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TITLE

RADIATION-INDUCED DIMENSIONAL CHANGES OF GRAPHITE
EFFECT OF SAMPLE SIZES LESS THAN ONE-HALF INCH DIAMETER

AUTHOR

J. W. Helm

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EFFECT OF SAMPLE SIZES LESS THAN ONE-HALF INCH DIAMETER

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PRELIMINARY REPORT

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RADIATION-INDUCED DIMENSIONAL CHANGES OF GRAPHITE
EFFECT OF SAMPLE SIZES LESS THAN ONE-HALF INCH DIAMETER

INTRODUCTION

The amount of radiation-induced dimensional change in graphite appears to depend on the size of the sample tested. A difference in irradiation behavior has been observed between 0.426 in. diameter, 4 in. long samples and 4 x 4 x 24 in. blocks⁽¹⁾ and also between these samples and 0.262 in. diameter, 1-3/4 in. long samples.⁽²⁾ The existence of a size effect influences the applicability of sample data⁽³⁻⁵⁾ to nuclear reactor conditions. In comparing reactor distortion measurements with sample contractions the best correlations have been made using data from the large blocks rather than the small samples.⁽⁶⁻⁸⁾ However, for irradiation experiments, particularly in high flux test reactors, samples are generally limited to a maximum size of about 1 in. diameter. In most experiments even smaller sizes are used so as to increase the sample inventory and thus make the irradiations more economically attractive.

To determine if a sample size effect exists for the small samples used in high flux irradiations, a series of non-instrumented capsules⁽⁹⁾ were irradiated in the Engineering Test Reactor (ETR). The study was limited to eight sample types so that the standard non-instrumented capsule⁽⁹⁾ could be used for the irradiations. The samples varied from 1/16 in. to 17/32 in. diameter, from 1-1/4 to 4 in. in length, and from cylindrical to quarter cylindrical geometries. This study supplements more extensive studies being conducted on a wider range of sample sizes under lower flux conditions.⁽¹⁰⁻¹²⁾

SAMPLE SELECTION AND PREPARATION

The samples were taken from a full-size extrusion of TSX graphite⁽¹³⁾ as shown in Figure 1. All 1/16 and 1/8 in. diameter cylindrical samples came from the "C" locations.

- I TRANSVERSE SAMPLES
- II PARALLEL SAMPLES
- S SOLID 1/2 ROUND
- B BOAT 1/2 ROUND

FIGURE 1
SAMPLING SCHEME

After machining the individual samples were annealed at 1800°C for 1 hr. at a pressure of 3×10^{-4} torr. In this study the standard sample for non-instrumented capsules 4 in. long, 1/4-round,⁽⁹⁾ was modified so that various shapes and lengths could be used. The shapes and sizes studied and their respective cross-sectional areas are described in Table I. A set of typical samples may be seen in the following section.

TABLE I
SAMPLE DESCRIPTION

<u>Sample Shape</u>	<u>Radius, in.</u>	<u>Length, in.</u>	<u>Cross-sectional Area^(b) in.²</u>	<u>Equivalent Dia.^(d) in.</u>
Quarter round ^(a)	3/8	4	0.1104	3/8
Quarter round	3/8	2	0.1104	3/8
Half round	3/8	4	0.2209	17/32
Half round hollowed out boat	3/8	4	0.0967 ^(c)	11/32
Cylinder	1/16	2-1/8	0.0123	1/8
Cylinder	1/16	1-1/4	0.0123	1/8
Cylinder	1/32	2-1/8	0.0031	1/16
Cylinder	1/32	1-1/4	0.0031	1/16

(a) Basic sample for non-instrumented capsules.

(b) Calculated from the major sample radius.

(c) Annular ring.

(d) Diameter to the nearest 1/32-in. of the cylinder having the same cross-sectional area as the sample tested.

EXPERIMENTAL PROCEDURE

The basic non-instrumented capsule is discussed in detail elsewhere.⁽⁹⁾ The different sample shapes used in this series of capsules were assembled into the standard 4 in. long cylinder.

The six capsules were installed in the ETR and a complete capsule history is given in Table II.

