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HIGH-TEMPERATURE GRAPHITE IRRADIATIONS:
800 TO 1200 °C --
INTERIM REPORT NO. 1

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INTRODUCTION

Irradiations are being conducted in the temperature range of 800 to 1200 °C to determine the dimensional stability of various types of graphites. There are available considerable data from lower temperature irradiations.⁽¹⁻³⁾ Data in this higher temperature range are very limited,⁽⁴⁻⁷⁾ even though the temperature range is of particular interest to operators and designers of graphite-moderated reactors.

The primary data obtained are the changes in physical dimensions for the various graphites. Secondary objectives of the experiment are measurements and determinations of other properties such as volume changes, crystallite parameters, Young's modulus, electrical resistivity, thermal conductivity, coefficient of thermal expansion, annealing effects, microscopy, and strength. They provide additional information on the effects of irradiation.

A capsule, designated GEH-13-10 and containing several different types of graphite, was irradiated in the Engineering Test Reactor (ETR). Results from this experiment are presented in this report. A discussion of the results in terms of the structure of polycrystalline graphite will be reported elsewhere.⁽⁸⁾

SUMMARY

A temperature-monitored irradiation test, GEH-13-10, was conducted in the K7 position of the ETR to determine the behavior of several types of graphite at temperatures from 800 to 1200 °C and neutron exposures up to 4.5×10^{21} nvt, $E > 0.18$ MeV. Mean sample temperatures agreed within ± 100 °C from one reactor cycle to the next.

The graphites irradiated were: conventional-coke graphites, CSF and TSGBF; needle-coke graphites, KC, EGCR, and TSX; and raw-coke graphites, 63-16, 63-17, and 63-38. The physical distortion of the

graphites tested showed a dependence on the temperature of irradiation, although this varied considerably for the various graphites. All the transverse samples of the conventional and needle-coke graphites contracted except one TSGBF sample, which expanded 3.3%. The samples irradiated at 1200 °C generally contracted more than those at 800 °C and about the same as other samples irradiated at 450 °C.^(1, 2) The samples of the raw-coke graphites contracted much less than those of the other graphites and in some cases expanded slightly in both the transverse and parallel orientations. The parallel samples of the conventional and needle-coke graphites contracted considerably more at 1200 °C than at 800 °C, and the amount was also considerably more than that for samples irradiated at 450 °C.

The raw-coke graphites, which all had nearly the same initial CTE and were very isotropic, showed differences in irradiation behavior, indicating the less-graphitic phase has an effect on the radiation-induced physical distortion. The large expansion in the TSGBF graphite was accompanied by an increase in CTE. Both the dimensions and the CTE also increased upon subsequent annealing, indicating the c expansion of the crystallites has become the controlling factor in dimensional behavior.

EXPERIMENTAL PROCEDURE

Sample Selection and Preparation

The conventional and needle-coke graphites were obtained from full-size extrusions, and the raw-coke graphites were obtained from molded pieces. The graphites are described in Table I.

The sampling schemes for the extruded graphites CSF, KC, TSGBF, EGCR, and TSX are shown in Figures 1-3. Since the raw-coke graphites are molded, the sampling scheme takes the form shown in Figure 4 for 63-16 and 63-17 and in Figure 5 for 63-38. Orientation and anisotropy effects have been discussed previously.⁽¹⁰⁾

After machining to a pie-shape cross section (1/6 of a circle), individual samples were annealed at 1800 °C for 1 hr at a pressure of 3×10^{-4} mm Hg.⁽¹⁾

TABLE I
GRAPHITE DESCRIPTION

Sample Designation	Block Numbers	Coke Source and Type	Block Size in.	Remarks ^(a)
CSF ^(b)	142	Cleves (conventional type)	4 x 4 x 50	Reactor grade, F-purified at 2700 °C, density 1.66 g/cm ³
TSGBF ^(b)	125	Texas-Lockport (conventional type)	3-3/4 x 3-3/4 x 48	Reactor grade, graphitized and F-purified in one step at 2450 °C, density 1.65 g/cm ³
KC ^(b)	187	Kendall (needle type)	3-3/4 x 3-3/4 x 48	Reactor grade AGOT, thermally purified, very well graphitized, density 1.7 g/cm ³
EGCR ^(b)	NC8	Continental-Lake Charles (needle type)	17-1/2 x 17-1/2	Reactor grade AGOT, EGCR graphite, density 1.72 g/cm ³
TSX ^(b)	185	Continental-Lake Charles (needle type)	6 x 6 x 48	Reactor grade, graphitized at 3000 °C, very anisotropic (CTE _I /CTE _{II} = 4.78), density 1.71 g/cm ³
Raw-coke ^(c) Base Stock	63-16	Texas-Lockport (raw coke)	6.5 diam x 6	Experimental grade, volatile content of raw coke 12%, coal-tar derivative binder, amount of binder carbon in baked product negligible, graphitized at 2500 °C, very isotropic (CTE _I /CTE _{II} = 1.02), density 1.72 g/cm ³
Raw-coke ^(c) 1 - impreg.	63-17	Texas-Lockport (raw coke)	4.1 diam x 6	Experimental grade, same base stock as 63-16 with one coal-tar-pitch impregnation, amount of carbon from impregnant in final product 5.7% graphitized at 2500 °C, very isotropic, (CTE _I /CTE _{II} = 1.02) density 1.78 g/cm ³
Raw-coke ^(c) No binder	63-38	Texas-Lockport (raw coke)	4 x 4 x 6	Experimental grade, volatile content of raw coke 12%, binder none, graphitized at 2500 °C, isotropic (CTE _I /CTE _{II} = 1.27) density 1.35 g/cm ³

(a) Further details on some of these materials may be found in Nuclear Graphite⁽⁹⁾

(b) Manufactured by Carbon Products Division, Union Carbide Corporation

(c) Manufactured by Great Lakes Carbon Corporation

FIGURE 1
Sampling Scheme - CSF, KC, TSGBF Graphites

FIGURE 2
Sampling Scheme - EGCR Graphite

FIGURE 3
 Sampling Scheme - TSX Graphite

FIGURE 4

Sampling Scheme - 63-16, 63-17 Graphites

FIGURE 5
Sampling Scheme - 63-38 Graphite

Experimental Details

Details on the capsule design, pre- and postirradiation measurements, and other experimental procedures are presented in Appendix A. The neutron exposure to the samples was determined experimentally and details are presented in Appendix B.

