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GRAPHITE IRRADIATIONS IN EBR-II:
CAPSULES BG-1, BG-2, AND BG-3

By

A. L. Pitner

Materials Research Section
Materials Department

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CAPSULES BG-1, BG-2, AND BG-3

A. L. Pitner

ABSTRACT

Nine grades of graphite were irradiated in the EBR-II reactor from 400 to 700 °C to maximum exposures of 8×10^{21} nvt ($E > 0.18$ MeV). Three isotropic graphites showed good dimensional stability in the irradiations. Apparently, there is no adverse difference in the behavior of graphite irradiated in fast or intermediate reactors. The excellent agreement obtained between exposure calculated from the activation of nickel and iron flux monitors attests to the accuracy of the monitor cross sections and calculated neutron spectrum employed in determining the exposures.

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INTRODUCTION

The extensive use of graphite in nuclear applications requires that its irradiation behavior be well defined so that property changes, particularly dimensional instabilities, may be incorporated into reactor designs. At Pacific Northwest Laboratory (PNL), a continuing program is being conducted to characterize the irradiation behavior of many different types of graphite and to identify those properties and manufacturing techniques which lend themselves to improved irradiation stability. Generally, high-exposure data are required to indicate trends in the irradiation behavior of graphite. This entails a considerable amount of irradiation time in thermal or intermediate reactors. A fast reactor such as the EBR-II, however, is rich in the high-energy neutrons which cause the radiation damage in graphite, and exposure can be accumulated rapidly. A program was undertaken to irradiate several types of experimental graphites in the EBR-II to high exposures. The types of graphite included in the irradiation were selected because of promising low-exposure behavior,⁽¹⁾ or because of some major variation in their manufacture.

It was unknown whether the high intensity fast flux environment of EBR-II would cause the graphite to exhibit different behavior than shown in intermediate test reactors used previously. The fast flux in EBR-II is of the order of 10^{15} n/cm²-sec, as compared to a typical value of 4×10^{14} n/cm²-sec ($E > 0.18$ MeV) available in the ETR intermediate reactor. It has been postulated that damage could possibly

accumulate much faster than it is annealed out in a high intensity neutron spectrum and, therefore, accelerate the damage process. To test this hypothesis, several conventional graphites for which a large amount of data have been previously collected in intermediate flux reactors were included in the irradiations.

SUMMARY

Nine grades of graphite were irradiated in the EBR-II reactor at temperatures from 400 to 700 °C to maximum exposures of 8×10^{21} nvt ($E > 0.18$ MeV). The dimensional stability of three isotropic graphites appears particularly good at the exposures achieved in the irradiations. An apparent anomaly between fast-reactor and intermediate-reactor irradiation results is attributed, at least partly, to the value of the lower energy limit that was utilized in the exposure calculations. Consequently, it appears there is no adverse difference between fast and intermediate reactor irradiation behavior of graphite. Excellent agreement is obtained between exposures calculated from the activation of nickel and iron flux monitors, which attests to the accuracy of the monitor cross sections and calculated neutron spectrum employed in determining the exposures.

EXPERIMENTAL PROCEDURE

The graphite types utilized in the irradiations are described in Table I. Samples cut from the molded or extruded bars were machined into cylinders 0.25 in. diam x 1.75 in. long. Orientations of the samples as cut from the large bars are shown as $\perp$ for transverse and $\parallel$ for parallel, depending on whether they were cut transverse or parallel to the extrusion axis, respectively. The ends of the samples were polished spherical. All samples were annealed at approximately 1600 °C to relieve any strains induced by machining. Measurements of length,

