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PHYSICAL PROPERTY MEASUREMENTS ON NFR GRAPHITE
II. UNIRRADIATED CORE GRAPHITE

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PHYSICAL PROPERTY MEASUREMENTS ON NPR GRAPHITE

II. UNIRRADIATED CORE GRAPHITE

August 29, 1961

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Nonmetallic Materials Development Operation
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RICHLAND, WASHINGTON

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PHYSICAL PROPERTY MEASUREMENTS ON NFR GRAPHITE

II. UNIRRADIATED CORE GRAPHITE

INTRODUCTION

Graphite for the NFR is a new type having a different coke, particle size, and higher graphitization temperature than graphites previously used for Hanford reactors. A previous report⁽¹⁾ covers the physical properties of the NFR reflector graphite. It is the purpose of this report to supply up-to-date, room temperature, pre-irradiation physical properties of NFR core graphite, and establish some statistical limits for material variations. Information secured for this report will be used to help evaluate the post-irradiation property measurements.

SUMMARY

Four bars of NFR core graphite produced by the National Carbon Company were evaluated. Thermal conductivity, electrical resistivity, coefficient of thermal expansion, tensile strength, compressive strength, and modulus of rupture data are summarized in Table I. A further statistical analysis of the data was carried out, and the results shown in Table II. It is demonstrated that anisotropy in the bars and the method of sampling effect the measured properties. TBX is shown to be more anisotropic than either CSF or TSGW graphites used in the construction of some of the present Hanford reactors.

DESCRIPTION OF GRAPHITE

Graphite used in this investigation was manufactured by the National Carbon Company and is designated TBX, see Appendix I. All four bars were specified for graphitization at 3000° C. Data in this report should represent the properties of

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at least 75 percent of the material specified for the core of the NPR. The bars as received were approximately 6" x 6" x 48" pieces. All four bars were from graphitization heat 118, each individual bar code can be found in Appendix II. Identification numbers assigned to the bars are: 160, 161, 163, 164.

SAMPLING PROCEDURES

Samples were taken in a manner that would avoid the center of the bar which in a reactor piece will generally be bored out.

Figure 1 shows the initial cuts for each individual bar. Modulus of rupture samples had the dimensions shown in Figure 2. Tensile specimens were rough cut as shown in Figure 3, while the final shape is shown in Figure 4. Samples for measuring thermal conductivity, electrical resistivity, and the coefficient of thermal expansion were rough cut and numbered as shown in Figure 5, then finished to a standard size of 0.426" diameter x 4" in length. Compressive strength samples had the final dimensions as shown in Figure 6.

MEASUREMENTS

Thermal Conductivity and Electrical Resistivity*

Measurements were made at room temperature on the Mark II apparatus and Kohlraush apparatus using standard techniques. (2)

Modulus of Rupture

Bars were broken with the load applied at the center of a 16-inch span with the 5-inch dimension vertical. Platten speed was approximately 0.02"/min.

Tensile Strength

Samples were pulled on an Instron testing machine with the specimen held in collet type grips. Crosshead speed was 0.01"/min.

* Since thermal conductivity, electrical resistivity, and the coefficient of thermal expansion are non-destructive test the same group of samples were used for these measurements.

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Compressive Strength

Samples were positioned with the 5" dimension vertical and the load applied to a 6" x 6" face. Platten speed was approximately 0.02"/min.

Coefficient of Thermal Expansion*

Measurements were made in air on a quartz differential dilatometer over the temperature range of 25° C to 425° C.

DISCUSSION

The average value, $\bar{x}$, of both transverse and parallel cuts for each particular property along with the standard deviation of the bars is summarized in Table I.