IRRADIATION RESULTS

The irradiation results for the samples from the GEH-13-10 capsule are presented in Tables II and III.* Length changes are based on end-to-end measurements with the exception of sample 187-2, for which the hole-to-hole measurements were used (Appendix A, Disassembly). Sample weights did not change more than 0.3% during the irradiation period.

All the tabulated data are presented graphically in the following subsections. For some data the comparative CSF data at 450 to 800 °C⁽¹⁾ are shown as a lightly cross-hatched area. Data obtained from more than one sample at any particular exposure are presented as a mean point, with a bar indicating the maximum and minimum values and not the standard deviation. The small number by the mean data point indicates the number of samples used for the average. Isothermal lines are drawn through the data points to indicate estimates of the general trends on most of the graphs. Since there is only one data point in most cases, the lines cannot be assumed to show exact indications of behaviour.

*See pages 32 and 33.

Length Change

The length-change data from Table II are presented as a function of exposure in Figures 6 and 7. Data for the samples taken transverse to the extrusion axis are shown in Figure 6. All data for the raw-coke graphites, both transverse and parallel to the molding force, are also included in Figure 6 since these materials are quite isotropic prior to irradiation (as indicated by CTE measurements). There is a rapid increase in the amount of contraction at 1000 °C but the amount at 1200 °C appears to be the same and even a little less than that at 1000 °C. For the CSF graphite, the length change for 950 °C is the same as that for 1100 to 1200 °C and a little less than that for 450 °C. For the EGCR graphite, the 1100 to 1200 °C isotherm is nearly the same as the 1000 °C isotherm for CSF. The TSX graphite shows much less contraction at these temperatures. The data points for 800, 950, and 1100 °C fall on one isotherm while the 1200 °C isotherm shows less contraction. The needle-coke graphite, KC, contracts even less than the TSX and shows more temperature dependence, with the 950 °C and 1100 °C data falling on two isotherms. The one sample of the conventional-coke graphite, TSGBF, on which data were obtained shows a very large expansion of nearly 3.3%. This expansion, confirmed by the hole-to-hole length measurement, would appear to indicate the TSGBF, a relatively non-graphitic material, has gone through turnaround and is expanding very rapidly.

Based on other data ⁽⁷⁾ at lower neutron exposures which show much larger contractions than those indicated by the trend lines drawn on Figure 6, it is possible that the CSF, EGCR, and TSX graphites may be rapidly approaching or even past turnaround at the exposure given. From the TSGBF data (discussed above) it appears that the materials having a somewhat less-graphitic structure reach at lower exposures than the graphites having a more ordered structure.

The raw-coke graphites show much less contraction than the other types, and in several cases they even show an expansion. The expansion may indicate that these materials may not show a contraction followed by an expansion but may continue to expand in much the same manner as the other materials do after going through the turnaround. At 950 °C, the base stock (63-16)

shows some anisotropy with the transverse sample expanding over three times as much as the parallel sample. At 1150 °C, the same material contracts 0.2%, but this is only one-fourth as much as the CSF graphite. Also at 1150 °C, the raw-coke graphite containing no binder (63-38) expands nearly 0.6%, whereas the parallel sample of the impregnated material (63-17) contracts over 0.9%. Thus, it appears that in 63-16, the slight amount of binder carbon causes a significant increase in density and a contraction. In contrast, an expansion is shown by the material having no binder (63-38). Since the impregnated sample was of parallel orientation, and the base stock does show some anisotropic behavior at 950 °C, the large difference in contraction of 63-17 and 63-16 at 1150 °C probably is due to a combination of the effects of impregnation and orientation.

The data for the parallel samples (excluding the raw-coke graphites) are presented in Figure 7. The amount of contraction increases very rapidly at 1000 °C and is much greater than the contraction at 450 °C. At 1200 °C, it is about the same as at 1000 °C. The CSF data for 1000 to 1200 °C fall on one isotherm between the 1000 °C and 1200 °C TSGBF data. The needle-coke graphites show less contraction than the CSF; the TSX also contracts more at 1000 °C than at 1100 to 1200 °C. At 1000 °C, the KC material contracts almost exactly the same as TSX.

Contraction rates based on the curves in Figures 6 and 7 at an exposure of 4×10^{21} nvt are shown in Figure 8. For the CSF transverse samples the rate decreases from 450 to 800 °C, then increases to 1000 °C and finally decreases and levels out at 1200 °C. The parallel CSF data show a slight decrease in contraction rate from 450 to 800 °C, a very rapid increase to 1000 °C and a leveling off to 1200 °C. The raw-coke graphites have a smaller contraction rate than transverse CSF samples, with the exception of the parallel impregnated raw-coke material. The material with no binder (63-38) shows the highest growth rate, and the base material (63-16) shows an intermediate rate which changes to a contraction at the highest temperature. The parallel samples show a higher rate than the transverse indicating an anisotropic irradiation behavior.

FIGURE 6
Physical Distortion of Graphite (Transverse) as a Function of Exposure

FIGURE 7
Physical Distortion of Graphite (Parallel) as a Function of Exposure

FIGURE 7
Physical Distortion of Graphite (Parallel) as a Function of Exposure

FIGURE 8

Rate of Length Change for Graphite as a Function
of Irradiation Temperature

Volume Change

The resultant volume changes were calculated from the estimated length change curves (Figures 6 and 7). The model used is a sample of square cross section with both the width and thickness taken as transverse directions and the length taken as the parallel direction for all the extruded materials. For the molded raw-coke base stock (63-16) the width and thickness are taken as parallel directions and the length as the transverse direction. The equation used is:

$$\frac{\Delta V}{V_0} = \left(1 + \frac{\Delta L}{L_1}\right)^2 \times \left(1 + \frac{\Delta L}{L_2}\right) - 1$$

where V = volume

L_1 = length transverse to the extrusion axis or the molding force

L_2 = length parallel to the extrusion axis or the molding force.

