**RADIATION STUDIES ON GRAPHITE
WITH CONTROLLED PROCESSING**

J. H. COX, JR.

AUGUST, 1967

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RADIATION STUDIES ON GRAPHITE
WITH CONTROLLED PROCESSING

By

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Ceramics and Graphite Research Section
Materials Department

August, 1967

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ABSTRACT

This investigation explored the effect of graphitization temperature, crystallite orientation, impregnation, and density on the dimensional stability of graphite under neutron irradiation. The irradiations in a Hanford test facility accumulated exposures up to 2×10^{21} nvt ($E > 0.18$ MeV) at temperatures in the range 550 to 700 °C.

The investigation of the effect of graphitization temperature involved five series of samples, with final processing temperatures of 700 to 3000 °C. Increased processing temperature resulted in an appreciably lower radiation induced contraction rate; however, the most pronounced effect was found at temperatures below 2500 °C.

In graphites identical except for crystallite orientation a linear relationship was found between coefficient of thermal expansion values and contraction rates. A higher coefficient of thermal expansion resulted in a lower contraction rate.

In graphites identical except in impregnant level, increasing the impregnant level above a threshold, 16% for the series studied, resulted in the contraction rate increasing with impregnant level.

Increasing the density of graphite did not affect the radiation induced dimensional changes.

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RADIATION STUDIES ON GRAPHITE
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INTRODUCTION

A number of process variables exist which are controllable during graphite manufacture. This investigation explores the effects of several of these variables upon the dimensional stability of graphite during neutron irradiation. Variables investigated are graphitization temperature, crystallite orientation, impregnation level, and density. We attempted to hold all variables constant except for the one being studied.

The graphitization temperatures were varied for graphitic materials made from needle coke, non-needle coke, or from a pre-graphitized coke and non-graphitic material such as glassy carbon and carbon black filler material; graphitization temperatures ranged from 700 to 3500 °C in the extreme cases. The crystallite orientation was varied in a sample set by utilizing different forming techniques to yield coefficient of thermal expansion values ranging from 0.5 to 3.0×10^{-6} °C. A sample set was impregnated with coal-tar pitch and with thermosetting resin to levels from 1.0 to 21.0 wt% impregnant. A density series was investigated ranging from 1.3 to 1.7 g/cc.

These studies aim to provide data which help to define the damage mechanism in nuclear graphite and to lead to the manufacture of a product more stable during neutron irradiation.

SUMMARY AND CONCLUSIONS

GRAPHITIZATION TEMPERATURE

Increasing the graphitization temperature and, therefore, the degree of graphitization, as determined by crystallite layer spacing and/or ordering, results in a lower contraction rate for all reported temperatures. The increase in temperature becomes less effective above 2500 °C; however, there is an appreciable reduction in contraction rate to 3000 °C. The reduced contraction with increased processing temperature is independent of coefficient of thermal expansion values.

CRYSTALLITE ORIENTATION

In graphites identical except in the relative orientation of the crystallites within the graphite matrix, one may predict contraction rates under neutron irradiation for different orientations based on contraction rate data for any one orientation. Although the calculated single-crystal dimensional changes found in this study are less than those found in pyrolytic graphite, these data are quantitatively consistent with a single-phase model.

IMPREGNATION

The impregnant level has a definite effect on the contraction rate of graphite under neutron irradiation. At higher impregnant levels, approximately 16 percent for the graphite studied in this work, the contraction rate increases with impregnant level. A plausible explanation is that at low impregnant levels the open pore volume is preferentially filled, and there is no effect on the dimensional behavior of the bulk. However, as the open pores are filled and the binder assumes part of the supporting structure, there is an increased contraction rate since the binder is contracting at a more rapid rate than the filler. These observations support a two-phase model.

DENSITY

In the series studied, including densities from 1.3 to 1.7 g/cc, there is no evidence that density alone has an effect on the radiation induced contraction in graphite.

EXPERIMENTAL PROCEDURE

PREPARATION

The samples were machined to 0.426-in. diameter by 4-in. long cylinders, and the rounded ends were polished. All samples with processing temperatures above 1000 °C were annealed at 1000 °C to relieve any residual machining stresses.

