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ISOSTATICALLY MOLDED P-03 GRAPHITE FOR LARGE STRUCTURES

V. Kachur and H. C. Nicholson
Materials Department
June 29, 1965

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ISOSTATICALLY MOLDED P-03 GRAPHITE FOR LARGE STRUCTURES

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TABLE OF CONTENTS

	<u>Page</u> v
ABSTRACT	
 PART I: EVALUATION OF ISOSTATICALLY MOLDED P-03 GRAPHITE	
INTRODUCTION	1
ISOSTATIC MOLDING OF P-03 GRAPHITE	2
PHYSICAL PROPERTIES OF ISOSTATIC P-03	3
MECHANICAL PROPERTIES OF ISOSTATIC P-03 GRAPHITE	10
DISCUSSION: PHYSICAL PROPERTIES OF P-03 GRAPHITE	18
DISCUSSION: STRENGTH PROPERTIES OF P-03 GRAPHITE	21
CONCLUSIONS	26
 PART II: DEVELOPMENT OF CEMENTED P-03 STRUCTURES	
INTRODUCTION	27
PROCESS DEVELOPMENT	27
EVALUATION OF THE P-03 CEMENTED JOINTS	28
DISCUSSION	32
CONCLUSIONS	33
RECOMMENDATIONS	34
 ACKNOWLEDGEMENTS	35
 REFERENCES	66

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LIST OF TABLES

<u>TABLE</u>		<u>PAGE</u>
1	THERMAL EXPANSION OF THE ISOSTATICALLY MOLDED P-03 GRAPHITE	36
2	STRESS VS. STRAIN RELATIONSHIP FOR ISOSTATICALLY MOLDED P-03 GRAPHITE IN TENSION AT ROOM TEMPERATURE	37
3	TENSILE TEST RESULTS ON THE ISOSTATICALLY MOLDED P-03 GRAPHITE	38
4	SUMMARY OF COMPRESSION TESTS ON THE ISOSTATICALLY MOLDED P-03 GRAPHITE AT ROOM TEMPERATURE	39
5	STRESS VS. STRAIN IN ROOM-TEMPERATURE COMPRESSION OF ISOSTATICALLY MOLDED P-03 GRAPHITE	40
6	SUMMARY OF FLEXURE TEST RESULTS ON THE ISOSTATICALLY MOLDED P-03 GRAPHITE	41

LIST OF FIGURES

1.	Appearance of the isostatically molded P-03 logs.	42
2.	Microstructure of the isostatically molded P-03 graphite.	43
3.	Thermal expansion of P-03	44
4.	Summary of electrical resistivity data for isostatic P-03 graphite.	45
5.	Thermal conductivity of the isostatically molded P-03, together with comparison data for the conventionally fabricated graphites.	46
6.	Graphite tensile specimens.	47
7.	Stress vs strain curve for isostatically molded P-03 graphite in tension, averaged for six specimens each from the axial and radial orientations.	48
8.	Tangent Young's modulus as a function of applied stress in tension.	49
9.	Stress vs. strain relationship in tension as a function of P-03 graphite's strength.	50
10.	Tensile strength of isostatically molded P-03 graphite as a function of temperature as determined on axial specimens.	51
11.	Permanent elongation of the thread-loaded L-2 specimens when pulled to fracture at a uniform strain rate.	52
12.	Elongation (non-elastic component only) of thread-loaded tensile specimen at high temperatures.	53
13.	Compressive strength of isostatically molded P-03 graphite at room temperature.	54
14.	Stress vs. strain for the P-03 graphite in compression, as determined at room temperature.	55
15.	Stress vs. strain curve for P-03 in compression, showing the influence of pre-loading.	56
16.	Variation of the tangent Young's modulus with applied stress in the room-temperature compression tests on P-03 graphite.	57
17.	Poisson's ratio of isostatically molded P-03 graphite at room temperature.	58
18.	Cemented tensile specimen.	59
19.	Comparative tensile strength of cemented P-03 graphite with in-process vs P-514 joint.	59
20.	Cemented ring flexure test specimen.	60
21.	Cemented flexure test specimen with cracks in the maximum fiber stress orientation.	60

LIST OF FIGURES

(cont.)

22.	Photomicrograph of a graphitized P-514 joint (50X).	61
23.	Photomicrograph of a graphitized "in-process" joint (50X).	61
24.	Hydrogen permeability of unimpregnated, silica-impregnated, and cemented P-03 with in-process or P-514 joints.	62
25.	Appearance of P-03 staves before cementing.	63
26.	Cemented P-03 staved cylinder in the as-cured stage.	64
27.	Appearance of cemented P-03 cylinder after machining.	65

ABSTRACT

The fine-grained premium grade P-03 graphite was successfully fabricated by an isostatic molding technique, resulting in flaw-free stock 7 inches in diameter and 72 inches long. Selected mechanical and thermophysical properties of this developmental material were evaluated for NERVA design purposes, and are presented in Part I of this report. The feasibility of producing a graphitized cemented joint equivalent in strength to that of the isostatically molded P-03 matrix is demonstrated in Part II.

PART I

EVALUATION OF ISOSTATICALLY MOLDED P-03 GRAPHITE

INTRODUCTION

To meet the structural graphite requirements of the NERVA reactor with a reasonable margin of safety, a number of commercial graphite materials has been surveyed to determine the optimum product for each hardware application.⁽¹⁻⁵⁾ One of the reactor components, the inner reflector, was typically fabricated from the coarse-grained graphite extruded as a 43-inch diameter by 72-inch long cylinder. This graphite is used primarily for large electrodes and molds; its application for the inner reflector was made necessary by the lack of better materials in the graphite industry. The inner reflector structural performance was hence limited by the low strength of this graphite, providing a potential constraint to the NERVA reactor operation. Elimination of this constraint required the availability of high-strength structural graphite in large molded or extruded cylinders.

For the purpose of this report, "high-strength graphite" is defined as graphitized material with compressive strength in excess of 10,000 psi, as compared to about 5,000 psi typical of the coarse-grained electrode stock. The definition of "large size" is not exact, since in the general case either a large diameter or a long length could be required, with the more precise dimensions being established by the hardware design. The current state of the art in the graphite industry is, unfortunately, such that the large high-strength products are not available from any source; furthermore, it is observed that increasing the size of any commercial product leads to an undesirable reduction in its strength.

To assess the availability of high-strength graphite for large structural applications in the NERVA reactors, a brief but wide-ranging survey was made of the graphite products readily available from various manufacturers. This survey covered both the extruded^(1, 3) and molded^(3,4,5) products in a large variety of sizes. Results of their evaluation were summarized in several previous reports. Perhaps the most important conclusion resulting from this survey was that conventional molded and extruded graphites offered only a minor capability for their improvement, and therefore an intensive study should be undertaken of the non-conventionally fabricated materials. On the basis of all available information, it was tentatively decided that the most likely means for

obtaining high-strength graphite in large sizes was by isostatically molding the P-03 grade material.^(5, 6) The reasons for selecting this particular grade were the following:

1. Well-established fabrication technology for the regularly molded P-03.
2. Excellent strength of this grade in tension, flexure, and compression.
3. Flaw-free macrostructure and good radiographic uniformity of the regularly molded P-03.
4. Manufacturer's demonstrated capability to produce small blocks of P-03 by the isostatic molding technique.
5. Manufacturer's cooperation in scaling up the isostatic P-03 graphite to large dimensions.