The calculated volume changes are shown in Figure 9 as a function of exposure. For the temperature range of 950 to 1200 °C, the data for each graphite are grouped together with the exception of the data for KC graphite at 950 and 1050 °C.

The conventional-coke CSF graphite shows the greatest volume change. The needle-coke graphites, EGCR and TSX, are grouped together with the latter showing the least volume change. At 950 °C, the KC graphite behaves nearly the same as CSF graphite at 800 °C.

The volume change of the TSGBF graphite is of particular interest since it appears to show a turnaround at very low exposures followed by a rapid volume increase. The raw-coke graphite, 63-16, appears to show a nearly constant rate of volume expansion.

FIGURE 9
Volume Distortion of Graphite as a Function of Exposure

Coefficient of Thermal Expansion

The coefficient of thermal expansion as a function of temperature was determined for some of the samples irradiated in GEH-13-10. The results of the CTE (from 25 °C to the final temperature) are presented in Table III* and in Figures 10 and 11. The CTE for the more graphitic materials, CSF, KC, TSX, and EGCR, changes only slightly with irradiation (Figure 10). The CTE of the less graphitic materials shows a much larger change with irradiation (Figure 11). The CTE of the TSGBF transverse material increases about 25% while that of the raw-coke graphite (63-38) decreases about 21% and that of the base material (63-16) decreases about 62%. For several of the graphites, the change in CTE decreased as the final temperature increased. It does not appear that this decrease is due to equipment malfunction. For the more-graphitic KC material, the sample irradiated at 950 °C to 2.8×10^{21} nvt (187-1) shows a slight increase in CTE while the sample irradiated at 1100 °C to 3.8×10^{21} nvt (187-2) shows a slight decrease.

These CTE results indicate that the less-graphitic areas in a graphite piece play an important role in the physical property changes occurring during neutron irradiation. This is particularly apparent in the less-graphitic materials (TSGBF and raw-coke) and is confirmed by the large decrease in CTE in the raw-coke graphite containing some binder material.

*See page 33.

FIGURE 10

Coefficient of Thermal Expansion of Graphite
(25 $^{\circ}\text{C}$ to the Final Temperature)

FIGURE 11

Coefficient of Thermal Expansion of Graphite
 (25 C to the Final Temperature)

Annealing Effects

The samples of the less-graphitic materials discussed in the previous section were annealed for 1 hr at 1700 °C at 3×10^{-4} Torr. The changes in c spacing are shown in Figure C1. For the TSGBF and raw-coke graphite (63-38) annealing causes no observable change in the layer spacing. However, for the raw-coke base material (63-16) a large increase in c spacing is observed.

The changes in CTE resulting from the annealing are shown in Figure 11. The CTE for the TSGBF graphite shows a large increase of approximately 86% compared to the unirradiated value, or about 61% in addition to the increase resulting from the irradiation. The CTE of the raw-coke graphite (63-38) also increases from the irradiated result and, therefore, shows a partial recovery back to the unirradiated value. The net change compared to the unirradiated result is -9%, or an increase of 12% from the irradiated result. The CTE of the raw-coke base graphite (63-16) also shows a slight recovery back toward the unirradiated value. The increase is +5% or a net change of -57% from the unirradiated result.

No observable change in physical length occurred as a result of the annealing of the raw-coke materials. However, it appeared the TSGBF sample (125-23) expanded during the annealing. Consequently, a separate annealing study was made on a small section of this sample. The first run at 600 °C for 1 hr at 3×10^{-4} Torr showed no observable changes in either length or weight. The second run at 1200 °C for 1 hr. at 2×10^{-4} Torr resulted in no weight change and an increase in length of 0.16%. The final run at 1600 °C for 1 hr at 9×10^{-5} Torr resulted in no weight change and an increase in length of 0.14%. Thus, using the irradiation length-change data for sample 125-25, the TSGBF graphite grew 3.28% during irradiation and about one-tenth of this or an additional 0.30% during annealing.

The annealing results for the TSGBF graphite indicate that the large increase in CTE cannot be explained by increases in crystallite c -axis growth as measured by c spacing, since there was no observable change in c . However, it would appear that the large increases must be due to c -axis

growth since the c direction CTE is much larger than the a direction. Conversely, from the intensity distribution of the x-ray traces, it appears that the TSGBF graphite tends to become more isotropic during the annealing because the transverse-to-parallel orientation ratio decreased approximately 20%.

The fact that the CTE increases for both the raw-coke graphites during annealing while the c spacing increases only for the base-stock (63-16) also indicates that the less graphitic areas play an important role in the mechanism of damage.

Microscopy

The surface appearance of several of the materials was examined after the irradiation and compared with unirradiated specimens. The changes in the TSGBF graphite are striking while the other materials show no major changes. The appearance of sample 125-23 is shown in Figure 12. This material has a very porous structure as is seen in the unirradiated specimen. After irradiation, a network of cracks is readily observable, particularly in the transverse surface. These cracks extend completely across the surface and are oriented in the transverse direction.

The microstructures of the samples were also examined, and the appearances of the surfaces are shown in Figures 13 and 14. The surface of the irradiated graphite shows a somewhat "crumbled" appearance. The crack structure has become noticeably more pronounced and a continuous but tortuous path of open cracks can be followed through the structure. This may partially account for the large growth seen in the TSGBF sample which was recovered intact after the irradiation (125-25). The appearance of sample 125-23 after the annealing study is also shown in Figures 13 and 14. The amount of "crumbling" appears to have stayed about the same or perhaps increased slightly, and the crack structure continues to be evident.