TABLE I. Graphite Description

Block Number	Sample Designation	Manufacturer	Coke Source and Type	Density	CTE /CTE		Remarks
					I	II	
NC-8	EGCR	UCC	Continental-Lake Charles (needle type)	1.70 g/cm ³	2.2		Reactor grade: AGOT; EGCR graphite. Maximum particle size 1/32 in. Coal tar pitch binder. One coal tar pitch impregnation. Graphitized at >2800 °C. Extruded.
233	CSF	UCC	Cleves (Conventional type)	1.66 g/cm ³	2.3		Barrett medium #30 coal tar pitch binder, no additives, one coal tar pitch impregnation. Halogen purification. Graphitized at 2800 °C. Extruded.
234	TSX	UCC	Continental-Lake Charles (needle type)	1.71 g/cm ³	3.9		Allied medium #30 coal tar pitch binder, one pitch impregnation. No additives. Halogen purification. Graphitized at 3000 °C. Extruded. Very anisotropic.
63-16	Raw-Coke Base Stock	GLCC	Texas Lockport (raw coke)	1.72 g/cm ³	1.02		Experimental grade. Volatile content of raw coke 12%. Coal tar derivative binder. Amount of binder carbon in baked product, negligible. Graphitized at 2500 °C. No impregnation. Molded. Very isotropic.
64-17	H315	GLCC	Texas Lockport (raw coke)	1.53 g/cm ³	1.3		Coal tar pitch binder. No additives. Particle sizing: (-48 mesh 98.1%) (-200 mesh 51.0%). No impregnation. Graphitized at 2500 °C. Molded. Graphitized "binder" coke in final product: none.
65-18	JO2	GLCC	Petroleum (raw coke)	1.72 g/cm ³	1.2		Commercial grade. Coal tar pitch derivative binder. One coal tar pitch impregnation. Graphitized at 2800 °C. Molded.
65-32	H319	GLCC	Petroleum (raw coke)	1.78 g/cm ³	1.1		Low CTE. Coal tar pitch binder. Special forming method. No impregnation. Graphitized at 2800 °C.
65-42	AXF-8Q1	POCO	Proprietary	1.79 g/cm ³	1.0		Fine grain. Binder not coal tar pitch. Isostatic mold. No impregnation. Halide purification. Graphitized at 2800 °C. High strength. High CTE.
66-6	DS-13	Pure		1.82 g/cm ³	1.4		No additives. Halide purification. No impregnation. Isostatic mold. Graphitized at <2800 °C.

coefficient of thermal expansion (CTE) (25 to 425 °C), Young's modulus (E), and electrical resistivity (ρ) were made on each sample before and after irradiation.

Eighteen samples were loaded into each capsule as shown in Figure 1. The samples were separated by graphite spacers 0.25 in. diam x 0.25 in. long. The spacers, which also contained the flux monitors and melt wires, were indented on the ends so the sample ends would not wear during irradiation. Samples and spacers were doubly encapsulated in stainless-steel tubes of 0.300 in. diam x 0.020 in. wall and 0.375 in. diam x 0.020 in. wall. Overall length of each capsule was 38 in.

FIGURE 1. *Irradiation Capsule Assembly*

Three capsules, BG-1, BG-2, and BG-3 were shipped to the EBR-II for irradiation on a space-available basis; i.e., when space was available in another experimenter's 19-pin subassembly. Hence, in-reactor time was dictated by the schedule of the primary experimenter. Capsule BG-1 was included in a subassembly in a Row-3 position, and BG-2 and BG-3 were irradiated in a subassembly in a Row-2 position. After discharge, the capsules were disassembled at the National Reactor Testing Station (NRTS) hot cell. Samples and flux monitors were shipped to PNL for postirradiation analysis.

IRRADIATION TEMPERATURES

Sample temperatures were calculated with the aid of a computer heat-transfer program, STHTP.⁽²⁾ The heat-transfer model is divided into nodes, and the program solves the heat-transfer equations for each node by an iterative process. Gamma heating values used in the calculations were 5.7 W/g for the Row-3 position and 6.5 W/g for the Row-2 position. Inspection of melt wires contained in the sample spacers after the irradiation confirmed the values of the calculated temperatures.