The subsequent portions of this report will describe the fabrication of isostatically molded P-03 graphite to the scaled-up dimensions, and the evaluation of resulting material for NERVA hardware.

ISOSTATIC MOLDING OF P-03 GRAPHITE

For the purposes of this report, isostatic molding refers to the procedure in which the combined raw materials (the so-called "green mix") are shaped to an intended stock configuration. The green mix, produced by blending the carefully sized coke particles with an appropriate binder, is packed into rubber molds. These molds are then sealed and submerged under a fluid contained within a pressure vessel. Hydrostatic pressure is applied on the fluid to provide a uniform molding force on the coke and binder constituents of the green mix. The resulting compacted mixture is then cured to set the binder and provide a strong, rough-molded configuration for the subsequent carbonizing and graphitizing thermal treatments.

Initial fabrication of the isostatically molded P-03 had been developed by the manufacturer as a small-scale, company-funded project intended for material improvement. The details concerning both the raw ingredients and fabrication are therefore considered to be within the manufacturer's proprietary domain, and hence will not be discussed in this report. The final

material resulting from isostatic fabrication is a strong, dense, flaw-free graphite suitable for critical structural applications in the NERVA reactor. The scale-up in size of this graphite for NERVA purposes was deemed to be feasible, but was substantially hampered by two factors:

1. Green-mix Limitations

It was considered difficult to cure and bake the scaled-up stock without structural defects (mainly cracks), especially if no loss of strength or density compared with the small-scale material was required.

2. Production Limitations

A large pressure chamber capable of sustaining about 25,000 psi was required for the green-mix fabrication.

In view of these limitations, the scale-up of isostatically molded P-03 was undertaken only on a limited basis. The largest P-03 configuration fabricated previously on the manufacturer's in-house equipment was a 7"D x 21"L cylinder.⁽⁵⁾ The primary objective of the scale-up effort was to produce a 7"D x 72"L cylinder with no sacrifice in either the density or strength compared with the small-scale stock. The scale-up effort thus did not involve a change in the stock diameter, but the length was to be increased by a factor of 3.5. Eight logs of the 7"D x 72"L size were fabricated by the manufacturer; four were intended for evaluation in this program, while the remainder was mainly for back-up purposes. The balance of this section describes the evaluation of the four primary logs. The properties of isostatically molded P-03 graphite are described in detail, both to characterize this relatively new material and to permit its consideration for advanced NERVA structures.

PHYSICAL PROPERTIES OF ISOSTATIC P-03

Two of the four 7"D x 72"L isostatically molded logs were received in damaged condition. One was broken into three pieces, and the other into two pieces. This was attributed by the manufacturer to the processing difficulties encountered in baking and graphitizing, and not to

any undesirable aspects of the material itself. The remaining two logs were found to be free of defects. All four logs were acceptable for evaluation; they are further described in the following table:

LOG NUMBER	CONDITION	DIMENSIONS		WEIGHT (pounds)	DENSITY (g/cc)
		Length (inches)	Diameter (inches)		
1	No damage	75	7.6	227.5	1.840
2	Broken, 2 pieces	39	7.6	120.5	1.816
		<u>32</u>	7.8	<u>96.3</u>	
		Total 71		Total 216.8	
3	Broken 3 pieces	24.8	7.6	72.5	1.819
		22	7.6	67.0	
		<u>25.8</u>	<u>7.7</u>	<u>80.5</u>	
		Total 72.6		Total 219.0	
4	No damage	73.3	7.8	225	1.800

Figure 1 shows the appearance of logs No. 1 and No. 2.

Density

Graphite density was determined routinely from the weight-to-volume ratio for regularly shaped specimens such as flexure bars and compression cylinders. Density values provide a useful parameter for interpreting both the strength data and structural quality of the original stock. The individual density values will be routinely listed with their corresponding specimens to facilitate interpretation of test data.

Microstructure

Representative microstructure of isostatic P-03 graphite (log No. 3) is given in Figure 2. The section is normal to the log axis, and located near the center of the log.

Thermal Expansion

Thermal expansion measurements were made on radiantly heated specimens in a graphite resistance furnace capable of reaching 2500°C (4532°F). Each specimen was mounted in a double-

beam expansion measuring device, and its elongation continuously recorded. The specimen temperature was continuously monitored with a two-color pyrometer, and periodically cross-checked with a disappearing-filament pyrometer. A flowing helium atmosphere was maintained within the furnace. The raw elongation versus temperature data were processed through a computer, and the average coefficient of linear thermal expansion calculated in 100°C intervals. Thermal expansion coefficients were determined on one radial and axial specimen each machined from log No. 1. The resulting information is summarized in Table 1. Figure 3 compares thermal expansion data for log No. 1 with the previous results⁽⁵⁾ determined on small-size isostatic P-03 graphite. In all cases, there is only a minor difference between axial and radial expansions, showing that isostatically molded P-03 is a fully isotropic material. At high temperatures, the coefficient of thermal expansion is noted to decrease for the denser specimens (see the insert in Figure 3).

A close check on the length of thermal expansion specimens machined from log No. 1 revealed the following:

Axial specimen - no detectable change

Radial specimen - expansion, about 0.0005 inch/inch

Expansion in the radial specimen occurred upon heating between 1900 and 2500°C during the test run. Review of the specimen length data for sub-size isostatic P-03 graphite (density 1.875 g/cc) showed identical changes. The slight radial expansion in both cases could be due to thermally induced relief of a locked-in stress within the specimens.

Electrical Resistivity

Measurements of electrical resistivity were made on flexure bars with a four-point bridge apparatus. An outer pair of electrodes, spaced 3.00 inches apart, served to hold the specimen. The inner pair of electrodes, usually spaced 2.50 inches apart, were pressed into contact with the specimen by a pneumatically operated mechanism. The excitation voltage consisted of a square wave having a frequency of 40 cycles per second; this voltage was low enough to keep the current to a few milliamperes and thus avoid introducing any perceptible errors due to self-heating within the specimen.

Electrical resistivity was defined by the specimen geometry and electrical resistance, with

$$R = r \times \frac{L}{A},$$

where R = electrical resistance of a flexure bar

r = electrical resistivity

A = specimen cross-sectional area

L = inter-electrode span, usually 2.50" (6.35 cm).

In conventionally fabricated graphites, the with-grain resistivity is lower than that across the grain. This results from a preferential alignment of plate-like coke particles when the "green mix" is shaped by either molding or extrusion. For the isostatically molded P-03 graphite, the resistivity should be identical in all directions for the following reasons:

1. P-03 coke particles appear to be small and nearly rounded (Figure 2).
2. The molding force is equal in all directions, providing a uniform material flow in the "green mix".