The microstructures of the raw-coke graphites are shown in Figure 15. The transverse and parallel orientations show no observable differences so only one is shown for each material. The changes induced by irradiation are apparently negligible. The graphite without binder (63-38) has a very open structure and shows more porosity than even the post-irradiated specimen of TSGBF. Perhaps this porosity allows the crystallites to change dimensions without affecting the bulk dimensions appreciably, at least to the exposures reported.

Transverse

Unirradiated

Parallel

Transverse

Irradiated

Parallel

FIGURE 12

Structure of TSGBF Graphite
(10X)

Unirradiated

Irradiated

Annealed

FIGURE 13
Microstructure of TSGBF Graphite
(Transverse)
(75X)

Unirradiated

Irradiated

Annealed

FIGURE 14
Microstructure of TSGBF Graphite
Parallel
(75X)

Unirradiated (63-16-110)

Irradiated (63-16-71)

Base Stock (63-16 Transverse

Unirradiated (63-38-3)

Irradiated (63-38-60)

No Binder (63-38) Parallel

FIGURE 15
Microstructure of Raw-Coke Graphites
(75X)

Strength

The tensile and compressive strengths of the TSGBF sample (125-23) that showed the large crack structure after irradiation (see section on Microscopy) were measured and compared to results on unirradiated materials. The results are given in Table IV. As expected, the strength decreases markedly.

TABLE IV
STRENGTH OF TSGBF GRAPHITE

	Strength, psi		<u>Percent Change</u>
	<u>Unirradiated</u>	<u>Irradiated</u>	
Tensile	1560	180	-88
Compressive	8540	3410	-60

Crystallite Parameters

The effects of irradiation on the crystallite parameters are presented in Appendix C. In general, the distance between alternate layer planes, c , increases with little or no temperature dependence over the range of 800 to 1200 °C. The apparent crystallite size in the c direction, L_c ; the unit cell dimension, a ; and the apparent crystallite size in the a direction, L_a ; generally decrease.

Young's Modulus of Elasticity

The data on Young's modulus, E , as a function of neutron exposure are presented in Appendix C. In general " E " increases rapidly and tends to approach a constant value.

Electrical Resistivity

The effects of irradiation on the electrical resistivity, ρ , are presented in Appendix C. In general, ρ increases significantly and remains at a constant value for all temperatures.

Thermal Conductivity

The thermal conductivity, k , decreases to nearly a constant value after irradiation. The data for the few samples measured are presented in Appendix C.

CONCLUSIONS

The length changes of the graphites tested over the temperature range of 800 to 1200 °C show a dependence on the temperature of irradiation, particularly in the parallel direction. The transverse samples irradiated at 1200 °C contract more than those irradiated at 800 °C and about the same as samples irradiated at 450 °C. Those irradiated at 1000 °C contract at the highest rate. This may indicate that turnaround⁽¹⁾ has already been passed and that the samples are expanding at these exposures. The parallel samples of the extruded materials contract considerably more at 1200 °C than at 800 °C, and the amount is significantly more than for samples irradiated at 450 °C. The contraction rate appears to be about the same at irradiation temperatures of 1000 and 1200 °C. This may be an indication of a lessening or even a saturation of contraction in the parallel direction that has not been observed previously for lower irradiation temperatures, even at much higher exposures.⁽¹⁾

The raw-coke graphites contract less than the other graphites, and even expand in some cases. Some indication of anisotropic behavior is shown by the base material at 950 °C, even though this material, as well as the impregnated material, is nearly isotropic ($CTE_{\parallel} / CTE_{\perp} = 1.02$) prior to irradiation. At 1150 °C the material containing no binder expands while the base stock contracts, and the parallel orientation of the impregnated material contracts significantly. Since these materials all had nearly the same initial CTE, it appears the binder phase has an effect on the amount of physical distortion induced by irradiation. Since the impregnated sample was of parallel orientation while the sample of the base stock was transverse, the large difference in contraction of 63-17 and 63-16 may be due to a combination of impregnation and orientation.

The CTE for the base material decreases significantly more than that of the no-binder material, indicating that as the less-graphic part of an isotropic material becomes oriented during irradiation, it tends to increase the a alignment of the supporting structure and thus decrease the CTE.

The length changes observed on the transverse sample of TSGBF graphite, 125-25, in conjunction with postirradiation studies on the companion sample, 125-23, provide several significant results:

- This graphite, that was heated to only 2450 °C during manufacture and is therefore less graphitic than any of the other conventional or needle-coke graphites, expands significantly upon irradiation. This suggests that the graphite may have gone through a very rapid contraction followed by turnaround⁽¹⁾ and even more rapid expansion. This, in turn may also indicate that transverse samples of some of the other materials may be near or even past turnaround and are, perhaps, expanding at the exposures reported.
- The irradiation expansion is not annealed out by heating above the irradiation temperature, instead, the sample continues to expand.
- The CTE increases significantly during irradiation and increases even more upon annealing.
- A large increase in c spacing occurs during irradiation and remains unchanged after annealing.
- The sample becomes full of cracks during irradiation, and these continue to open up on annealing.
- The tensile strength decreases drastically upon irradiation under these conditions.

From these observations, it appears that in an anisotropic graphite containing a large amount of less-graphitic material, the expansion of the crystallites in the c direction becomes a controlling factor in physical property changes and causes a turnaround in the dimensional behavior. A predominant expansion in the c direction would also explain the increases in CTE, both during irradiation and subsequent annealing; however, it would not explain the apparent tendency toward a more isotropic structure as indicated by the X-ray trace after annealing. A detailed discussion of the current concepts of the two-phase model for the explanation of the observed behavior of graphite is presented elsewhere.⁽⁸⁾

In applying the length-change data reported here to reactor conditions, consideration should be given to the differences between small samples and large blocks. For both the transverse and parallel orientations, a large block, 4 x 4 x 24 in., contracted approximately twice that of small 0.43 in. diam x 4 in. samples. ⁽¹¹⁾ Consequently, such a factor should be considered when applying these data to large reactor blocks.