EXPOSURE DETERMINATION

Flux monitors used in the capsules were iron, nickel, and 0.1% cobalt contained in aluminum. The cobalt activation is generally used to correct thermal-neutron burnup of ^{58}Co in the $^{58}\text{Ni}(n,p)^{58}\text{Co}$ reaction. However, it was not necessary to make use of this correction because there is essentially no thermal neutron flux in the EBR-II core, as determined from spectrum calculations and cobalt activation. The exposures were calculated by using the computer program FOILS⁽³⁾ which combines the monitor activation with its effective activation cross section and total irradiation time to compute the neutron dose. The effective activation cross section of each flux monitor is obtained from

$$\sigma_{\text{eff}} = \frac{\int_0^{\infty} \sigma(E) \phi(E) dE}{\int_{E_L}^{\infty} \phi(E) dE} \quad (1)$$

where $\sigma(E)$ and $\phi(E)$ are the monitor cross section and calculated neutron flux as a function of energy, respectively. The value is consequently dependent upon how accurately the cross sections and calculated flux spectrum are known. The

value of E_L usually chosen as 0.183 MeV (lethargy value of 4) seems to produce inaccuracies of less than 10% in the radiation damage analysis in graphite. That is, if only neutrons with energy greater than 0.183 MeV are considered in calculating the damaging exposure, then irradiation results from different reactor spectrums seem to agree well. The values of the total flux calculated by the above technique for capsules BG-2 and BG-3 are shown in Figure 2, as deduced from the $^{54}\text{Fe}(n,p)^{54}\text{Mn}$ and $^{58}\text{Ni}(n,p)^{58}\text{Co}$ monitor reactions. The excellent agreement between the two types of monitors attests to the accuracy of the values of $\sigma(E)$ and $\phi(E)$ used in the analysis.

RESULTS AND DISCUSSION

The effects of the irradiations on the coefficient of thermal expansion, Young's modulus, electrical resistivity, and length are shown for all samples in Table II. CTE values, in general, changed only slightly with irradiation until the higher exposures ($>5 \times 10^{21}$ nvt) were attained, whereupon a consistent decrease in CTE was evidenced in all graphite types. Young's modulus increased with exposure, generally to not more than twice the original value. This increase in modulus seems to saturate rapidly. Electrical resistivity of all samples doubled or tripled with exposure, the effect again saturating very early in the irradiations.

The radiation-induced length changes of all graphites are shown in Figures 3, 4, and 5 for capsules BG-1, BG-2, and BG-3, respectively. A parallel and transverse sample of each graphite type was included in each capsule. Because the flux drops off very fast vertically in the EBR-II core, only samples near the center received significant exposure. Capsule BG-1 accumulated less exposure than BG-2 and BG-3 because of shorter in-reactor time. Comparison of the dimensional stability of