Electrical resistivity measurements would thus offer a simple method for determining the degree of isotropy in the isostatically molded logs. The following results were obtained:

<u>SPECIMEN ORIENTATION</u>	<u>NUMBER OF TESTS</u>	<u>SPECIMEN DENSITY</u>			<u>ELECTRICAL RESISTIVITY</u>		
		<u>Low</u>	<u>Average</u> (grams per cm ³)	<u>High</u>	<u>Low</u>	<u>Average</u> (milliohm - cm)	<u>High</u>
A. Measurements on flexure bars (test section 0.44 x 0.44 x 3.0 inches) from log No. 1							
Axial	2	1.818	1.819	1.820	1.75	1.76	1.77
Radial	2	-----	1.829	-----	-----	1.75	----
B. Measurements on small cylinders (test section 0.50"D x 1.50"L) from log No. 1							
Axial	3	1.807	1.815	1.821	1.94	1.96	1.98
Axial	3	1.823	1.833	1.841	1.70	1.75	1.81
Radial	2	1.813	1.815	1.817	1.91	1.94	1.96
Radial	4	1.824	1.830	1.836	1.66	1.74	1.80
C. Measurements on flexure bars (test section 0.44 x 0.44 x 2.50 inches) from log No. 3							
Axial	6	1.839	1.840	1.843	1.51	1.52	1.55
Axial	4	1.844	1.845	1.846	1.52	1.53	1.55
Axial	2	1.849	1.850	1.851	-----	1.52	----

<u>SPECIMEN ORIENTATION</u>	<u>NUMBER OF TESTS</u>	<u>SPECIMEN DENSITY</u>			<u>ELECTRICAL RESISTIVITY</u>		
		<u>Low</u>	<u>Average</u>	<u>High</u>	<u>Low</u>	<u>Average</u>	<u>High</u>
-continued-		(grams per cm ³)			(milliohm - cm)		

D. Measurements on flexure bars selected from item C, before and after baking at 2725°C (4940°F) for 25 minutes in flowing helium atmosphere

Pre-bake	5	1.841	1.845	1.849	1.51	1.52	1.53
Post-bake	5	1.841	1.845	1.849	1.55	1.58	1.62

All resistivity values determined on the isostatic stock are summarized in Figure 4. Electrical resistivity is seen to decrease with specimen density, since more conductive paths become available in the denser material. Also shown in Figure 4 is the analytical curve derived previously (see WANL-TME-1031) from the with-grain electrical resistivity measurements on regularly molded P-03. It is evident that this curve provides a good fit to electrical resistivity values of specimens from logs No. 1 and No. 3. The observed scatter might be caused by the size effect and moisture content within the specimens. The difference between axial and radial resistivities is found to be small compared to the scatter range obtained for a single orientation, thereby confirming the isotropic nature of the isostatically molded P-03 graphite.

To define the influence of absorbed water vapor on the electrical resistivity of P-03, five axial flexure bars machined from log No. 3 were baked in a flowing helium atmosphere at 2725°C for 25 minutes. Their dimensions, measured to 0.001 inch with a micrometer, were found to be unchanged. However, a systematic increase in electrical resistivity values had occurred:

SPECIMEN DENSITY (g/cc)	ELECTRICAL RESISTIVITY		
	Initial	Post-bake	Change
		(milliohm - cm)	
1.841	1.52	1.62	+0.10
1.843	1.51	1.59	+0.08
1.845	1.53	1.57	+0.04
1.846	1.52	1.55	+0.03
1.849	1.51	1.55	+0.04
Average: 1.845	1.52	1.58	+0.06 (+4%)

This increase was most likely due to the loss of absorbed water rather than any changes in the resistivity of graphite matrix.

Thermal Conductivity

Measurements of thermal conductivity were made on three cylindrical specimens (0.50" diameter x 0.90" length) from log No. 3. The heat flow was parallel to both the log and specimen axes, with a temperature gradient of less than 10°C present across the flats during steady-state conditions. Armco iron was used as the comparative standard. The following results were obtained:

<u>AVERAGE TEMPERATURE</u>		<u>SPECIMEN DENSITY</u>	<u>THERMAL CONDUCTIVITY</u>		
(°C)	(°F)	(g/cc)	$\left(\frac{\text{cal-cm}}{\text{cm}^2 \text{ sec}^\circ\text{C}} \right)$	$\left(\frac{\text{watt-cm}}{\text{cm}^2 \text{ }^\circ\text{C}} \right)$	$\left(\frac{\text{BTU-ft}}{\text{ft}^2 \text{ hr }^\circ\text{F}} \right)$
80	176.0	1.832	0.150	0.627	36.2
83	181.4	1.833	0.181	0.758	43.8
82	179.6	1.836	0.147	0.613	35.4
Average values:		1.834	0.159 (+13.9, -7.6%)	0.666	38.5

These specimens are expected to have an electrical resistivity (see Figure 4) of 1.60 ± 0.05 milliohm-cm. The product of thermal conductivity times electrical resistivity for conventional graphites is a constant equal to 31×10^{-5} (ohm-calories)/(sec°C) at room temperature.⁽⁷⁾ From the above data on isostatically molded P-03 log No. 3, this constant becomes the following:

$$\begin{array}{l}
 \text{Average value} \\
 (\text{Electrical resistivity}) \times (\text{thermal conductivity}) = \text{constant} \\
 1.60 \times 10^{-3} \text{ ohm-cm} \times 0.159 \frac{\text{cal-cm}}{\text{cm}^2 \text{ sec}^\circ\text{C}} = 25.4 \times 10^{-5} \frac{\text{ohm-cal}}{\text{sec}^\circ\text{C}} \\
 \\
 \text{High value} \\
 1.55 \times 10^{-3} \quad \times 0.181 \quad = 28.1 \times 10^{-5} \\
 \\
 \text{Low value} \\
 1.65 \times 10^{-3} \quad \times 0.147 \quad = 24.3 \times 10^{-5}
 \end{array}$$

The average constant of 25.4×10^{-5} for isostatic P-03 (log No. 3 specimens) is thus 22% smaller than the value calculated for regular graphite.

An accurate measurement of thermal conductivity is generally conceded to be difficult. The three values determined above show a large scatter (+13.9, -7.6%) from their average. This

scatter masks an expected increase of thermal conductivity with specimen density, and indicates that the heat losses and interface effects during the test are appreciable. It is therefore a matter of considerable practical interest to determine an accurate value of the resistivity x conductivity constant for the isostatically molded graphite, since this would permit an easy determination of thermal conductivity from the relatively fast and simple measurements of this graphite's electrical resistivity.

Additional data on thermal conductivity of isostatic P-03 graphite was obtained from the measurements of thermal diffusivity by a thermal pulse technique. These measurements were made on a 1.83 g/cc sample of sub-size developmental stock by the Graphite Research and Development Section of Battelle's Pacific Northwest Laboratories.⁽⁸⁾ The resulting data provides thermal conductivity of isostatic P-03 over a wide range of temperatures:

TEMPERATURE		THERMAL CONDUCTIVITY		
(°C)	(°F)	$\left(\frac{\text{cal-cm}}{\text{cm}^2 \text{ } ^\circ\text{C}} \right)$	$\left(\frac{\text{watt-cm}}{\text{cm}^2 \text{ } ^\circ\text{C}} \right)$	$\left(\frac{\text{BTU-ft}}{\text{hr ft}^2 \text{ } ^\circ\text{F}} \right)$
25	77	0.148	0.620	35.8
81	178	0.158	0.661	38.2
		0.160	0.670	38.7
137	279	0.169	0.708	40.9
		0.177	0.741	42.8
208	406	0.177	0.711	41.4
280	536	0.158	0.661	38.2
		0.164	0.686	39.7
348	658	0.156	0.654	37.7
424	795	0.150	0.628	36.3

These results, presented in Figure 5, show good agreement with WANL thermal conductivity values at 81°C (178°F). Figure 5 also summarizes the literature data^(7, 9) for both the high and low thermal conductivity values for various graphites over a wide temperature range. Thermal conductivity of isostatic P-03 is seen from Figure 5 to be intermediate between the limiting values of lamp-black base grade CEQ and the highly graphitized grades AGOT, H4LM, and CS-312.