This irradiation program is continuing, and 75% of these samples are currently being reirradiated with some new samples in a capsule designated GEH-13-11.

TABLE II
GEH-13-10 EXPERIMENTAL RESULTS

GEN-15-10 EXPERIMENTAL RESULTS																									
Capsule Position	Temperature, °C	Exposure nvt x 10 ⁻²¹ (E > 0.18 MeV)	Sample Code	Sample Number	Length Change, % ΔL/L ₀	Crystallite Parameters, Å												Young's Modulus			Electrical Resistivity			Post 1 Hr Anneal at 1700 °C	
						c		% Δc/c ₀	L _c		% ΔL _c /L _{c0}	a		% Δa/a ₀	L _a		% ΔL _a /L _{a0}	psi x 10 ⁻⁶		% ΔE/E ₀	ohm-cm x 10 ⁴		% Δρ/ρ	c Final	% Δc/c ₀
						Initial	Final		Initial	Final		Initial	Final		Initial	Final		Initial	Final		Initial	Final			
1	800	1.72	CSF I	142-71	-0.148 ± 0.001	6.727	6.732	+0.074	520	220	-57.7	2.4628	2.4593	-0.014	1305	560	-57.1	0.81	1.20	+48.1	15.92	44.64	+180.4		
			CSF II	142-249	-0.234 ± 0.001	6.698	6.737	+0.582	370	223	-39.7	2.4608	2.4593	-0.006	668	470	-29.6	1.52	2.38	+56.6	7.72	23.51	+204.5		
			TSX I	185-C1	-0.128 ± 0.002	6.729	6.724	-0.074	505	380	-24.8	2.4616	2.4596	-0.008	1775	1105	-37.7	0.79	1.40	+77.2	12.52	37.04	+195.8		
			TSX II	185-202	-0.254 ± 0.001	6.724	6.739	+0.223	435	210	-51.7	2.4620	2.4584	-0.015	1115	600	-46.2	2.49	3.36	+34.9	6.11	19.83	+224.5		
			EGCR I	NC8-115	-0.164 ± 0.001	6.726	6.733	+0.104	555	240	-56.8	2.4620	2.4583	-0.015	1465	380	-67.2	0.97	1.58	+62.9	10.50	32.79	+212.3		
			EGCR II	NC8-250	-0.188 ± 0.001	6.702	6.734	+0.418	495	230	-53.5	2.4614	2.4595	-0.008	1330	890	-33.1	1.32	2.05	+55.3	6.79	22.97	+238.3		
2	950	2.82	CSF I	142-72	-0.448 ± 0.002	6.711	6.737	+0.387	480	140	-70.8	2.4618	2.4583	-0.014	850	273	-67.9	0.83	1.05	+26.5	15.35	44.25	+188.3		
			TSX I	185-C3	-0.342 ± 0.003	6.716	6.731	+0.223	528	127	-75.9	2.4622	2.4595	-0.011	1780	390	-78.1	0.90	1.37	+52.2	12.61	39.16	+210.5		
			KC I	187-1	-0.089 ± 0.001	6.722	6.728	+0.089	535	165	-69.2	2.4622	2.4623	+0.001	1415	620	-56.2	0.85			13.14	46.27	+252.1		
			KC II	187-201	-0.828 ± 0.001	6.722	6.727	+0.074	610	159	-73.9	2.4619	2.4596	-0.009	1650	1560	- 5.4	1.42			6.42	23.97	+273.4		
			63-16 I	63-16-70	+0.363 ± 0.002	6.746	6.769	+0.341	338	96	-71.0	2.4609	2.4555	-0.022	725	2399	+231.0	1.65	13.10	+693.9	16.82	33.07	+ 96.6		
			63-16 II	63-16-270	+0.108 ± 0.002	6.750	6.771	+0.311	350	107	-69.4	2.4603	2.4571	-0.013	798	360	-54.9	1.29	8.76	+501.6	15.34	30.87	+101.2		
3	1100	3.81	CSF I	142-73	-0.714 ± 0.001	6.723	6.745	+0.327	428	128	-70.1	2.4619	2.4607	-0.005	760	405	-46.7	0.77	1.10	+42.9	14.18	45.31	+219.5		
			TSX I	185-C4	-0.429 ± 0.001	6.722	6.736	+0.208	520	128	-75.4	2.4623	2.4592	-0.013	1170	372	-68.2	0.88	1.41	+60.2	12.56	36.13	+187.7		
			KC I	187-2	-0.278 ± 0.010	6.722	6.742	+0.298	450	136	-69.2	2.4630	2.4608	-0.009	1625	390	-76.0	1.30			12.74	46.30	+263.4		
			EGCR I	NC8-116	-0.903 ± 0.002	6.719	6.739	+0.298	465	119	-74.4	2.4620	2.4598	-0.009	2000	690	-65.5	0.98	1.61	+64.3	10.47	33.68	+221.7		
			TSGBF I	125-25	+3.284 ± 0.002	6.773	6.809	+0.532	295	76	-74.2	2.4561	2.4554	-0.003	430	190	-55.8	0.88	1.03	+17.0	30.84	60.15	+ 95.0		
			TSX II	185-206	-2.211 ± 0.002	6.719	6.731	+0.179	490	128	-73.9	2.4638	2.4495	-0.058	730	1720	+138.4	2.41	3.40	+41.1	5.62	19.90	+254.1		
4	1200	4.51	CSF I	142-76	-0.776 ± 0.001	6.720	6.749	+0.432	445	98	-78.0	2.4614	2.4593	-0.009	935	590	-36.9	0.83	1.09	+31.3	15.63	43.99	+181.4		
			CSF II	142-251	-4.502 ± 0.005	6.712	6.754	+0.626	372	117	-68.5	2.4604	2.4573	-0.013	872	425	-51.3	1.66	2.33	+40.4	7.93	24.31	+206.6		
			TSX I	185-C5	-0.352 ± 0.001	6.716	6.729	+0.194	405	163	-59.8	2.4623	2.4622	-0.001	1430	1140	-20.3	0.86	1.44	+67.4	12.72	37.65	+196.0		
			TSX II	185-204	-3.296 ± 0.002	6.723	6.738	+0.223	468	120	-74.4	2.4616	2.4592	-0.010	1230	600	-51.2	2.36	3.32	+40.7	5.74	19.25	+235.4		
			EGCR I	NC8-119	-1.006 ± 0.002	6.723	6.746	+0.342	570	126	-77.9	2.4610	2.4611	+0.001	875	342	-60.9	0.93	1.51	+62.4	10.94	35.21	+221.8		
			EGCR II	NC8-254	-3.445 ± 0.001	6.723	6.749	+0.387	520	119	-77.1	2.4592	2.4573	-0.008	1085	420	-61.3	1.49	1.97	+32.2	4.88	23.24	+376.2		
5	1150	4.47	CSF II	142-252	-4.788 ± 0.002	6.728	6.739	+0.134	600	108	-82.0	2.4622	2.4599	-0.009	1600	520	-67.5	1.70	1.95	+14.7	8.28	39.74	+367.9		
			TSGBF I	125-23	Broken	6.738	6.822	+1.247	290	71	-75.5	2.4582	2.4580	-0.001	315	285	- 9.5				27.40			6.824	+1.276
			TSGBF II	125-213	-4.321 ± 0.001	6.756	6.817	+0.901	300	74	-75.3	2.4607	2.4564	-0.017	750	203	-59.6	6.71	6.51	-3.0	19.75	49.01	+148.2		
			63-16 I	63-16-71	-0.194 ± 0.002	6.748	6.769	+0.311	290	93	-67.9	2.4606	2.4557	-0.020	565	172	-69.6	1.68			15.94			6.807	+0.874
			63-17 II	63-17-250	-0.939 ± 0.002	6.740	6.784	+0.653	320	85	-73.4	2.4603	2.4564	-0.016	445	275	-38.2	1.22	11.64	+854.1	14.23	30.13	+111.7		
			63-38 I	63-38-60	+0.570 ± 0.023	6.756	6.812	+0.829	350	73	-79.1	2.4610	2.4595	-0.006	570	180	-68.4	0.69	4.24	+514.5	38.50	78.19	+103.1	6.811	+0.814
6	1000	3.41	CSF I	142-79	-0.793 ± 0.008	6.722	6.752	+0.446	465	123	-73.5	2.4601	2.4600	-0.001	960	390	-59.4	0.77	1.11	+44.2	14.57	39.05	+168.0		
			CSF II	142-253	-3.354 ± 0.004	6.723	6.744	+0.312	560	93	-83.3	2.4611	2.4570	-0.017	920	435	-52.7	1.65	2.01	+21.8	8.00	42.66	+433.3		
			TSX II	185-205	-2.348 ± 0.005	6.723	6.742	+0.283	510	132	-74.1	2.4627	2.4578	-0.020	1115	325	-70.9	2.39	3.37	+41.0	5.73	18.73	+226.9		
			KC II	187-202	-2.358 ± 0.003	6.769	6.743	-0.386	465	122	-73.8	2.4635	2.4600	-0.014	2000	490	-75.5	1.36	1.97	+44.9	6.47	25.85	+299.5		
			EGCR II	NC-253	-1.970 ± 0.013	6.722	6.738	+0.238	555	125	-77.5	2.4632	2.4596	-0.015	1450	590	-59.3	1.53	2.06	+34.6	6.56	24.47	+273.0		
			TSGBF II	125-214	-3.933 ± 0.002	6.760	6.804	+0.651	320	75	-76.6	2.4582	2.4596	+0.004	535	273	-49.0	7.47	7.10	-4.9	21.66	58.54	+170.3		

