the irradiation of these materials was undertaken for several reasons. Because the UHTREX core graphite is manufactured from petroleum-coke filler which is graphitized prior to forming, it is substantially different from any graphite on which irradiation data has been obtained^(4,5,6) Furthermore, the maximum core temperature of 1650 °C is well beyond that temperature for which any irradiation data are available. Little information exists on the behavior of carbons under irradiation⁽³⁾ and there is none at the expected UHTREX temperatures. Consequently, a program was initiated to:

Determine the dimensional stability of the graphite and carbon materials used in UHTREX to neutron exposures equal to or greater than the expected lifetime exposure. Provide a direct comparison of the dimensional changes with CSF graphite on which a large body of information^(4,6) is currently available.

MATERIALS

The materials studied were the UHTREX core graphite (grade CGL), the reflector carbon (grade CGM), the support carbon (grade CGN), and an insulating or porous carbon (grade B260, designated PC). The insulating carbon to be used in UHTREX was not available when the study was made so grade B260 was used as a substitute. All these materials and the conventional nuclear graphite, CSF, used as a reference material are described in Table I.

TABLE I
MATERIAL DESCRIPTION

Material Designation	Block Number	Block Size, in.	Remarks	Approximate Service Conditions in UHTREX		
				Maximum Exposure, nvt x 10 ²⁰ E > 0.18 MeV	Temperature	
					°F	°C
CGL	CL-1	5 1/8 diam x 6	UHTREX core graphite, graphitized petroleum coke, maximum particle size, 0.032 in., coal tar-pitch binder, molded, baked at 800 °C, impregnated, graphitized at 2800 °C, very isotropic (CTE ₁ /CTE ₂ = 1.05 up to 1600 °C), density, 1.76 g/cm ³ .	5	2000 to 3000	1090 to 1650
CGM	CM 2	5 1/8 diam x 6	High density reflector carbon, petroleum coke filler, maximum particle size, 0.065 in., coal tar-pitch binder, molded, baked at 800 °C, impregnated, rebaked at 900 °C, very isotropic (CTE ₁ /CTE ₂ = 1.1 up to 1600 °C), density, 1.73 g/cm ³ .	2.5	1800 to 2000	980 to 1090
CGN	CN-2	5 1/8 diam x 6	Normal-density support column carbon, same as CGM except not impregnated or rebaked, very isotropic (CTE ₁ /CTE ₂ = 1.1 up to 1600 °C), density, 1.61 g/cm ³ .	2	1300 to 2000	900 to 1090
B260	PC	5 diam x 12	Insulating carbon, density, 0.59 g/cm ³ .	0.2 ^(a)	550 to 1800 ^(a)	290 to 980 ^(a)
CSF	222	4 x 4 x 50	Reactor grade, Cleves petroleum coke, conventional type, standard coal-tar-pitch, extruded, F purified, graphitized at 2800 °C, anisotropic (CTE ₁ /CTE ₂ = 2.4 at 400 °C), density, 1.66 g/cm ³ .			

(a) Conditions for insulating carbon used.

The sampling scheme for all the UHTREX materials is illustrated in Figure 1, and for the CSF graphite in Figure 2.

FIGURE 1
Sampling Scheme
UHTREX Materials

FIGURE 2

Sampling Scheme CSF Graphite

EXPERIMENTAL PROCEDURE

The samples were machined to a quarter-round shape for use in a standard noninstrumented capsule.⁽⁷⁾ The sample sections were removed from the original blocks by means of a power hack saw. Plastic jigs were used to hold four of these sections together while they were being turned on a lathe. Considerable breakage occurred during the machining of the extremely hard and

brittle CGM carbon, and the same problem was encountered (to a lesser degree) in preparing the CGN and porous carbon samples.

Since all materials were to be irradiated up to 1000 °C, and all but the porous carbon at higher temperatures, the samples were heat treated prior to encapsulation. This was required so that the capsule internal pressure would not build up excessively, that the heat-transfer medium in the capsule would not change with a resultant change in irradiation temperature, and that the samples would not oxidize while being irradiated.

The samples were heat treated for several hours to insure a low pressure and controlled release of the gases as the furnace temperature was increased (Table II). The samples were maintained at the maximum temperature for 1 hr before the furnace power was shut off. The effects of heat treatment are discussed in the following section.