MECHANICAL PROPERTIES OF ISOSTATIC P-03 GRAPHITE

This section presents the results of measurements of strength, Young's modulus, and Poisson's ratio on isostatically molded P-03 graphite. Strength measurements were made in tension, flexure (several techniques), and compression. Of these, only tensile tests could be conveniently performed above room temperature; tensile strength of P-03 graphite was consequently measured up to 2700° C (4900° F).

Room-temperature strength measurements were supplemented for selected specimens by determination of stress-strain curves. The strains were measured by cementing resistance-type strain gages to the specimen surface. Both Young's modulus and Poisson's ratio were determined from data obtained in this manner.

Tensile Strength at Room Temperature

Measurements of tensile strength at room temperature were made with two specimen designs (Figure 6):

1. Shoulder-loaded type, characterized by a 0.50"D x 2.0"L gage section and a 4.75-inch total length.
2. Thread-loaded type, having a 0.25"D x 1.0"L gage section and a 4.0-inch total length.

The shoulder-loaded specimen was suitable for use with strain gages, while the thread-loaded type could be tested most readily at elevated temperatures. All tests were conducted at a 0.02 inch per minute strain rate, except where stated otherwise.

The shoulder-loaded specimen was found to be unsatisfactory for reliable testing of P-03 graphite, despite its successful use in testing the weak, coarse-grained graphites (tensile strength below 2000 psi). Undesirable sub-head fractures were encountered in 75% of the 36 P-03 specimens tested; the cause of these fractures has not been completely defined. Sub-head fractures were also encountered in testing similar specimens machined from ATJ graphite.⁽⁴⁾ These fractures could, however, be readily eliminated by reducing the gage section's diameter from 0.50-inch diameter to 0.40 inch, thereby increasing the area ratio between the loaded shoulder and gage section.

The following test results were obtained on 36 shoulder-loaded specimens machined from the center and each end of log No. 1:

<u>ORIENTATION AND FRACTURE</u>	<u>NO. OF TESTS</u>	<u>AVERAGE T.S.</u> (psi)	<u>INDIVIDUAL TENSILE STRENGTH VALUES</u> (psi)
Axial, In-gage	7	4800	4450, 4660, 4740, 4780, 4910, 4940, 5130
Axial, Sub-head	17	4865	3600, 4190, 4490, 4730, 4780, 4790, 4960, 4990, 5000, 5000, 5050, 5080, 5080, 5120, 5160, 5210, 5480
Radial, In-gage	2	4345	3860, 4830
Radial, Sub-head	10	4755	4030, 4510, 4540, 4620, 4720, 4790, 4870, 5070, 5160, 5240

Tensile strength of specimens with sub-head fractures was defined as the maximum nominal stress reached by the gage section for the fracture load. The nominal stresses at the site of sub-head fracture were about 52% lower, although the exact values could not be calculated due to the variation of cross section with the fillet radii.

Stress vs. Strain in Tension

Twelve of the thirty-six shoulder-loaded specimens from log No. 1 were equipped with resistance-type strain gages to define the tensile stress-strain behavior of isostatic P-03 graphite. Six axial and six radial specimens were tested at room temperature. There was no significant difference between these two groups in their deformation behavior. The measured strain values are listed in Table 2, together with the calculated values of Young's modulus; the latter are tangent moduli calculated for consecutive 500-psi increments of applied stress. Figure 7 shows the average and limiting stress-strain curves for P-03.

The tangent Young's modulus is plotted in Figure 8 as a function of the applied stress. It was found to decrease at higher stress levels. There was no significant difference in the average

modulus values between axial and radial orientations. The stress vs. strain results at fracture, together with the resulting values of the secant Young's modulus, were observed to be the following:

STRESS AT FRACTURE (psi)	MODE OF FAILURE	STRAIN AT FRACTURE		SECANT MODULUS	
		Radial (mils per inch)	Axial	Radial (10 ⁶ psi)	Axial
3860	In-gage	2.605		1.48	
4540	Sub-head	2.845		1.60	
4660	In-gage		3.020		1.55
4720	Sub-head	2.770		1.70	
4830	In-gage	3.140		1.54	
4870	Sub-head	2.840		1.72	
4940	In-gage		3.080		1.60
5000	Sub-head		2.910		1.72
5050	Sub-head		3.010		1.68
5080	Sub-head		3.050		1.66
5130	In-gage		3.300		1.55
5240	Sub-head	3.180		1.65	

Finally, an effort was made to correlate the observed strength of these tensile specimens with their stress-strain behavior at sub-fracture loading. These results, shown in Figure 9, indicate that detection of weak specimens from their pre-fracture stress-strain curves is not feasible. The weakest tensile specimen had exhibited the highest deformation at all stress levels, but its detection by this criterion would have been greatly hampered by the large spread in the strain values of the other specimens.

Tensile Tests at High Temperatures

Thread-loaded specimens (Figure 6) used for high-temperature tensile tests were machined from the central section of log No. 3. Comparison data for the sub-size isostatic P-03 stock were available from the previous tests.⁽⁵⁾ Graphite density was in the 1.83 to 1.85 g/cc range for log No. 3, averaging approximately 1.84 g/cc; the sub-size stock was somewhat more dense, ranging from 1.84 to 1.87 g/cc. All specimens were oriented parallel to the cylinder axis.

The high-temperature tests were made between room temperature and 2800° C (5070° F) on sub-size material, and up to 2500° C (4530° F) on the scaled-up stock. The test results are

presented in Table 3, and summarized in Figure 10. Specimens machined from the scaled-up P-03 log were found to be somewhat stronger than those from the small developmental stock.

High-Temperature Elongation

The length of individual tensile specimens was measured with a micrometer before and after testing to define the magnitude of graphite elongation at various temperatures. Elongation values larger than one mil (0.001 inch) over the nominal 4.0-inch specimen length could be reliably determined by this technique. Values smaller than one mil were not considered reliable due to the possibly imperfect fit at the specimen fracture face. Figure 11 summarizes the high-temperature elongation data for isostatic P-03 graphite. Elongation is seen to increase rapidly with temperature above 1800° C (3270° F).

Some additional data on graphite elongation could be gleaned from the testing machine chart's record of load and cross-head motion. This data provided information on the specimen's plastic behavior near its failure load, including an independent value for the specimen's elongation which was found to be in good agreement with the micrometer measurements. The two sets of data were combined in Figure 12 to show the qualitative influence of test temperature and applied stress on specimen elongation.

High-Temperature Tensile Fatigue Tests

Two thread-loaded tensile specimens (Figure 6) were used to explore the behavior of P-03 graphite in tensile fatigue. These tests were made at 2000° C (3630° F), where the P-03 graphite's ductility is 200-300% greater than at room temperature. To minimize possible creep effects, the cross-head travel rate was increased to 0.20 inch per minute, or ten times the value used for tensile tests.

The specimens were cyclically loaded in tension between a pre-set maximum stress and a low residual stress. This test was arbitrarily terminated after one hundred cycles in the absence of failure. The following results were obtained from these preliminary tests on axial specimens from log No. 3:

TENSILE STRESS RANGE		TEST TEMPERATURE		SPECIMEN BEHAVIOR
MINOR	MAJOR	(°C)	(°F)	
(psi)	(psi)			
Specimen No. 1:				
40	2570	2000	3630	No fracture after 100 cycles;
140	6155	2000	3630	No fracture after 100 cycles; Post-test cumulative elongation: 2.0 mils

Results of tensile re-test on this specimen at 0.02 inch per minute cross-head rate and 2000°C (3630°F) : Tensile strength = 7640 psi, Elongation = 0.5 mil.