TABLE II
CAPSULE HISTORY

<u>Capsule Number</u>	<u>ETR Position</u>	<u>ETR Elevation, in.</u>	<u>Installation Date</u>	<u>Removal Date</u>	<u>Effective Days*</u>
GEH-13-209	E5 SW	9 to 15	10-3-63	12-8-63	43.4
GEH-13-210	N5 SW	24 to 30	"	"	43.4
GEH-13-211	K7 SW	10 to 16	"	1-19-64	71.8
GEH-13-212	K7 SW	24 to 30	"	"	"
GEH-13-213	E5 NE	9 to 15	"	"	"
GEH-13-214	N5 NW	12 to 18	"	"	"
GEH-13-215	K7 SW	30 to 36	"	"	"
GEH-13-216	K7 SW	18 to 24	"	"	"

*Full power operation at 175 MW

The capsules were returned to BNW and disassembled.⁽⁹⁾ The 1/16 in. diameter samples were all so badly warped after the irradiation they could not be measured. The appearance of the irradiated samples from capsule 215 is presented in Figure 2. The 1/8 in. diameter samples showed some warping also, but were measurable.

Sample irradiation temperatures, which are estimated to be $\pm 100^{\circ}\text{C}$, are determined by heat-transfer calculations⁽¹⁴⁾ and confirmed by comparison of reference TSX length data with data obtained from instrumented capsule irradiations.⁽⁴⁾

EXPOSURE DETERMINATION

Neutron exposures are based on the foils of nickel and iron contained in each capsule. These are used in conjunction with the activation cross

FIGURE 2
IRRADIATED SAMPLES

sections for each material and neutron-spectra calculations for the appropriate capsule position. All exposures are based on the mean of the nickel and iron results.⁽³⁻⁵⁾

IRRADIATION RESULTS

Length-change results are presented in Table III and plotted in Figure 3. For all capsules the TSX standard 1/4 round sample data agreed quite well with the TSX 1/4 round reference data.⁽⁴⁾ From the temperature curves it is apparent that capsule 215 operated at the lower end of the 500-600°C temperature range. Since comparisons between the 2 in. and 4 in. long samples in the same capsule were required, the neutron exposures were corrected for the axial flux profile in the appropriate reactor position.

The transverse samples larger than 1/8 in. diameter all showed about the same irradiation behavior within a spread of $\pm 0.03\%$. However, the 1/8 in. cylinders showed larger contractions than the other transverse samples. At the higher exposures they contracted nearly as much as the parallel samples of the other sizes, indicating contraction about twice the other transverse samples.⁽⁴⁾ At the lower exposures, the 2-1/2 in. long samples contracted less than the 1-1/4 in. long samples, but at the higher exposures the contractions were about the same.

In the parallel orientation the samples larger than 1/8 in. diameter all showed about the same irradiation behavior. The 1/8 in. diameter by 1-1/4 in. long parallel samples also showed about these same length changes. However, the 2-1/2 in. long samples showed much less contraction than the other sizes. Comparing only the 1/8 in. diameter cylinders, the 2-1/2 in. long parallel samples contracted much less than all the others, which, with the exception of one transverse sample, E, contracted about the same amount.

In general it appears that the samples taken from the center of the block, indicated by C, contract nearly the same as those taken from nearer