TABLE III									
SUPPLEMENTARY EXPERIMENTAL RESULTS									
Sample Code	% Change	Coefficient of Thermal Expansion ^(a)							Mean
		100 °C ^(a)	200 °C	300 °C	400 °C	500 °C	600 °C	700 °C	
CSF 1 (Unirradiated)		3.33	3.47	3.63	3.78	3.89	4.01	4.11	
CSF II (Unirradiated)		1.14	1.24	1.38	1.52	1.68	1.81	1.93	
TSX 1 (Unirradiated)		2.92	3.35	3.75	3.97	4.14	4.25	4.33	
EGCR1 (Unirradiated)		2.53	2.66	2.73	2.94	3.12	3.79	3.95	
EGCR II (Unirradiated)		1.20	1.12	1.28	1.37	1.46	1.63	1.80	
EGCR II (NC8-253)		0.93	1.16	1.28	1.37	1.51	1.63	1.76	
	$\Delta CTE/CTE_0$	-22.5	+3.6	0	0	+3.4	0	-2.2	-2.5
KC 1 (Unirradiated)		3.85	4.12	4.26	4.50	4.61	4.77	4.92	
KC 1 (187-1)		4.00	4.23	4.46	4.67	4.79	4.92	5.13	
	$\Delta CTE/CTE_0$	+3.9	+2.7	+4.7	+3.8	+3.9	+3.1	+4.3	+3.8
KC 1 (187-2)		3.22	3.77	4.00	4.26	4.47	4.68	4.92	
	$\Delta CTE/CTE_0$	-16.4	-8.5	-6.1	-5.3	-3.0	-1.9	0	-5.9
TSGBF 1 (Unirradiated)		3.47	4.00	4.18	4.47	4.68	4.82	4.92	
TSGBF 1 (125-23)		4.62	5.14	5.43	5.71	5.90	6.01	6.09	
	$\Delta CTE/CTE_0$	+13.3	+28.5	+29.9	+27.7	+26.1	+24.7	+23.8	+24.9
TSGBF 1 (125-23) ^(b)		6.13	7.43	8.10	8.43	8.75	8.92	9.23	
	$\Delta CTE/CTE_0$	+76.6	+85.8	+93.8	+88.6	+87.0	+85.1	+87.6	+86.3
63-16 1 (Unirradiated)		4.62	5.38	5.88	6.27	6.55	6.75	6.92	
63-16 1 (63-16-71)		1.60	1.93	2.15	2.42	2.58	2.80	2.94	
	$\Delta CTE/CTE_0$	-65.4	-64.1	-63.4	-61.4	-60.6	-58.5	-57.5	-61.6
TSGBF 1 (63-16-71) ^(b)		1.73	2.12	2.40	2.79	3.03	3.10	3.16	
	$\Delta CTE/CTE_0$	-62.6	-60.6	-59.2	-55.5	-53.8	-54.1	-54.3	-57.2
63-38 1 (Unirradiated)		4.62	5.00	5.22	5.42	5.53	5.57	5.89	
63-38 1 (63-38-60)		3.33	3.72	4.10	4.34	4.55	4.72	4.86	
	$\Delta CTE/CTE_0$	-27.9	-25.6	-21.5	-19.9	-17.7	-15.3	-17.5	-20.8
63-38 1 (63-38-60) ^(b)		4.00	4.28	4.60	4.86	5.17	5.38	5.54	
	$\Delta CTE/CTE_0$	-13.4	-14.4	-11.9	-10.3	-6.5	-3.4	-6.0	-9.4