Specimen No. 2:

140	6155	2000	3630	No fracture after 2 cycles
140	6155	2000	3630	

Cross-head rate was increased from 0.2 to 2.0 inches per minute; specimen broke at 5315 psi, with 1.1 mil cumulative elongation.

One regular tensile specimen (machined from the same site in log No. 3 as the above specimens) was tested at 2000°C (3630°F) and 0.02 inch per minute cross-head rate; its tensile strength and elongation were 9080 psi and 2.0 mils, respectively. Comparison of these results with the preliminary tensile fatigue data indicates that exposure of graphite to high-temperature tensile fatigue conditions serves to lower its strength.

Compression Tests

All compression tests on the isostatic P-03 graphite were made at room temperature. Specimens were usually loaded to destruction at a cross-head rate of 0.020 inch per minute. Their densities were determined from the weight-to-volume ratio (prior to testing) to characterize the material and allow a meaningful comparison of results with those from the sub-size P-03 given previously.⁽⁵⁾

Compression specimens from log No. 1 were secured from broken flexure bars; their final dimensions were 0.75 x 0.75 x 1.50 inches. The load was always applied on the 0.75 x 0.75-inch faces. Thirty specimens were tested, representing ten each from the center and both ends of the log. Four specimens were equipped with resistance-type strain gages to define the compressive stress-strain curves.

Compression tests on log No. 3 were made on thirteen small cylindrical specimens (size 0.50"D x 0.90"L). This was also the specimen design used previously to determine compression strength of sub-size graphite, and the two sets of results are therefore directly comparable. One specimen from log No. 3 was equipped with resistance strain gages.

Results of all compression strength tests are summarized in Table 4 and Figure 13. Cylindrical specimens were found to be about 15% stronger than the secondary specimens obtained from broken flexure bars. The latter specimens exhibited both low strength and a large scatter; their results are not regarded as representative of the material's inherent strength. Comparison of the axial compressive strength between the sub-size (7"D x 21"L) and scaled-up (7"D x 72"L) isostatic P-03 stock shows substantially no degradation in strength for the larger material.

Stress vs. Strain in Compression

Four compression specimens from P-03 log No. 1 and one from log No. 3 were equipped with resistance strain gages. The following results were obtained on these specimens at fracture:

<u>LOG AND ORIENTATION</u>	<u>SPECIMEN SIZE</u> (inches)	<u>DENSITY</u> (g/cc)	<u>COMPRESSIVE STRENGTH</u> (psi)	<u>TOTAL STRAIN</u> (mils/inch)
No. 1, Axial	0.75 x 0.75 x 1.50	1.813	14,550	9.67
		1.825	16,480	9.48
No. 1, Radial	Same	1.819	17,770	11.45
		1.825	18,250	10.56
No. 3, Axial	0.50"D x 0.90"L	1.830	21,250	10.93

Compression-loaded specimens are thus seen to undergo roughly 1.0% axial deformation prior to fracture.

The stress-strain data from compression tests are summarized in Table 5. Values of tangent Young's modulus, calculated for 1000-psi stress intervals, are also given in this table. The stress-strain curves are plotted in Figure 14 to define the magnitude of specimen deformation as a function of the applied loading. Figure 15 shows the appearance of the compression stress-strain curve for

repetitive loading conditions; the hysteresis upon unloading is relatively small and the specimen acquires a small permanent contraction whose magnitude is about 5% of the total strain reached at the maximum loading.

The calculated values of tangent modulus are plotted in Figure 16 as a function of the applied compressive stress. They are seen to decrease upon the initial loading, going from 2.0×10^6 psi at very low stress to 1.4×10^6 psi at the 13,000 psi stress level; upon continued loading the value of tangent modulus begins to increase, reaching 2.6×10^6 psi at the 18,000 psi stress level. No marked dependence was noted on either the size or orientation of these specimens. The modulus does, however, increase with graphite's density.

Poisson's Ratio in Tension and Compression

One compressive and one tensile specimen were equipped with resistance strain gages in order to define the magnitude of Poisson's ratio in both compression and tension. Both specimens had 0.50-inch diameter. The resulting values of Poisson's ratio are presented in Figure 17.

Flexure Tests on P-03 Graphite

Flexure strength of graphite (also called transverse strength) is a parameter related to the material's tensile strength. It is a relatively easy measurement to make in large quantities for quality control purposes. A small number of flexure tests was therefore undertaken on isostatically molded P-03 to establish the extent to which they reflect this material's quality, and to allow correlation with other properties.

Flexure tests were made primarily on simple beam specimens in four-point loading. The machine load was applied on a specimen by rounded steel pins in a hinged fixture designed to eliminate off-center loading. The following specimens and loading conditions were used, unless stated otherwise:

<u>LOG NUMBER</u>	<u>SPECIMEN SIZE</u>	<u>OUTER SPAN</u>	<u>INNER SPAN</u>	<u>CROSS-HEAD RATE</u>
1	0.75x0.75x5.00"	4.50"	1.50"	0.020 inch per min.
3	0.44x0.44x3.50"	3.00"	1.00"	0.020 inch per min.

In addition to these principal tests, a small number of special flexure tests was undertaken to define the effects of specimen geometry and loading conditions on flexure strength. All values of flexure strength were calculated from the conventional modulus-of-rupture formulas applicable to the particular test geometry. No effort was made to apply the small corrections necessary to compensate for the load-pin friction and other effects. Specimen densities were determined from the weight-to-volume ratio of as-machined flexure bars in order to characterize the test material.

The flexure test results are presented in Table 6. There was no loss in flexure strength of the scaled-up P-03 logs when compared to the previously obtained data⁽⁵⁾ for sub-size material:

<u>STOCK SIZE</u> (inches)	<u>AVERAGE DENSITY</u> (g/cc)	<u>AVERAGE FLEXURE STRENGTH</u> (psi)	<u>NUMBER OF SPECIMENS</u>
7"D x 21"L	1.845	6265	6
7"D x 72"L	1.842	7190	6

The scaled-up stock had somewhat higher strength than the sub-size material. No significant difference in strength between the axial and radial orientations was found.

One series of flexure tests was made to determine the difference in strength between 3-point and 4-point loading. Specimen cross-section was 0.44 x 0.44 inch. The following results were obtained:

<u>LOG NUMBER AND ORIENTATION</u>	<u>SPECIMEN DENSITY</u> (g/cc)	<u>4-POINT TEST</u> (psi)	<u>3-POINT TEST</u> (psi)	<u>RATIO $\frac{3-POINT}{4-POINT}$</u>
No. 1, Axial	1.817	7300	8140	1.115
	1.818	6580	7570	1.150
No. 3, Axial	1.840	7740	8280	1.070
	1.844	7740	8840	1.140
	1.846	7700	8280	1.075
	1.851	8015	8620	1.075

Average: 1.104

Flexure strength in 3-point loading is about 10% higher than that determined from 4-point loading.