TABLE II
HEAT-TREATMENT RUNS

<u>Run No.</u>	<u>Material</u>	<u>Maximum Temperature, °C</u>	<u>Maximum Pressure, Torr</u>	<u>Total Run Time, hr</u>
1	CGL	1700	4×10^{-4}	4
2	CGM	1700	4×10^{-4}	16
3	CGN	1700	4×10^{-4}	15
4	PC	1300	3×10^{-3}	16
5	Felt ^(a)	1600	3×10^{-3}	11

(a) Used as a thermal radiation barrier in the two highest temperature capsules.

Sample lengths for the CGL, CGN, and CSF materials were measured from end-to-end by a Bausch and Lomb DR-25 Optical

Gage (Type 33-14-23). The lengths of the CGM and PC materials were measured between two small holes by a Bausch and Lomb Bench Comparator (Type 33-12-11). The latter was required because the ends of the CGM and PC materials could not be polished to a satisfactorily uniform surface. The standard deviation of these measurements was 3×10^{-5} and 20×10^{-5} in., respectively.

The samples were weighed and then assembled into cylinders.⁽⁷⁾ Graphite felt was wrapped around the assembled cylinder to form a thermal radiation barrier in the two capsules to be operated at the maximum temperature. The capsules were filled with helium or argon and sealed. They were all irradiated in the Engineering Test Reactor (ETR) during Cycle 72 (May 28 to June 20, 1965, 19.1 effective days at full power). A complete history of the capsules is given in Table III.

TABLE III
CAPSULE HISTORY

<u>Capsule Number</u>	<u>Atmosphere</u>	<u>ETR Position</u>	<u>ETR Elevation, in.</u>	<u>Remarks</u>
GEH-13-280	Argon	N5 NE	15 to 21	Felt Wrap
-281	Argon	N5 NE	21 to 27	Felt Wrap
-282	Argon	J12 NE	17 to 23	
-283	Argon	J12 NE	23 to 29	
-284	Argon	N5 SW	13 to 19	
-285	Argon	N5 SW	25 to 31	
-286	Argon	E5 SW	15 to 21	Capsule Failed
-287	Argon	E5 SW	21 to 27	
-288	Argon	E5 NE	19 to 25	
-289	Helium	J12 NE	9 to 15	
-290	Helium	N5 SE	19 to 25	

Disassembly proceeded as previously described.⁽⁷⁾ Capsule 286 leaked in reactor and was full of water, while the samples

in capsule 290 were accidentally broken during the disassembly. Sample CM2-203 in capsule 285 was found to be broken in three pieces when the capsule was opened. There was no apparent reason for this fracture since the other three samples were in good condition. The thermal expansion of CM2-203 might have been prevented by the sample holder, although this seems unlikely. The samples were weighed and the length measurements (described above) were repeated. The CGM and PC materials were extremely radioactive, up to 30 R/hr at 3 in., which made measuring and handling a major problem.

Sample irradiation temperatures were determined by heat-transfer calculations.⁽⁷⁾ Confirmation of the temperatures by comparison of the CSF sample data with data from instrumented capsules^(4,6) was inconclusive due to the low neutron exposures. However, the uncertainty in calculated sample temperatures up to 1200 °C are estimated to be ± 100 °C because data from previous capsules⁽⁷⁾ (exactly the same as these UHTREX capsules) has been confirmed by data from instrumented capsules. Sample temperatures above 1200 °C are estimated to be about ± 150 °C because these are the first capsules containing felt warp and no data from instrumented capsules are available to confirm the temperatures. Additional irradiations of other graphites are underway to insure that sample temperature calculations in the capsules containing the felt wrap are accurate.

Neutron exposures are based on the activities of nickel and iron foils^(4,5,6) contained in each capsule. The activities are used in conjunction with the activation cross sections for each foil material, and neutron-spectra calculations^(4,5,6) for the respective capsule positions.

EFFECTS OF HEAT TREATMENT

The quantity and composition of gas given off by these materials was measured while heat treating them to 1000 °C.

Large quantities of gas were released, with the CGM and CGN carbons giving off about 5 times as much gas as the CGL or CSF graphites. The porous carbon gave off 80 times as much gas as the CSF; however, this material will not be used in UHTREX. (See Appendix A for the experimental details and the composition of the gas given off.) The possible effects of a large amount of gas being given off during reactor startup should be considered by the reactor designers and operating personnel.