(a) 25 °C to the temperature tabulated
(b) Annealed for 1 hr at 1700 °C.

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APPENDIX A

EXPERIMENTAL PROCEDURE

Capsule Design

The GEH-13-10 capsule used the same basic design as the H-3⁽¹⁾ and H-4, -5, -6⁽²⁾ capsules. The major difference was that the sample assembly was made up of six 1/6-round wedge-shaped samples. The other differences were in the type of end plugs and in the lead-out tubing. The capsule was instrumented with 12 thermocouples: 7 were Geminol N-Geminol P, 1 Chromel-Alumel, 2 platinum versus Pt-10 wt% Rh, and 2 W-5 wt% Re versus W-26 wt% Rh. Complete as-built blueprints are shown H-3-9653, Rev. 2.

Preirradiation Measurements

Lengths were measured physically and optically on all samples. See HW-81250A⁽²⁾ for a discussion of the methods and equipment for these and the other measurements taken. The thermal-pulse technique was used to measure the thermal conductivity.⁽¹²⁾

Installation and Irradiation

The GEH-13-10 capsule was installed in the K7 position of the ETR on January 22, 1964. Initially only 11 of the 12 thermocouples were operable as one of the W-Re couples failed prior to reactor startup. As the irradiation progressed, the thermocouples started failing. In all cases, the failure occurred after the temperature transient associated with a reactor scram. The remaining W-Re thermocouple, which is a very brittle material, failed first. By the end of the irradiation, a period of 7 mo, only the two Pt-Rh thermocouples were functioning.

The capsule was removed from the ETR on August 20, 1964, after completing 127.1 effective days of full-power reactor operation and was shipped to the hot cell for disassembly.

Disassembly

The disassembly procedure consisted of cutting off the capsule ends with a power hacksaw and then slitting the capsule shell longitudinally with a milling machine at two places 180° apart. As soon as the capsule was opened the cause for the thermocouple failures was apparent. The aluminum support rods between the aluminum cooling rings in the top-half of the capsule had warped due to a combination of high temperature and in-core capsule vibration. Since the thermocouples did not have enough slack they finally pulled out of the samples or broke apart. The Pt-Rh thermocouples apparently were ductile enough to stretch and consequently did not fail. They had pulled out of the samples and therefore the indicated temperatures were not true sample temperatures. Analysis of the thermocouple recorder charts showed at what time they had pulled out of the samples.

The movement of the upper cooling rings also closed up part of the gaps allowed for thermal expansion between the samples and sample holders at the top three sample assemblies. However, the gap remaining was approximately $1/32$ in. which would allow ample room for thermal expansion even at 1200°C so that the samples should not have been under a stress at operating temperatures. During removal of the samples from the holders, the ends of two samples, 125-23 and 187-2, snapped off. Several samples appeared to be somewhat bowed while 125-23 was appreciably bowed. Some carbon deposit was observed on the inner wall of the capsule shell especially at the lower end. Some carbon deposit was observed on the inner wall of the capsule shell especially at the lower end.

Thermocouple Calibration

The two Pt-Rh thermocouples were calibrated against a new Pt-Rh thermocouple after the capsule disassembly. One of the couples failed just after the calibration run began, but the other was still operable at 900 °C. The resulting calibration is shown on Figure A-1.

Sample Temperatures

The temperature difference shown in Figure A-1 was considered when the sample mean irradiation temperatures were determined. See Appendix A of HW-71500B⁽¹⁾ for a detailed discussion.

Postirradiation Measurements

All measurements were repeated on all samples after the irradiation. Since the ends of 125-23 and 187-2 had broken off these were measured from hole-to-hole. However, when 125-23 was set up in the jig (used to hold the samples for the hole-to-hole measurement) it snapped apart. The appearance of 125-23 after breaking is shown in the upper part of Figure A-2. The sample was then reassembled and the amount of bowing is shown in the lower part of Figure A-2. The amount of bow at the center is estimated to be 3/32 in. The general appearance of 33 of the samples is shown by a typical sample in Figure A-3. One of the other bowed samples, 63-38-60, is shown in Figure A-4. The bow is estimated to be 3/64 in.

Due to the breakage, sample 63-16-71 was not measured from hole-to-hole as it also was warped approximately 1/16 in. All other samples were measured both from end-to-end and from hole-to-hole.