DISCUSSION: PHYSICAL PROPERTIES OF P-03 GRAPHITE

Fabrication History

The production aspects of isostatically molded 7"D x 72"L logs of P-03 graphite had presented a formidable problem. The manufacturer stated that scale-up of the largest developmental stock (7"D x 21"L) was technologically feasible,⁽⁶⁾ although it had not been attempted. This scale-up to larger stock size was very desirable since the previous evaluation of a developmental stock⁽⁵⁾ showed P-03 to be a promising structural grade which offered a major strength improvement over the presently available conventional graphites. It was decided to undertake the scale-up on a limited basis to determine the process feasibility and establish the magnitude of any strength losses with larger size. The subsequent fabrication of eight 7"D x 72"L logs showed the scale-up to be feasible. This achievement would not have been possible without the harmonious cooperation demonstrated by the manufacturer and the isostatic press facility.⁽¹⁰⁾

Originally, the scale-up effort on P-03 was intended to increase both the diameter and length of the largest (7"D x 21"L) developmental stock. However, the diameter increase was ruled out as requiring more fabrication effort than could readily be covered within the scope of available time, manpower, and funding. Therefore, only the increase in length was undertaken. Details concerning the fabrication of isostatically molded P-03 graphite are considered to be proprietary with the manufacturer, and were thus unavailable for presentation in this report. The prime purpose of the scale-up project was to provide the 7"D x 72"L P-03 graphite for evaluation testing; this objective has been achieved.

Density and Microstructure

Mechanical properties of a given graphite are governed largely by its coke particle size, density, and the degree of structural anisotropy resulting from the forming process. Isostatically molded P-03 graphite is essentially isotropic, and its strength is therefore uniformly high regardless of orientation. The high density of P-03 provides good surface finish, resistance to abrasion, and close dimensional tolerances on hardware components. The coke particle size is considerably finer than that used in most commercial graphites. All these features serve to make this graphite particularly useful for intricate structural applications that involve high design stresses.

No inherent defects (such as cracks, voids, inclusions, or spongy areas) were detected in the isostatic P-03 graphite upon visual examination of over one hundred machined specimens. Limited radiographic inspection of P-03 supported the negative results of visual examination, indicating that the isostatic material is uniformly free from such flaws. Additional evidence for the material's uniformity is evident from density measurements, which showed that specimen densities varied only between 1.81 and 1.85 g/cc regardless of the log or location being sampled; this is a negligibly small variation. The typical log's microstructure is shown in Figure 2; it is characterized by an abundance of fine particles in the interstices between the principal grains, which presumably strengthen the graphite by preventing the formation of large unfilled pores. The degree of bonding between individual particles is quite well developed.

Conventional graphites are usually found to exhibit an anisotropic structure which results from preferential orientation of coke grains during the shaping process. In the case of isostatic forming, the grains are expected to be oriented randomly, thereby minimizing anisotropy. Isostatically molded P-03 is hence believed to have uniform properties in all orientations. This uniformity is evident for example, from the lack of any marked difference between the radial and axial values of electrical resistivity and linear thermal expansion.

Thermal Conductivity

One of the important physical parameters required of structural graphite is its thermal conductivity. This is of considerable interest in those structures exposed to either non-uniform temperatures or transient heating. Thermal conductivity is also one of the more difficult properties to measure with good precision, as well as one that is strongly affected by neutron irradiation in a nuclear environment.

Preliminary measurements of thermal conductivity on the isostatically molded P-03 stock (log No. 3) gave the following value at 82° C (180° F):

$$\begin{aligned}
 &0.159 \frac{\text{cal-cm}}{\text{cm}^2 \text{ sec } ^\circ\text{C}} \quad (\text{Scatter for 3 tests: } \pm \begin{matrix} 14\% \\ 8\% \end{matrix}) , \\
 &\text{or} \\
 &0.666 \frac{\text{watt-cm}}{\text{cm}^2 \text{ } ^\circ\text{C}} , \\
 &\text{or} \\
 &38.5 \frac{\text{BTU-ft}}{\text{ft}^2 \text{ hr } ^\circ\text{F}} .
 \end{aligned}$$

This result was in excellent agreement with thermal conductivity values determined on equal-density sample by Battelle Pacific Northwest Laboratories.⁽⁸⁾

Thermal conductivity of P-03 is compared in Figure 5 to that of two conventionally fabricated types of graphite. These types, chosen to give the upper and lower limits for thermal conductivity of graphitized materials, were the following:

1. Coarse-grained extruded grades H4LM, AGOT, and CS-312, characterized by high conductivity.^(7, 9)
2. Fine-grained molded grade CEQ, characterized by low conductivity.⁽⁷⁾

Thermal conductivity of isostatically molded P-03 (Figure 5) is well within these "limiting" values at all temperatures.

The coarse-grained extruded grades, such as H4LM, have a high thermal conductivity which varies greatly with temperature, and is decreased by exposure to neutron irradiation. The petroleum-residue coke from which these graphites are made has a well-developed crystalline orientation which, by analogy to single crystal graphite flakes, results in a high thermal conductivity along the basal planes and low conductivity across them. The preferential grain alignment during extrusion then leads to high conductivity along the extrusion axis and a lower one along the diameter, corresponding to the with-grain and across-grain orientations, respectively.

The effectiveness of fast neutron irradiation at an ambient temperature in decreasing thermal conductivity of the coarse-grained H4LM graphite is indicated by the following results:⁽¹²⁾

TEST TEMPERATURE		GRAIN ORIENTATION	NUMBER OF TESTS	THERMAL CONDUCTIVITY		
(°C)	(°F)			Low	Average	High
(cal-cm)/(cm ² sec °K)						
A. <u>Unirradiated control stock:</u>						
44	111	WG	3	0.440	0.459	0.471
B. <u>After irradiation to 1.2 x 10¹⁷ nvt of fast energy(> 1 mev) neutrons:</u>						
50	122	WG	4	0.249	0.252	0.258

Thermal conductivity decreases by 45%, primarily as the result of increased structural disorder within the initially well-crystallized coke particles. Comparative results are not available for P-03 graphite, but would be predicted to be less than that found for H4LM.

The lower limit to thermal conductivity of graphite is determined in Figure 5 by the CEQ molded grade; this is a relatively isotropic, fine-grained product made with lamp-black and heated to graphitization temperatures. Grade CEQ is typical of disordered graphites with irregularly oriented microcrystalline structure.

DISCUSSION: STRENGTH PROPERTIES OF P-03 GRAPHITE

The previous evaluation of P-03 graphite showed that isostatically molded developmental stock has uniformly high strength in all directions.⁽⁵⁾ This conclusion has been amply confirmed for the scaled-up isostatically molded P-03 logs in this study. The strength of isostatic P-03 was found to be higher than that of the best structural graphites (excluding, perhaps, a few small-size developmental materials) which have been tested in the course of WANL's graphite evaluation program. Therefore, when both the high strength and large stock size are considered together, the scaled-up isostatic P-03 becomes a uniquely superior graphite.