As discussed above, the samples had to be heat treated prior to encapsulation. The length and weight changes resulting from the heat treatment (See Table II) were measured and the results are presented in Table IV.

TABLE IV
EFFECT OF HEAT TREATMENT ON UHTREX MATERIALS

<u>Material</u>	<u>Weight Change, %</u>	<u>Length Change, %</u>
CGL	-0.09 ± 0.02	+0.01 ± 0.005
CGM	-1.54 ± 0.10	+0.07 ± 0.05
CGN	-0.93 ± 0.18	+0.09 ± 0.02
PC	-7.39 ± 0.33	-1.69 ± 0.51
CSF	-0.006 ± 0.002	-0.002 ± 0.001

Because these measurements indicate sizeable changes in all cases except for the CGL graphite, the heat treatment may have an influence on the subsequent irradiation behavior, particularly for the carbons processed at temperatures lower than the heat-treatment temperature.

IRRADIATION RESULTS

Length and weight changes for the irradiated materials are presented in Table V. The weight changes for the samples in all the capsules except 283 seem quite reasonable, considering the outgassing behavior of the materials prior to irradiation. In

capsule 283, the presence of 2 CGM samples evidently led to the large weight losses, as those samples appear quite pitted. In contrast, the CSF and CGL samples appear undamaged. The porous carbon shows the largest weight losses as it also did when heat-treated (Table IV).

TABLE V
IRRADIATION RESULTS

Capsule Number	Irradiation Temperature, °C	Exposure, nvt x 10 ⁻²⁰ (E > 0.18 MeV)	Sample Number	Sample Code	Length Change, %	Weight Change, %
280	1800	2.75	CL1-1	CGL	+0.002	-0.017
280	1800	2.75	CL1-5	CGL	-0.001	-0.022
280	1800	2.75	CL1-205	CGL	-0.001	-0.023
280	1800	2.75	222-8	CSF	+0.004	-0.018
281	1800	2.23	CL1-6	CGL	-0.004	-0.034
281	1800	2.23	CL1-201	CGL	+0.003	-0.025
281	1800	2.23	CL1-202	CGL	-0.003	-0.028
281	1800	2.23	222-202	CSF	+0.005	-0.013
282	1400	7.29	CL1-3	CGL	-0.900	-0.033
282	1400	7.29	CL1-7	CGL	-0.946	-0.039
282	1400	7.29	CL1-204	CGL	-1.042	-0.033
282	1400	7.29	222-1	CSF	-0.701	-0.023
283	1300	6.55	CL1-2	CGL	-0.452	-1.961
283	1300	6.55	CM2-1	CGM	-2.428	-1.651
283	1300	6.55	CM2-17	CGM	-2.465	-0.944
283	1300	6.55	222-2	CSF	-0.187	-0.236
284	1000	2.74	CL1-203	CGL	-0.066	-0.031
284	1000	2.74	CM2-202	CGM	-1.133	-0.054
284	1000	2.74	CN2-204	CGN	-0.309	-0.058
284	1000	2.74	222-201	CSF	-0.026	-0.013
285	1200	2.40	CL1-8	CGL	-0.068	-0.042
285	1200	2.40	CM2-203	CGM	Broken	
285	1200	2.40	CN2-3	CGN	-0.292	-0.076
285	1200	2.40	222-3	CSF	-0.014	-0.016
286	Capsule Failed					
287	1000	2.27	CL1-4	CGL	-0.002	-0.062
287	1000	2.27	CM2-18	CGM	-0.287	-0.074
287	1000	2.27	CN2-1	CGN	-0.061	-0.124
287	1000	2.27	222-4	CSF	+0.010	-0.135
288	1000	2.64	CM2-219	CGM	-1.270	-0.041
288	1000	2.64	CN2-203	CGN	-0.385	-0.040
288	1000	2.64	PC-203	B260	-1.201	-0.409
288	1000	2.64	222-203	CSF	-0.050	-0.015
289	900	5.18	CN2-2	CGN	-0.265	-0.482
289	900	5.18	CN2-201	CGN	-0.247	-0.293
289	900	5.18	PC-206	B260	-1.732	-2.889
289	900	5.18	222-6	CSF	+0.025	-0.261
290	600	2.05	Samples Broken			