Property measurements included length, coefficient of thermal expansion, and crystallite dimensions. Lengths were measured with a Bausch and Lomb DR-25 Optical Gage (Type 33-14-23). The length measurements had a standard deviation of 2×10^{-5} in. Coefficient of thermal expansion values were determined from 25 to 425 °C using a quartz drive and a Starrett gage (No. 656-611) allowing a standard deviation of approximately 5×10^{-2} in/in °C. The apparent crystallite dimensions and the crystallite lattice spacings in the $\underline{c}$ and $\underline{a}$ directions were determined by X-ray measurements. Those measurements in the $\underline{a}$ direction were made from the reflection (110) on solid samples; measurements in the $\underline{c}$ direction were made from the reflection (002) on powdered samples. The apparent crystallite size, L , was calculated from the breadth of the X-ray diffraction line by the Scherrer equations,

$$L = \frac{\kappa \lambda}{\beta \cos \theta}$$

where κ is the shape factor and β is the intrinsic line breadth.

The samples were irradiated in hollow graphite bars equipped with sliding covers at the Hanford reactor irradiation facilities. The irradiation temperature varied from 550 to 700 °C and the capsule atmosphere, the same for all samples, was a mixture of helium and carbon dioxide.

The incident neutron exposures ($E > 0.18$ MeV) are determined from the activation of nickel, iron, and cobalt monitors, and from the calculated neutron spectra for the irradiation facility. All wire counting was done with the gamma analytical system⁽¹⁾ at Hanford. The experimental details for determining the neutron exposures are discussed by Morgan⁽²⁾ in the description of the computer code (FOILS) used.

SAMPLE MANUFACTURE

Five heat treatment series were available for this study:

- 1) The first series consists of a molded Texas coke series⁽³⁾ processed at various temperatures from 1300 to 2700 °C. The degree of graphitization, as determined by crystallite size and layer spacing, increased with increasing graphitization temperature.
- 2) The second series was extruded from needle coke and graphitized at 200 °C steps, from 2400 to 3000 °C.
- 3) The third series⁽⁴⁾ was extruded from needle coke and pregraphitized at 2900 °C. The final process temperature was varied from 700 to 2900 °C.
- 4) The fourth series was glassy carbon⁽⁵⁾ heat treated at 1000, 2000, and 3000 °C.
- 5) The fifth series was molded from a mix of 50% carbon black and 50% binder coke.⁽⁶⁾ The formed pieces were processed at 1400, 3000, and 3500 °C.

The series of controlled-orientation samples⁽⁶⁾ was made by either molding, extruding, or a combination of both. The green mix, consisting of Continental coke flour and 30 medium pitch as binder, was extruded as 5-1/8 in. diameter rounds. The green stock was reformed by molding with the force applied parallel to the extrusion axis. The molding pressure was adjusted to yield plugs with a reversal of the orientation induced by the initial extrusion. A final set was molded as 6-1/8 in. diameter rounds directly from the green mix. The stock was then graphitized at 3000 °C.

The impregnation-level series of samples⁽⁶⁾ was made from base stock of a mix of Continental coke filler and 30 medium coal-tar pitch binder extruded with a 4 1/2 by 4 1/2 in. cross section. The binder-carbon content varied from 10.5 to 12%. The formed bars were impregnated with coal-tar pitch and with thermosetting resin to various levels from 1 to 21 wt%. After impregnation, the samples were graphitized at 3000 °C. The processing of this series increased the amount of less-graphitic carbon in the selected filler system so that irradiation effects originating in the non-graphitic portions could be more easily perceived. The most convenient method by which carbon may be added to a particular filler system is by impregnating previously formed graphite with either pitch or resins and then coking the impregnant. This allows one to hold other properties more constant than by varying the initial binder level.

The density series included graphites molded and extruded from needle coke. Densities ranged from 1.3 to 1.7 g/cc.

RESULTS AND DISCUSSION

GRAPHITIZATION TEMPERATURE

The initial property measurements for all graphitization series are given in Table I. As shown for the Texas coke series, there is no relationship between density or coefficient of thermal expansion and the graphitization temperature. However, the degree of graphitization, as indicated by the crystallite size and layer spacing, increases with increasing graphitization temperature. The irradiation results are given in Figure 1. Each data point shown represents an average of two samples. It is clearly shown that the dimensional stability is increased for the more graphitic material, i.e., higher graphitization temperature.