The only structural graphite stock available in the 7"D x 72"L size prior to the fabrication of isostatic P-03 logs was the conventionally extruded graphite, a generally weak material abounding with miscellaneous flaws.⁽¹⁾ The strength properties of extruded graphite could be improved somewhat through one or more reimpregnation treatments; these consisted of forcing

the pitch-like binder under hydrostatic pressure into the inter-connected pores of the graphitized extrusion, followed by a baking treatment to drive off the volatiles and carbonize the binder. The resulting material was still weaker structurally than P-03, as shown in the following comparison:

<u>PROPERTY</u>	<u>UNITS</u>	<u>EXTRUDED AND RE- IMPREGNATED GRAPHITE</u>	<u>ISOSTATICALLY MOLDED GRAPHITE</u>
Stock size	inches	7"D x 72"L	7"D x 72"L
Grade		890 - S	P-03
Density	(g/cc)	1.68 - 1.78	1.81 - 1.85
Flexure Strength, WG	(psi)		
Low		3780	6445
Average		4300 (16)	7190 (6)
High		4940	7740
Tensile Strength, WG	(psi)		
Low		2480	3600
Average		2730 (13)	4865 (17)
High		2910	5480
Compressive Strength, WG	(psi)		
Low		6120	18,150
Average		6560 (6)	20,300 (6)
High		6860	21,350
Source of data		WANL-TME-741	This report

The results given above are typical values determined on specimens of equal size, with the number of tests given in parentheses. Strength values of extruded graphite in the across-grain direction would be even lower than the quoted with-grain values. Isostatically molded P-03 graphite is noted to exhibit both a higher density and superior strength.

Tensile Strength and Elongation

Sufficient test data has been generated on P-03 graphite to define its tensile stress-strain behavior at room temperature. The data from high-temperature tests are less conclusive, although it permits the stress-strain behavior to be characterized up to 2800°C (5070°F). These tests have shown that isostatically molded P-03 is uniformly strong in both the axial and radial orientations. No evidence was found for any significant grain alignment effects on either the tensile strength or Young's modulus.

Tensile strength measurements with shoulder-loaded specimens were not entirely reliable, since three-quarters of these specimens broke at the shoulder (nominal load-bearing cross section = 0.302 in^2) instead of the gage section (cross section = 0.198 in^2). This occurrence could be explained by either of the following assumptions:

1. The presence of triaxial stress at the loaded shoulder serves to induce local failure by shear.
2. Triaxial stress at the loaded shoulder tends to activate a microstructural stress-raiser which increases local stress to the values which equal or exceed the gage section's tensile stress.

Empirically, it has been found that reducing the gage section from 0.50 inch to 0.40 inch in diameter eliminates sub-head fractures altogether.⁽⁴⁾ Also, such fractures have never been observed in the thread-loaded tensile specimens, where the thread roots might be expected to act as stress-raisers. Sub-head fractures are hence considered to be caused by improper specimen design.

Tensile strength of P-03 increases with temperature, reaching a maximum near 2300°C (4170°F). The magnitude of this increase is about 100%, in good agreement with tensile data for other graphites.⁽⁴⁾ The behavior of initial Young's modulus is expected to increase slightly with temperature, and it may be estimated that this increase will be approximately 50% of the 1.7×10^6 psi value observed at room temperature.

The specimen elongates when loaded in tension, deforming both elastically and plastically during the high-temperature tests. The magnitude of elastic elongation has not been determined at high temperatures. The total plastic deformation in the specimens was determined by measuring their length before and after each test. The magnitude of this deformation amounts to less than one mil for the entire shoulder-loaded specimen at temperatures below 1500°C (2730°F), but increases considerably at higher temperatures. Most of the deformation is presumed to occur within the gage section, with only a minor contribution by the specimen's threads and shoulders. At low temperatures, about 65% of total elongation within the 4.0-inch long specimen is estimated

to occur within the 1.25-inch gage length and the tangential portion of inner fillets; in this case the in-gage plastic strain (measured in mils per inch) is numerically equal to 52% of the specimen's elongation (mils). At high temperatures the percentage of in-gage elongation is expected to be even higher.

An effort had been made to define the high-temperature (2000°C or 3630°F) tensile strength of isostatic P-03 under cyclic tensile loading. One specimen had successfully undergone one-hundred cycles of exposure to 6155-psi maximum tensile stress, a loading which amounted to 68% of the ultimate strength at this temperature. Upon being cooled to room temperature and then re-tested in tension at 2000°C (3630°F), this specimen broke at only 7640 psi or 84% of the expected strength; this indicates some susceptibility of graphite to structural damage under high-temperature cyclic loading. Another specimen was successfully tested through two cycles of exposure to the 6155 psi maximum tensile stress; it failed, however, at only 5315 psi (about 58.5% of expected strength) when the loading rate was suddenly increased by a factor of ten at the beginning of the third cycle. This indicates a possible sensitivity of graphite to early failure under rapidly applied loads. No further tests were made to extend these exploratory results.

Compression Tests

Sufficient data has been obtained to determine both the strength and the stress-strain curves for P-03 in room-temperature compression. No dependence on orientation is observed in these results, but both the strength and Young's modulus tend to increase at higher density.

Compressive strength of the scaled-up isostatically molded P-03 is on the order of 20,000 psi, a value considerably higher than that of both conventionally molded and extruded structural graphites. Review of the compression test results showed that the secondary specimens machined from the broken flexure bars were weaker by about 1,000 psi (or 5%) than cylindrical specimens with equal density. The latter specimens also had somewhat smaller scatter, and should hence be more representative of the actual strength values.

The tangent Young's modulus (Figure 16) in compression tends to decrease with applied stress up to about 13,000 psi. At higher stresses, this modulus begins to increase up to the specimen's fracture. The Poisson's ratio shows a uniform increase with applied compressive strength (Figure 17) up to the point of fracture. This data is expected to facilitate stress analysis in P-03 structures.

Compression tests could not be carried out at elevated temperatures. Compressive strength of structural graphites, however, generally increases with temperature in much the same way as for tensile loading. Near 2300°C (4170°F) the isostatically molded P-03 might therefore have a compressive strength of up to 40,000 psi.

Diameter Scale-up Possibilities for P-03

In view of the successful scale-up in length of the developmental P-03 stock, it is of considerable interest to increase the log diameter. The 7-inch diameter is rather restrictive with respect to the fabrication of any large monolithic hardware shapes. Its scale-up to larger dimensions is considered to be a difficult undertaking, due primarily to the problem of removing the volatile decomposition products generated within the log by the binder during the carbonization bake treatment. When inadequately vented, these volatiles could readily build up within the impermeable material, producing internal damage. Once the scaled-up log has been successfully processed through the carbonizing bake, it may be readily graphitized with no major problems.

To explore the possibility of diametral scale-up for P-03, we may refer to the industrial technology dealing with extruded graphites. Conventionally extruded graphites are commonly fabricated in 72-inch lengths and various stock diameters ranging from a fraction of an inch to 45 inches. To cover this size range with a single type of coke, the manufacturers have found it necessary to control the size distribution of coke particles intended for the "green mix"; a useful fabrication parameter in this respect is the maximum coke particle size (often called the maximum grain size) used in the coke mix. This is defined under plant conditions as the coarsest

screening sieve used for coke particle control prior to blending with the mix. The maximum coke particle sizes for extrusion mixes appear to have been standardized at 8, 16, 33, and 65 mils. The larger the size, the weaker is the final material. The high strength of P-03 is due largely to its fine-grained particle mix, with the maximum coke size estimated to be 3 mils.

A brief survey of the manufacturers' product data has indicated that 33-mil mix is being used to produce extrusions ranging from 3 up to 40 inches in diameter. It is therefore quite probable that isostatically molded graphite could be produced up to 40 inches in diameter with such a mix. Availability of isostatic P-03 graphite in this size range would considerably enhance its usefulness as a premium structural material. However, a strength reduction of perhaps 40% can be expected by comparison with the size evolution of extruded graphites.