The length changes resulting from the irradiations are shown in Figure 3. The CGL graphite data agree very closely with the CSF graphite data at the lower exposures. At higher exposures the CGL samples contract about 1 1/2 to 2 1/2 times

the CSF transverse samples, and the amount of contraction becomes appreciable. The parallel samples of CGL contract slightly more than the transverse, but the behavior is quite isotropic when compared with the behavior of CSF graphite.⁽⁶⁾

FIGURE 3

Physical Distortion of UHTREX Materials as a Function of Exposure

The CGM carbon contracts considerably more and the CGN carbon contracts slightly more than the CGL graphite. The parallel samples of CGM contract about four times the amount of the transverse samples. The porous carbon contracts about the same amount as the CGM parallel samples. At 900 °C, the transverse and parallel samples of CGN show nearly identical behavior. However, at the higher temperatures, CGN shows anisotropic behavior with the parallel samples contracting five to six times the transverse.

The graphite felt used as a thermal radiation barrier was recovered intact and appeared undamaged, but no attempts at measurements were made. Since the 1000 and 1800 °C data agree so closely, it is possible that the higher temperature was not achieved (see discussion on irradiation temperatures above).

SUMMARY AND CONCLUSIONS

The dimensional stability of the graphite and carbon materials used in UHTREX was determined to neutron exposures equal to expected lifetime exposures, and the behavior of these materials was compared with a conventional nuclear graphite. Irradiations were conducted in the ETR to maximum neutron exposures of 7×10^{20} nvt, $E > 0.18$ MeV at sample temperatures between 900 and 1800 °C. At irradiation temperatures of 1000 and 1800 °C, CGL graphite behaves about the same as CSF, and the length changes are less than -0.06% in both orientations at the maximum exposures. The isotropic behavior of CGL is expected because the preirradiation CTE ratio of 1.02 indicates a very isotropic material. At 1300 to 1400 °C, the amount of contraction has increased sharply to about -1%. The increase in contraction over CSF at these conditions is probably due to the impregnation treatment that CGL received. It appears that CSF length-change data could be applied for CGL to exposures of about 2×10^{21} if an increased contraction of 1 1/2 times was assumed. However, for application

to UHTREX, the data obtained exceed the expected lifetime exposure (5×10^{20} nvt) for CGL so this should not be necessary.

In all cases the carbons contract more than the CGL, and the more dense impregnated carbon contracts the most. CGM shows very large length changes, probably because of the impregnant material added during manufacture. However, at the maximum expected lifetime UHTREX conditions (2.5×10^{20} nvt at 2000 °F), the maximum contraction is only 1.3%. At the conditions studied, the CGN carbon contracted a maximum of 0.4%.

Length changes in the porous carbon were about the same as in CGM. Since expected reactor exposures are only about one-tenth of those achieved in the tests, the length changes should be of slight concern; assuming, of course, that the insulating carbon actually used in UHTREX shows behavior similar to the porous carbon tested. Extrapolations of the carbon data to higher exposures should not be attempted because no data on these types of materials are available.

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APPENDIX A

Outgassing studies were made on the UHTREX materials as an adjunct to the main irradiation program to determine whether large quantities of gas could be released inside the irradiation capsules. For the outgassing, the carbon samples were placed in a quartz tube and attached to a vacuum system. Before heating the carbon sample, the system was evacuated for 1 to 2 hr. The samples were then heated with a tube furnace to about 1000 °C, and the gases evolved were removed with a Toepler pump and collected in a section of the line with known volume. The out-gassing process was continued for 16 to 18 hr. The pressure of the resulting gas mixture was observed and the composition determined by gas chromatography.

The results are listed below:

<u>Carbon Type</u>	<u>Quantity of Gas, ml at NTP per 100 g of Sample</u>	<u>Approximate Composition, %</u>	<u>Comments</u>
CGL	16	45 CO 35 CO ₂ 15 H ₂ 5 H ₂ O Trace of hydrocarbon	
CGN	84	60 CO 25 CO ₂ 1-2 H ₂	Also contained 2-10% H ₂ O
CGM	88	80 H ₂ 5 CO 4 H ₂ O 1 CO ₂ 3 N ₂ 1 CH ₄	
B-260 Porous carbon	1200	Dry gas	Also several milliliters of liquid H ₂ O was evolved. Sample outgassed for only 6 hr and gases were still being liberated quite rapidly.
Reactor graphites (CSF, TSX)	~15		

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