Although the increased degree of graphitization with temperature for the needle coke series is less apparent than for

TABLE I. Graphitization Series: Initial Property Measurements

Graphitization Temperature °C	Density g/cc	Coefficient of Thermal Expansion (25-400 °C) x 10 ⁶ /°C		Crystallite Dimensions, Å			
		⊥		L _c	c _o	L _a	a _o
Texas Coke—Coal-tar Pitch, Molded							
1370	----	----	----	---	----	---	-----
1800	1.51	3.95	3.12	100	6.87	120	-----
2100	1.50	3.51	3.01	175	6.82	162	-----
2300	1.50	3.61	2.70	215	6.79	320	-----
2570	1.50	4.29	2.85	300	6.76	350	-----
2700	1.53	3.56	2.75	290	6.76	440	-----
Needle Coke—Coal-tar Pitch, Molded							
2400	1.81	3.01	1.36	227	6.749	427	2.4619
2600	1.82	2.84	1.12	222	6.742	442	2.4622
2800	1.83	2.52	1.08	258	6.742	577	2.4619
3000	1.83	2.50	0.96	230	6.739	463	2.4614
Pregraphitized Needle Coke (2900 °C)—Coal-tar Pitch, Molded							
700	1.46	4.77	1.35	490	6.729	500	2.4557
900	1.51	4.11	1.68	520	6.732	780	2.4602
1400	1.48	3.41	1.15	485	6.729	1305	2.4595
2000	1.48	3.32	1.24	368	6.729	420	2.4585
2350	1.46	3.75	1.16	470	6.734	712	2.4580
2750	1.47	3.23	1.01	490	6.728	1485	2.4643
2900	1.50	3.17	1.04	600	6.726	705	2.4604
Carbon Black (50%)—Coal-tar Pitch, Molded							
1400	1.55	4.18	4.04	87	6.800	---	-----
3000	1.60	4.38	4.24	152	6.803	226	2.459
3500	1.65	----	4.16	163	6.790	211	2.461

the Texas coke series, the crystallite dimensions provide some indication of greater graphitization. The irradiation data given in Figure 2 are consistent with those found in Texas coke series. Although the samples have received only one low exposure, the increased stability with increased graphitization temperature is quite apparent.

FIGURE 1. Texas Coke Graphitization Series, Radiation Induced Length Changes (Transverse Orientation)

FIGURE 2. Needle Coke Graphitization Series, Radiation Induced Length Changes

For the pregraphitized needle coke series the only apparent change with increased graphitization temperature is a reduction in the coefficient of thermal expansion; this is consistent with the needle coke graphitization series. The irradiation results are given in Figures 3 and 4. Although increasing processing temperature increases stability over the whole range, the effect is less pronounced above 2000 °C.

The irradiation data for the carbon black graphitization series are presented in Figure 5. The lines on Figure 5 are a least square fit to the data; each line represents from 5 to 15 data points. As shown, the 1400 °C material contracts rapidly compared to the 3000 and 3500 °C materials. The 3000 °C material is slightly more stable than that graphitized at 3500 °C. This difference may be due to the difference between cooling rates in the resistance furnace used for the 3000 °C treatment and the induction furnace for the 3500 °C treatment.

The glassy carbon material has a density of about 1.5 g/cc and a coefficient of thermal expansion of 2.0 to 2.2×10^{-6} 1/°C. The irradiation data are presented in Figure 6. Again the increased processing temperature has improved the stability. Thus even for a turbostratic non-graphitizing material a higher processing temperature improves dimensional stability.

CRYSTALLITE ORIENTATION

The initial coefficient of thermal expansion and the contraction rates due to neutron exposures are listed in Table II for the controlled orientation series. The contraction rate is plotted against the coefficient of thermal expansion in Figure 7 for a first test of the data; as shown, the relationship is linear within one standard deviation.

An alternative analysis of the data consists of solving the Simmons' ⁽⁷⁾ equations for single-crystal dimensional changes, knowing the crystal coefficient of thermal expansion, the

FIGURE 3. *Pregraphitized Needle Coke Series, Radiation Induced Length Changes (Transverse Orientation)*

FIGURE 4. *Pregraphitized Needle Coke Series, Radiation Induced Length Changes (Parallel Orientation)*

FIGURE 5. Carbon Black Graphitization Series, Radiation Induced Length Changes

FIGURE 6. Glassy Carbon Graphitization Series, Radiation Induced Length Changes

TABLE II. Controlled Orientation Series: Contraction Rates
(Graphite is needle coke-coal-tar pitch, molded and/or extruded.)