TABLE I

<u>Property</u>	<u>Transverse $\bar{x}$</u>	<u>Transverse σ (estimated standard deviation of bars)</u>	<u>Parallel $\bar{x}$</u>	<u>Parallel σ (estimated)</u>
Thermal conductivity (cal cm ⁻¹ sec ⁻¹ °C ⁻¹)	0.273	2%	0.530	2%
Electrical resistivity (ohm/cm)	10.99 x 10 ⁻⁴	2%	6.22 x 10 ⁻⁴	4%
Coefficient of thermal expansion (10 ⁻⁶ in/in/°C)	3.39	1%	0.89	2%
Tensile strength (psi)	957	28%	1668	7%
Compressive strength (psi)	4186	5%		
Modulus of rupture (psi)	2740	30%		

Further statistical tests were applied in an effort to provide numbers that would be of more practical use to a design engineer. Two different statistical techniques

* Since thermal conductivity, electrical resistivity, and the coefficient of thermal expansion are non-destructive test the same group of samples were used for these measurements.

were used depending on the type of physical property being considered. Thermal conductivity, electrical resistivity, and thermal expansion values are averages of the different regions within each sample. Properties of this type can have a statistical technique applied to produce a 95% tolerance limit. One can then say that if every bar of TSX graphite from the 3000° C zone of the graphitization furnace were tested, then 95% of the bars tested would have values that would fall within these tolerance limits.

Strength measurements such as tensile strength, compressive strength, and modulus of rupture present a different problem. The strength of any one sample is dependent on the weakest region in the sample. Using a statistical technique that takes into consideration this "weakest link", a 95% survival estimate can be made. This survival estimate predicts that 19 out of 20 bars tested will be stronger than the stated value. Statistical limits of this nature should be more useful for design purposes than the average value ($\bar{X}$). A summary of these data is given in Table II. The limited number of modulus of rupture samples tested does not allow this type of statistical treatment.

TABLE II

<u>Property</u>	<u>Transverse*</u>	<u>Parallel*</u>	<u>Remarks</u>
Thermal conductivity	0.262-0.284	0.519-0.541	Tolerance limits
Electrical resistivity $\times 10^4$	9.64-12.32	4.89-7.56	Tolerance limits
Coefficient of thermal expansion $\times 10^6$	3.31-3.46	0.874-0.912	Tolerance limits
Tensile strength	583	1294	95% survival est.
Compressive strength	3606		95% survival est.

* Values in this table were derived from the normal samples; does not include data shown in Table III.

In an effort to better explain the nature of graphite and as an aid to those attempting to understand the wide variations in physical properties across an individual bar or from bar to bar, a group of parallel samples were taken as shown in Figure 7 and a group of transverse samples taken as shown in Figure 8. Table III lists the values for the coefficient of thermal expansion and thermal conductivity of these samples.

TABLE III

Coefficient of Thermal Expansion ($10^{-6}/^{\circ}\text{C}$)				Thermal Conductivity ($\text{cal cm}^{-1} \text{sec}^{-1} ^{\circ}\text{C}^{-1}$)			
Sample #	Parallel	Sample #	Transverse	Sample #	Parallel	Sample #	Transverse
213	0.84	91	3.36	213	0.487	91	0.319
214	0.84	92	3.54	214	0.493	92	0.256
215	0.84	93	3.60	215	0.501	93	0.229
216	0.84	94	3.76	216	0.480	94	0.235
217	0.82	95	3.91	217	0.524	95	0.240
218	0.82	96	3.76	218	0.502	96	0.279
219	0.81	97	3.61	219	0.544	97	0.250
220	0.82	98	3.43	220	0.511	98	0.271
221	0.78			221	0.495		
222	0.78			222	0.535		
223	0.82			223	0.530		
224	0.83			224	0.490		
225	0.80			225	0.478		
226	0.82			226	0.526		
227	0.83			227	0.450		

It is readily apparent that the values obtained for these two properties are dependent on the position within the bar from which the samples were taken. Physical properties are dependent on the size of the extrusion, and the amount of material removed in taking the finish cuts on the rough bar. Extrusion size affects the anisotropy, that is the orientation of the particles, thus large bars have less orientation in the center than small bars. Since maximum orientation is at the bar edge uneven removal of stock in machining a finished shape can produce a bar that is more orientated on one side than the other.