FIGURE 2. Total Flux for BG-2 and BG-3 Calculated from Fe and Ni Activation

TABLE II. Irradiation Results*

Type	Orientation	Exposure [nvt x 10 ⁻²¹ (E > 0.18 MeV)]	Temperature (°C)	CTE _o (in./in./°C x 10 ⁶)	CTE _{irr}	E _o (psi x 10 ⁶)	E _{irr}	ρ _o (ohm cm x 10 ⁴)	ρ _{irr}	$\frac{\Delta L}{L}$
NC8	⊥	1.0	400	3.12	2.73	0.78	1.16	11.0	31.9	-0.10
NC8	⊥	2.4	650	3.35	3.33	0.67	1.22	10.8	32.3	-0.44
NC8	⊥	5.2	625	3.48	3.82	0.80	1.71	10.9	31.3	-1.16
NC8		0.1	400	1.63	1.34	1.69	2.68	6.1	19.2	-0.03
NC8		3.6	450	1.60	1.50	0.95	2.85	6.7	22.0	-1.00
NC8		7.2	675	1.64	1.26	1.58			21.3	-2.64
234	⊥	0.3	400	3.46	3.50	0.64	1.54	13.3	32.7	-0.02
233	⊥	3.0	675	3.71	3.45	0.64	1.37	12.7	34.3	-0.53
233	⊥	3.3	450	3.72	3.79	0.65	1.53	12.8	33.7	-0.76
233		0.02	400	1.89	1.67	1.60	2.41	8.1	19.9	-0.00
233		4.7	625	1.74	1.71	1.69	3.65	7.4	21.8	-1.73
233		8.3	700	1.77	1.40	1.47	3.36	7.7	22.3	-3.52
234	⊥	0.8	400	4.36	3.95	0.64	1.32	14.9	37.7	-0.08
234	⊥	1.5	600	4.38	4.13	0.68	1.21	14.6	38.6	-0.26
234	⊥	7.9	700	4.31	4.22	0.70	1.42	13.8	35.6	-0.73
234		0.4	400	1.28	0.95	2.16		6.1	17.2	-0.04
234		1.0	450	1.11	0.71	2.11	3.89	5.7	18.4	-0.26
234		7.1	675	1.15	0.55	1.41		5.7	18.3	-3.01
63-16	⊥	1.8	600	6.76	6.89	1.51	2.07	15.2	28.9	-0.07
63-16	⊥	6.6	650	6.40	6.14	1.48	2.11	15.3	27.2	-0.64
63-16	⊥	8.3	700	6.89	4.12	1.72	2.26	16.0	27.0	-0.89
63-16		0.4	400	6.19	6.46	1.70	1.99	14.9	28.9	+0.01
63-16		4.6	575	6.25	6.36	2.24	2.08	14.8	27.8	-0.43
63-16		8.0	675	7.11	4.01	1.89	2.33	14.9	25.8	-1.19
64-17	⊥	0.2	400	6.67	6.30	1.77	1.26	14.5	34.3	+0.03
64-17	⊥	0.5	400	5.98	6.02	1.33	1.32	13.9	31.9	-0.01
64-17	⊥	5.7	625	6.54	6.19	1.32	1.48	13.9	32.9	-0.74
64-17		0.3	400	4.85	4.82	1.19		11.1	27.2	-0.01
64-17		0.7	400	4.33	3.98	1.35	1.79	11.0	25.9	-0.03
64-17		8.0	675	5.02	4.00	1.21	1.81	11.5	27.2	-2.14
65-18	⊥	0.6	400	6.97	6.90	1.95	2.09	12.8	27.5	+0.03
65-18	⊥	2.1	625	6.56	7.12	1.76	2.29	13.9	29.1	-0.05
65-18	⊥	7.7	675	6.76	5.89	1.84	2.19	13.6	26.3	-0.22
65-18		0.5	400	5.77	5.78	1.58	2.24	11.8	24.0	+0.00
65-18		1.3	550	5.77	5.57	1.53	1.25	12.4	26.5	-0.11
65-18		2.2	400	5.99	5.93	1.64	2.39	11.8	25.0	-0.18
65-32	⊥	0.06	400	3.72	3.75	1.54		9.5	21.9	-0.02
65-32	⊥	1.5	400	3.84	3.07	0.59		9.9	23.5	-0.28
65-32	⊥	7.6	675	3.58	3.50	0.69		9.3	22.8	-2.12
65-32		0.04	400	3.31	3.21	2.10		8.4	19.2	-0.01
65-32		1.2	400	3.33	3.35	1.62		8.7	21.3	-0.20
65-32		5.7	625	3.43	3.25			8.8	21.9	-1.69
65-42	⊥	1.3	400	6.81	7.37	2.08	2.33	15.5	28.6	-0.00
65-42	⊥	2.0	400	7.36	7.59	1.56	2.36	15.5	28.5	-0.01
65-42	⊥	2.9	650	7.50	7.48	1.60	2.31	15.5	28.6	+0.08
65-42		1.7	400	6.92	7.57	2.09		14.9	27.8	-0.01
65-42		3.1	675	7.01	6.94	1.92		15.9	27.6	+0.09
65-42		7.3	650	7.02	6.11	2.14	2.80	15.5	25.5	+0.10
66-6	⊥	0.6	425	5.13	5.60			17.4		-0.15
66-6	⊥	2.6	425	5.37	5.89			16.9		-0.46
66-6	⊥	2.8	425	5.05	6.20			16.7		-0.48
66-6		2.6	650	3.98	4.21			13.8	22.7	-0.63
66-6		4.2	575	3.79	4.27	2.07		13.8	22.1	-1.16
66-6		7.9	700	3.98	3.22	2.07		13.7	21.5	-2.15

* Blank fields indicate that the measurement could not be obtained.