Sample Set T/L Ratio	Orientation	Initial CTE $\times 10^6$ (25-100 °C)	Contraction Rate ¹ % Length Change/ 10^{21} nvt (E > 0.18 MeV)	Standard Deviation
6.18	Transverse	3.03	0.0832	± 0.0185
2.39	Transverse	2.37	0.0896	± 0.0168
0.78	Parallel	2.26	0.1020	± 0.0156
0.78	Transverse	1.76	0.1125	± 0.0154
2.39	Parallel	0.99	0.1156	± 0.0161
6.18	Parallel	0.49	0.1328	± 0.0164

¹Each contraction rate represents seven exposure points and two samples at each point. The contraction rates were computed over the exposure range 0.2 to 1.3×10^{21} nvt.

FIGURE 7. Controlled Orientation Series, Radiation Induced Contraction Rate Versus Coefficient of Thermal Expansion

aggregate coefficient of thermal expansion, and the rate of dimensional change with exposure. Thus, using the coefficients and contraction rates given in Table II, and the single-crystal coefficients given by Riley,⁽⁸⁾

$$\alpha_c = 26.75 \times 10^{-6}/^{\circ}\text{C} \quad (25 - 100 \text{ }^{\circ}\text{C})$$

$$\alpha_a = -1.15 \times 10^{-6}/^{\circ}\text{C} \quad (25 - 100 \text{ }^{\circ}\text{C})$$

and the Simmons' equations,

$$\alpha_x = A_x \alpha_c + (1 - A_x) \alpha_a$$

$$\frac{1}{\ell_x} \frac{d\ell_x}{d\gamma} = A_x \frac{1}{X_c} \frac{dX_c}{d\gamma} + (1 - A_x) \frac{1}{X_a} \frac{dX_a}{d\gamma}$$

the accommodation factor A_x may be found for each aggregate coefficient. Knowing the accommodation factor and the bulk growth rate, one may find the single-crystal growth rates. The growth rates were determined as $-0.167 \pm 0.007 \frac{\% \Delta L}{L}/10^{21}$ nvt for the a-axis and $+0.390 \pm 0.014 \frac{\% \Delta L}{L}/10^{21}$ nvt for the c-axis.

The calculated single-crystal dimensional changes, based on the present work, are much lower than those based on pyrolytic data.^(9,10) However, one might expect different apparent single-crystal changes over a range of processing temperatures or varying degrees of graphitization of the material. That is, as the material becomes more graphitic, one would expect a lower apparent crystal contraction rate as ordering in the bulk graphite improves dimensional stability. This effect of ordering can be seen in the graphitization series currently being studied.

Based on these data, the Simmons' equations provide a good relationship between the coefficient of thermal expansion and radiation induced contraction rates for materials identical except for the crystallite orientation.

IMPREGNATION

The impregnation-level series may be approached in either of two ways: One may consider the dimensional stability relative to either the impregnant level or the impregnant plus binder level. The binder level for all samples, as listed in Table III,

TABLE III. Impregnation Level Series
(Graphite is needle coke-coal-tar pitch, extruded)

Sample Set	Impregnant Levels						Apparent Density (g/cc)
	Binder and Impregnant (percent)						
	Coal-Tar Pitch			Resin	Total		
	Binder	Impregnant	Total	Impregnant	Impregnant	Binder and Impregnant	
60	10.5	1.0	11.5	----	1.0	11.5	1.55
30	11.2	6.8	18.0	----	6.8	18.0	1.54
15	12.0	6.5	18.5	----	6.5	18.5	1.59
C-1 to 10	10.5	3.5 (2) ¹	14.0	----	3.5	14.0	1.64
B-1 to 10	11.2	10.3 (2)	21.3	----	10.3	21.3	1.63
A-1 to 10	12.0	10.2 (2)	22.2	----	10.2	22.2	1.66
C-11 to 20	10.5	1.0	11.5	6.0	7.0	17.0	1.69
A-11 to 20	12.0	6.5	18.5	7.5	14.0	26.0	1.73
C-21 to 30	10.5	1.0	11.5	8.8 (2)	9.8	19.8	1.74
B-21 to 30	11.2	6.8	18.0	9.6 (2)	16.4	27.6	1.74
A-21 to 30	12.0	6.5	18.5	10.0 (2)	16.5	28.5	1.78
I-1	12.0	6.5	18.5	11.8	18.3	30.3	1.78
I-2	12.0	6.5	18.5	9.5	16.0	28.0	1.79
I-3	12.0	6.5	18.5	14.5 (2)	21.0	33.0	1.80
I-4	12.0	6.5	18.5	11.0 (2)	17.0	29.0	1.81

¹Designates the number of impregnation steps

is about 12%. Thus, for this sample series, including both binder and impregnant, the nature of the curve is not changed--only the origin is shifted. The data from Tables III and IV, for the contraction rate relative to the impregnant level, are presented in Figures 8 and 9 for the transverse and parallel samples respectively. A definite effect of impregnant is shown at the higher impregnant levels, above 16% approximately.