FIGURE A1

Calibration of Irradiated Thermocouple
Platinum versus Pt-10 wt% Rh

FIGURE A2
Sample 125-23

FIGURE A3
Sample 142-79

FIGURE A4
Sample 63-38-60

APPENDIX BEXPOSURE DETERMINATION

The neutron exposures to the samples irradiated in the GEH-13-10 capsule were determined experimentally from the nickel and iron flux monitor foils contained in the capsule cooling rings. The methods and equations discussed in detail elsewhere^(1, 2) were again used for these exposures with one exception. The activation cross sections for both the $\text{Ni}^{58} (n, p) \text{Co}^{58}$ and $\text{Fe}^{54} (n, p) \text{Mn}^{54}$ reactions were revised, using the most recently available data. The Ni^{58} branching-ratio curve is shown in Figure B1 and the energy-dependent cross section for the Ni^{58} reaction in Figure B2. The Fe^{54} cross section is shown in Figure B3. The fluxes calculated from the nickel data are about 12% greater than those from the iron data. All exposures reported herein are based on the mean of the nickel and iron results.

FIGURE B1
Branching Ratio
 $\text{Ni}^{58} (n, p) \text{Co}^{58}$

FIGURE B2
Activation Cross Section
 $\text{Ni}^{58} (n, p) \text{Co}^{58}$

FIGURE B3
Activation Cross Section
 $\text{Fe}^{54} (n, p) \text{Mn}^{54}$

APPENDIX C

SUPPLEMENTAL FIGURES

The data on crystallite parameters, Young's modulus, electrical resistivity, and thermal conductivity are presented in Figures C1 through C15 so that comparisons among graphites may be easily observed. Isothermal lines are drawn through the data prints on most of the graphs to indicate estimates of the general trends.

Crystallite Parameters

The effects of irradiation on the crystallite parameter $\underline{c}$, the distance between alternate layer planes, are shown in Figures C1 and C2. There is little or no temperature dependence over the range of 800 to 1200 °C. CSF (Figure C1) shows an increase in $\underline{c}$ with exposure, KC stays nearly constant, and TSGBF shows a large increase. The raw-coke graphites show a relatively large change with the material having no binder (63-38) showing the greatest increase. The TSX graphite stays nearly constant and the EGCR increases slightly, Figure C2.

The data on apparent crystallite size in the $\underline{c}$ direction, L_c , are presented in Figures C3 and C4. As has been seen previously at lower irradiation temperatures, ^(1, 2) there is a decrease in L_c with exposure. In general, the data for the higher temperatures, 950 to 1200 °C, appear to decrease more than the 800 °C data. For the CSF graphite, the L_c isotherm at 950 to 1200 °C, apparently, is well below the 450 °C isotherm.

The effects of neutron exposure on the unit cell dimension (a) and the apparent crystallite size in the $\underline{a}$ direction (L_a) are presented in Figures C5 and C8. For all the graphites, $\underline{a}$ appears to decrease with neutron exposure. The KC graphite, Figure C5, shows the least change.

The apparent crystallite size L_a , Figures C7 and C8, also shows a general decrease. An exception is the raw-coke no-binder graphite, 63-38, which shows a very large increase.

FIGURE C1
 Crystallite Layer Spacing (c) of Conventional and Raw-Coke Graphites
 as a Function of Exposure

FIGURE C2

Crystallite Layer Spacing (c) of Needle-Coke Graphites as a Function of Exposure

FIGURE C3

Apparent Crystallite Size (L_c) of Conventional and Raw-Coke Graphites
as a Function of Exposure

FIGURE C4

Apparent Crystallite Size (L_c) of Needle-Coke Graphites as a Function of Exposure

FIGURE C5

Crystallite Layer Spacing (a) of Conventional and Raw-Coke Graphites
as a Function of Exposure

FIGURE C6
Crystallite Layer Spacing (a) of Needle-Coke Graphites as a Function of Exposure

FIGURE C7

Apparent Crystallite Size (L_c) of Conventional and Raw-Coke Graphites
as a Function of Exposure

FIGURE C8

Apparent Crystallite Size (L_a) of Needle-Coke Graphites as a Function of Exposure

Young's Modulus of Elasticity

The data on Young's modulus, E , as a function of neutron exposure are presented in Figures C9 through C11. The data for transverse samples of all the graphites except the raw-coke graphites are shown in Figure C9. For all the graphites, E increases rapidly and then tends to reach a constant value regardless of irradiation temperature. The conventional-coke graphites show less increase than the needle-coke types.

The data for the parallel samples of all the graphites except TSGBF and the raw-coke types are shown in Figure C10. For all types shown, E again increases rapidly and tends to a constant value, although there appears to be more dependence on irradiation temperature than for the transverse samples.

The data for the TSGBF parallel samples and for the raw-coke graphites are shown in Figure C11. The Young's modulus is considerably greater than for all the other graphites tested. The data for TSGBF appear to show a slight decrease of E with exposure. The raw-coke no-binder graphite, 63-38, shows an increase of several times and the other two raw-coke materials show increases of nearly an order of magnitude. The 63-16 material shows quite anisotropic behavior at 950 °C.

FIGURE C9

Young's Modulus of Graphite (Transverse) as a Function of Exposure

FIGURE C10

Young's Modulus of Graphite (Parallel) as a Function of Exposure

FIGURE C11

Young's Modulus of Raw-Coke Graphites as a Function of Exposure

Electrical Resistivity

The data on the effect of irradiation on electrical resistivity, ρ , are presented in Figures C12 and C13. In general, ρ increases significantly and remains at a constant value for all temperatures. There appears to be some temperature dependence for the parallel samples of CSF and TSGBF graphites (Figure C12). There is a significant difference between the raw-coke graphite containing no binder (63-38) and the other two raw-coke graphites, Figure C12.

FIGURE C12

Electrical Resistivity of Conventional and Raw-Coke Graphites as a Function of Exposure

FIGURE C13

Electrical Resistivity of Needle-Coke Graphites as a Function of Exposure

Thermal Conductivity

Thermal conductivity as a function of temperature was measured on some samples by the thermal-pulse method.⁽¹²⁾ As observed previously⁽¹²⁾ the thermal conductivity decreases to a nearly constant value over the temperature range of 25 to 700 °C. The results for the samples measured are presented in Figures C14 and C15.

FIGURE C14

Thermal Conductivity of Graphite as a Function of Temperature

FIGURE C15

Thermal Conductivity of Graphite as a Function of Temperature

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