Anisotropy is customarily measured by the ratio of transverse to parallel coefficients of thermal expansion (CTE); thus for different combinations of these data, the CTE ratio could vary from 5.0 to 3.9. This emphasizes the importance of exercising care in using property values determined for any one sample as representative of the entire bar, or worse as representative of a large group of similar bars. Further consideration of these conditions forces one to conclude that the position in a bar that was sampled and measured must be specified if comparisons are to be made between similar bars to between different types of graphite.

Relative comparisons can be made of the coefficients of thermal expansion and thermal conductivities but only if the values for TEK are taken from the same relative position in the bar as the CSF and TBGF values.⁽³⁾ These data are summarized in Table IV.

TABLE IV

	CSF		TSOF		TSX	
	Transverse	Parallel	Transverse	Parallel	Transverse	Parallel
Coefficient of thermal expansion $\times 10^6$ (25-425° C)	3.5	1.5	4.4	2.7	3.8	0.81
CTE ratio transverse/parallel	2.3		1.6		4.7	
Thermal conductivity (cal $\text{cm}^{-1} \text{sec}^{-1} \text{°C}^{-1}$)	0.26	0.41	0.17	0.20	0.24	0.50
Thermal conductivity ratio transverse/parallel	0.62		0.85		0.48	

The values given for TSX in the above table are not to be used as a measure of the graphite in the core since they were taken from a position that would be bored out of a reactor block. Only the values in Tables I and II are indicative of the graphite in the finished FPR core.

Insufficient data are available on the strength of CSF or TSOF to make comparisons of these properties with TSX.

FUTURE TESTING

Samples of this material are presently being irradiated. After discharge, the following measurements will be made: thermal conductivity, electrical resistivity, coefficient of thermal expansion, X-ray (C_0 , L_c), length, and weight.

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APPENDIX 1

NPR CORE GRAPHITE SPECS.

Type TSX-18

Vendor:	National Carbon Company
Coke:	Continental-Lake Charles - No. 1
Particle Size:	45-55 weight percent through 200 mesh 100 weight percent through 20 mesh
Pitch:	#30 medium coal tar binder - Allied Chemical Company
Raw Bar Dimension:	Approximately 6-1/2" x 6-1/2"
Graphitization Temperature:	Minimum 2800° C 75% - 3000° C
Impregnant:	#15 vacuum impregnation pitch - Allied Chemical Company
Number of Impregnations:	One
Purity:	Minimum + 0.25 dih
Purification:	High temperature gaseous - after graphitization - temperature in excess 2000° C
Density:	1.71 ± 0.03 g/cc

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APPENDIX II

TSX GRAPHITE BAR CODES

In addition to the manufacturer's code, graphite type and heat number, the following table gives supplemental data for the bars used in these tests.

<u>Our</u> <u>Code</u>	<u>Graphitiza-</u> <u>tion heat</u>	<u>Graphitization Position</u> <u>Row-Column</u>	<u>Forming lot</u> <u>number</u>	<u>Purification</u> <u>run number</u>	<u>Purification posi-</u> <u>tion Row & Column</u>
161	118	10-3	30	902	31-3
163	118	16-4	51	902	31-2
160	118	10-3	53	927	30-4
164	118	30-6	52	927	30-2

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FIGURE 1

Graphite Bar Layout

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FIGURE 2

Modulus of Rupture Sample

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FIGURE 3

Tensile Sample Layout

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FIGURE 4
Tensile Sample

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FIGURE 5

Core Piece Layout - S'd. Sample

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FIGURE 6

Compression Test Sample

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FIGURE 7

Core Piece Layout - Parallel Sample Study

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FIGURE 8

Core Piece Layout Transverse Sample Study

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