TABLE IV. Impregnation Level Series: Contraction Rates

Sample Set	Orientation	Initial CTE x 10 ⁶ (25-100 °C)	Contraction Rate ¹ % Length Change/ 10 ²¹ nvt (E > 0.18 MeV)	Standard Deviation
60	Transverse	2.05	0.091	±0.014
	Parallel	0.54	0.124	±0.013
30	Transverse	2.25	0.086	±0.013
	Parallel	0.65	0.128	±0.015
15	Transverse	2.54	0.091	±0.014
	Parallel	0.44	0.124	±0.013
C-1 to 10	Parallel	----	0.116	±0.009
B-1 to 10	Parallel	----	0.116	±0.005
A-1 to 10	Parallel	----	0.101	±0.013
C-11 to 20	Parallel	----	0.114	±0.009
A-11 to 20	Parallel	----	0.117	±0.006
C-21 to 30	Parallel	----	0.130	±0.007
B-21 to 30	Parallel	----	0.142	±0.004
A-21 to 30	Parallel	----	0.142	±0.005
I-1	Transverse	2.13	0.164	±0.011
	Parallel	0.86	0.156	±0.009
I-2	Transverse	2.13	0.136	±0.017
	Parallel	0.78	0.133	±0.017
I-3	Transverse	2.23	0.183	±0.019
	Parallel	0.82	0.194	±0.014
I-4	Transverse	2.36	0.133	±0.012
	Parallel	0.71	0.132	±0.013

¹Each contraction rate represents data for seven exposure points with an average of three samples each. The contraction rates were computed over the exposure range 0.2 to 1.3 x 10²¹ nvt.

FIGURE 8. *Impregnant Level Series, Radiation Induced Contraction Rate Versus Percent Impregnant (Transverse Orientation)*

FIGURE 9. *Impregnant Level Series, Radiation Induced Contraction Rate Versus Percent Impregnant (Parallel Orientation)*

One explanation of this behavior is the preferential filling of open pores until the binder becomes part of the supporting structure. Up to an impregnant level of 16% there is no effect because the impregnant is filling the open pores and shrinking away during graphitization. However, as the impregnant level approaches the open pore volume, the impregnant becomes part of the supporting structure after going through the graphitization step in processing. As a result the dimensional behavior of the bulk under neutron irradiation is determined not only by the behavior of the filler material but also by the behavior of the binder and impregnant material, as indicated by de Halas and Yoshikawa.⁽¹¹⁾ As the impregnant level is raised, it contributes more to the bulk behavior.

DENSITY

The initial property measurements for the density series are given in Table V.

TABLE V. Density Series: Initial Property Measurements

Material	Density (g/cc)	Coefficient of Thermal Expansion (25-425 °C) $\times 10^6/^{\circ}\text{C}$		Crystallite Dimensions, Å			
		I	II	$\frac{L}{c}$	$\frac{c}{a}$	$\frac{L}{a}$	$\frac{a}{a_0}$
Needle Coke Coal-Tar Pitch	1.41	4.73	1.21	527	6.727	684	2.4621
Needle Coke Petroleum Pitch	1.30	5.05	1.23	496	6.736	673	2.4627
TSX	1.71	3.52	0.89	645	6.727	685	2.4644

The irradiation data for the density series is presented in Figure 10. As a reference, TSX graphite is also plotted. The data show that low density does not necessarily imply a high contraction rate; both low density sample sets show improved stability compared to the standard TSX.

FIGURE 10. Low Density Series, Radiation Induced Length Changes

LIMITATION

It should be noted that these results are reported for relatively low exposure compared to reactor lifetime exposures. As a result, although several conclusions can be made relating the radiation-induced contraction rate of graphite to processing variables, full reactor lifetime exposures are required to determine behavior of a graphite moderator assembly.

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