EFFECT OF SAMPLE SIZE

Capsule Number	Temperature, °C	Exposure nvt x 10 ⁻²¹ (E > 0.18 MeV)	Orientation	Sample Number	Sample Shape	$\Delta L/L, \%$
209	400-500	0.40	⊥	184-E17	1/4-rnd, 4"	-0.014
		0.44	⊥	184-C1	1/4-rnd, 2"	-0.015
		0.35	⊥	184-M1	1/4 rnd, 2"	+0.001
		0.40	⊥	184-ST1	1/2 rnd, 4"	-0.001
210	500-600	0.80	⊥	184-E18	1/4-rnd, 4"	-0.087
		0.74	⊥	184-C2	1/4-rnd, 2"	-0.079
		0.86	⊥	184-M2	1/4-rnd, 2"	-0.093
		0.80	⊥	184-ST2	1/2-rnd, 4"	-0.066
211	650-750	2.40		184-219	1/4-rnd, 4"	-0.461
		2.16		184-C201	1/4-rnd, 2"	-0.482
		2.64		184-M201	1/4-rnd, 2"	-0.528
		2.40		184-SP1	1/2-rnd, 4"	-0.479
212	500-600	2.45		184-220	1/4-rnd, 4"	-0.800 ^b
		2.25		184-C202	1/4-rnd, 2"	c
		2.70		184-M203	1/4-rnd, 2"	-0.806
		2.45		184-SP2	1/2-rnd, 4"	c
213	500-600	0.75	⊥	184-E19	1/4-rnd, 4"	a
		0.66	⊥	184-C3	1/4-rnd, 2"	a
		0.75	⊥	184-M3	1/4-rnd, 2"	+0.045 ^a
		0.81	⊥	184-BT1	1/2-rnd, boat, 4"	b
		0.75	⊥	A	1/8 dia, 2-1/2"	-0.391
		0.75	⊥	B	1/8 dia, 1-1/4"	-0.477
		0.75		C	1/8 dia, 2-1/2"	broken
		0.75		D	1/8 dia, 1-1/4"	a
214	500-600	0.62	⊥	184-E20	1/4-rnd, 4"	-0.101
		0.67	⊥	184-C4	1/4-rnd, 2"	-0.047 ^a
		0.67	⊥	184-M4	1/4-rnd, 2"	-0.067 ^a
		0.62	⊥	184-BT2	1/2-rnd boat, 4"	-0.094
		0.62	⊥	E	1/8 dia, 2-1/2"	-0.106
		0.62	⊥	F	1/8 dia, 1-1/4"	-0.235
		0.62		G	1/8 dia, 2-1/2"	-0.085
		0.62		H	1/8 dia, 1-1/4"	-0.226
215	500-600	2.50		184-221	1/4-rnd, 4"	-0.798
		2.12		184-C203	1/4-rnd, 2"	-0.644 ^b
		2.88		184-M202	1/4-rnd, 2"	-0.844
		2.50		184-BP1	1/2-rnd, boat, 4"	-0.850
		2.50	⊥	I	1/8 dia, 2-1/2"	-0.662
		2.50	⊥	J	1/8 dia, 1-1/4"	-0.652
		2.50		K	1/8 dia, 2-1/2"	-0.385
		2.50		L	1/8 dia, 1-1/4"	-0.631
216	650-750	3.50		184-222	1/4-rnd, 4"	-0.810
		3.50		184-C204	1/4-rnd, 2"	-0.705
		3.50		184-M202A	1/4-rnd, 2"	-0.830
		3.50		184-BP2	1/2-rnd, boat, 4"	-0.877
		3.50	⊥	M	1/8 dia, 2-1/2"	-0.859
		3.50	⊥	N	1/8 dia, 1-1/4"	-0.798
		3.50		O	1/8 dia, 2-1/2"	-0.324
		3.50		P	1/8 dia, 1-1/4"	-0.862

^aWarped

^bEnds partially eroded

^cEnds badly eroded

FIGURE 3

PHYSICAL DISTORTION OF SMALL SIZED GRAPHITE SAMPLES
AS A FUNCTION OF EXPOSURE

the edge of the block, indicated by M. This agrees with previous data which indicated no difference in contraction behavior between these locations.⁽⁴⁾

SUMMARY AND CONCLUSIONS

Six capsules were irradiated in the ETR to determine the effect of sample size on the dimensional behavior of graphite under neutron irradiation. Sample temperatures ranged from 400-800°C and the maximum neutron exposure was about 4×10^{21} nvt, $E > 0.18$ MeV. For samples having equivalent diameters between 11/32 in. and 17/32 in., there appears to be no effect of sample size on dimensional behavior. However, the 1/8 in. diameter samples show a decided effect with the transverse samples showing somewhat more and the longer parallel samples less contraction than the other sizes. This partially agrees with previous data, wherein 4 x 4 x 24 in. bars were found to contract about twice as much as 0.426 in. diameter by 4 in. long samples in both orientations,⁽¹⁾ and these samples were observed to contract at a rate 20 to 30% greater than 0.262 in. diameter by 1-3/4 in. long samples^(2,15) in the transverse direction. Consequently, the 2-1/2 in. long 1/8 in. diameter parallel samples in the present study appear to agree with these data and the transverse samples to contradict them.

There is no apparent reason that any of the present data should be invalid with the possible exception of the warpage observed in some cases. The warpage was so severe in the 1/16 in. diameter samples they could not be measured. The bowing is most likely due to differential stresses built up during irradiation. Possibly part of these stresses were induced by temperature gradients, but most were probably caused by relief of internal stresses during neutron irradiation. There was little or no mechanical restraint of the samples so that external stresses were quite small.

The difference in behavior for the 1/8 in. diameter samples is most likely due to differences in relief of internal stress as has been speculated

previously.⁽¹⁾ The stresses generated in the bar as it cools after graphitization may be relieved by removal of the small samples from the bar. The fact that the 2-1/2 in. long parallel cylinders contracted so much less than the others could possibly be explained by the effect of differences in flow line structure along the section of the bar from which the samples were taken, although it would be expected that there would be more effect of flow lines in the transverse samples.

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