FIGURE 3. Radiation Induced Length Change - Capsule BG-1

FIGURE 4. Radiation Induced Length Change - Capsule BG-2

FIGURE 5. Radiation Induced Length Change - Capsule BG-3

the different graphite types may be made from the figures. Previously, it has been common to compare the irradiation behavior of experimental graphites against that of CSF (Block No. 233), a conventional nuclear graphite. Experimental grades 63-16, 64-17, 65-18, and 65-42 display less contraction than CSF, while grades 65-32 and 66-6 appear to contract at about the same rate as CSF. Types 63-16, 65-18, and 65-42 behaved quite isotropically and showed the least contractions. Type 65-42, the most isotropic of the graphites irradiated, actually expanded slightly and retained this small expansion to the highest exposures attained.

Figures 6, 7, and 8 show comparisons of the behavior of conventional nuclear graphites CSF, NC8, and TSX irradiated in the EBR-II and in intermediate flux reactors (GETR and ETR). The curves represent data accumulated from irradiations performed in intermediate flux reactors, and the points represent the EBR-II irradiation results. The curves for TSX had to be extrapolated so that a comparison could be made. The arrows indicate where, for agreement, the points should fall on the curves. In nearly every case, the length changes appear to occur faster in the EBR-II.

In general, irradiation in EBR-II seems to induce the same damage at exposures about 20% less than those required in intermediate flux reactors. This would infer that damage does occur at an accelerated rate in a fast reactor.

Part of the difference noted above, however, can be attributed to the E_L value used in the calculations to determine the fast neutron exposures. As stated previously, E_L is generally chosen as 0.183 MeV for convenience (lethargy value of 4) and usually produces discrepancies of less than 10%. However, a more optimum value of E_L for carbon has been shown to be 0.065 MeV.⁽⁴⁾

FIGURE 6. Comparison of Irradiation Behavior of CSF Graphite in EBR-II and Intermediate Flux Reactors

FIGURE 7. Comparison of Irradiation Behavior of NCS Graphite in EBR-II and Intermediate Flux Reactors

FIGURE 8. Comparison of Irradiation Behavior of TSX Graphite in EBR-II and Intermediate Flux Reactors

Since there is a greater proportion of neutrons between 0.065 and 0.183 MeV in EBR-II than in an intermediate reactor, neglecting these neutrons would tend to underestimate the damaging neutron exposure more in EBR-II than in an intermediate reactor. The ratio of the neutron flux greater than 0.065 MeV to the flux greater than 0.183 MeV in EBR-II is 1.21. Although the flux analysis of the intermediate reactors utilized in graphite irradiations is not in a form that will allow calculation of this ratio, it would certainly be less than 1.21. The ratio for the water-moderated intermediate reactor PLUTO in England is 1.15. Consequently, if the E_L value of 0.065 MeV were used in determining the EBR-II exposures, better agreement with intermediate reactor data would result.

CONCLUSIONS

It has been demonstrated that irradiation of graphite in a core position in the EBR-II results in the accumulation of valuable high-exposure data at a rapid rate. Equal fast neutron exposures are attained about three times faster in the EBR-II than in the ETR. Full utilization of this facility is limited, however, because of the low priority of graphite when the highly demanded space in the EBR-II core is assigned.

Graphite Types 63-16, 65-18, and 65-42 showed good dimensional stability in the irradiations. All behaved quite isotropically and displayed little length change. Samples from Block 65-42 appeared to be particularly stable to high exposures, at least at the temperatures achieved in these irradiations.

Comparison of data obtained in these EBR-II irradiations to data accumulated in intermediate flux reactors suggests that damage in graphite may occur at an accelerated rate in a high intensity fast flux reactor. However, at least part of the apparent 20% exposure discrepancy can be attributed to the use of 0.183 MeV as the lower energy limit value in calculating the

damaging neutron exposures. Utilization of the more accurate value of 0.065 MeV for the lower energy limit would definitely reduce the disagreement. It is concluded that if any high intensity flux effect exists, it is not of great magnitude. Graphite irradiations currently being conducted in the EBR-II to very high exposures will provide additional information in resolving this question.

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