CONCLUSIONS

The following principal conclusions have been reached from this study:

1. Isostatically molded graphites of the P-03 type are superior to conventionally fabricated structural graphites, and represent a major advance in the graphite industry.
2. Isostatically molded P-03 shows no evidence of preferential grain orientation, and the latter's influence on both the physical and mechanical properties is negligible.
3. Fabrication of the 7"D x 72"L shapes from isostatic P-03 has been successfully accomplished; this represents a major scale-up in length over the developmental stock.
4. The scaled-up P-03 graphite showed no detectable loss in strength relative to sub-size developmental stock, and had a flaw-free structure.
5. Diametral scale-up may be attempted in the future, if required for hardware fabrication; this is expected to be considerably more difficult to accomplish than the successful scale-up in length.
6. Sufficient mechanical and physical data has been generated in this study to allow the consideration of isostatically molded P-03 graphite for those NERVA applications where a high-strength, uniformly isotropic dense graphite is required.

PART II

DEVELOPMENT OF CEMENTED P-03 STRUCTURES

INTRODUCTION

The inner reflector cylinders used in the NRX-A1, NRX-A2, and NRX-A3 reactors represent the quality of graphite hardware made from H4LM electrode-grade graphite. This material is satisfactory for the intended industrial uses, but is not a high-quality graphite for structural applications. Its high permeability, coarse grain structure, abundant porosity, low density, high ash content, low strength in comparison with other graphites, and poor radiographic quality are a few of the disadvantages which are inherent in this class of graphite.⁽¹³⁾ Although the initial material cost may be low, the cost of the finished acceptable reflector is high, and the overall quality of this part is unpredictable.

This portion of the report describes the state-of-the-art methods related to fabrication of a cemented P-03 barrel. While P-03 has not been molded isostatically in sizes large enough to yield a monolithic cylinder, it has been produced in sizes which can be machined into staves that subsequently can be cemented together. This method of fabrication potentially offers more control on the starting materials, makes inspection of the graphite staves by radiography more valid, and, in general, offers a method of producing a sound, uniform inner reflector whose strength is considerably higher, whose permeability is lower and whose performance is more predictable. In addition, it is suggested that isostatic molding not only eliminates the anisotropy of the material, but may produce uniformity of changes occurring through radiation effects; i.e., preferential shrinkage (or swelling) in one direction with most extruded graphites or those molded graphites exhibiting pronounced anisotropy.

PROCESS DEVELOPMENT

The functions of the cemented joint in the staved barrel are to strengthen the staves, and to reduce the leakage of hydrogen between the stove interfaces. Prior WANL experience, reported in WANL-TME-847⁽¹⁴⁾ with P-514 cement, suggested its use with graphitized P-03 graphite. The heat treatment cycles for this cement may be summarized as follows: curing at a rate of 15°F/hr. to 260°F, carbonization from room temperature to 1600°F at a rate of 120°F/hr. in argon, and graphitization at 3450°F for 6 hours in argon.

Another process considered is the "in-process" method of cementing. It seemed reasonable that a cemented joint which could be produced before the carbonization of the graphite staves would be stronger, and perhaps more impermeable, than a joint produced after the staves had been graphitized. In practice, the proprietary binder that was used in the P-03 was applied on two isostatically molded pieces of P-03 in the condition prior to curing. The pre-wetted faces of the two pieces were placed together to effect the joint. After this, the blocks were placed in the curing furnace where the cemented blocks were covered with a carbon and sand packing. The cure and carbonization were done in the same furnace, which is heated slowly until the binder in the cemented blocks is converted to carbon. Subsequently, the blocks were resistance-heated to graphitize them.

Early in this program, small cemented blocks of P-03 were produced by these methods. Flexure and tensile specimens were machined from these blocks for a quantitative comparison of the properties of cemented joints produced by the two methods. Metallographic examination of these joints was made as well as permeability measurements. After these tests were made, four cemented staved cylinders were produced by cementing as-graphitized staves with P-514 cement. Since the "in-process" cementing method was not sufficiently developed, cylinders were not evaluated. These barrels were made for hoop stress tests which were conducted by the Engineering Mechanics Group at WANL.

EVALUATION OF THE P-03 CEMENTED JOINTS

Mechanical Tests

During cementing of graphite for the NRX-B effort,⁽¹⁴⁾ it appeared that machining adversely affected the strength of a carbonaceous joint if the machining was done after the cement was graphitized. To determine if this would be true with cemented P-03, tensile specimens were machined from P-03 blocks cemented with P-514 cement while the cement was in the as-cured condition and compared to those machined after the cement had been graphitized. The former specimens were carbonized and graphitized before being tested in tension. The type of cemented tensile specimen used is shown in Figure 18. Note that a constant radius was used so that the cemented joint had the smallest cross section to promote tensile failure in the joint.

The average tensile strength of the specimens machined in the as-cured state was 3070 psi, while those specimens machined after graphitization averaged 2350 psi. This indicates that machining lowered the tensile strength of a P-03 cemented specimen; therefore, all machining of P-03 joined with P-514 cement mentioned hereafter was done after the cure cycle. The carbonization and graphitization of these cemented joints was done after machining.

Another group of tensile specimens prepared by both the "in-process" and P-514 methods was tested at room temperature, 2375°F (1300°C), 3450°F (1900°C), and 4536°F (2500°C). Figure 19 shows the comparative strength of the joints produced by the two methods. With few exceptions, the "in-process" joints failed in the graphite adherend rather than in the joint; however, the P-514 samples all failed in the cemented joint. These data confirm that the "in-process" method produced stronger joints than the P-514 process.

Ring flexure tests were made on annular samples illustrated in Figure 20. These samples were stressed with the cemented joint both parallel and normal to the load axis. The results of these room temperature tests are as follows:

	<u>AVERAGE RING FLEXURE STRENGTH</u>	
	<u>Joint Orientation</u>	
	<u>Parallel</u>	<u>Perpendicular</u>
P-514 joint	2955 psi	6543 psi
"In-process" joint	7255 psi	8197 psi

In all cases, the failure occurred in the joint regardless of method of processing or specimen orientation. Again, the "in-process" joint was significantly stronger.

Shrinkage cracks were observed to be present in the material joined using the "in-process" technique and they will be discussed in a subsequent part of the report. The shrinkage cracks present in the "in-process" joint area were oriented to have a minimum effect on the tensile or ring flexure strength previously reported. The effect of these undesirable cracks on the flexural strength of the "in-process" cemented joint was demonstrated when the cracks were oriented in samples so they were in the plane of the maximum fiber stress. Figure 21 illustrates the

orientation of the cemented joint and cracks in these samples. The results of four of these tests show that the cracks lower the flexural strength of the graphite by approximately 1500 psi; however, the strength of these samples was not as low as might be expected with cracks as large as these in the cross section of the samples.

The standard flexural test specimen has the cemented joint located in the middle of and across the width of the test piece. Four-point loading of specimens of this type showed that the average flexural strength of the P-514 joint was 2650 psi, while the in-process joint was 5130 psi.

Metallographic Examination of Cemented Joints

The graphitized joints made with P-514 cement are porous since the binder of this cement loses water and other decomposition products during polymerization. A typical P-514 joint is shown in Figure 22 which reveals rather large pore areas in its microstructure. These pores would be filled by the colloidal silica impregnant which is used to make the reactor grade inner reflector barrel less permeable to hydrogen.

Examination of the "in-process" joints shows both extremely good and bad areas. In most of the joint, pores or voids are smaller than those in the P-03 on either side of the joint. Large shrinkage cracks appear intermittently in the P-03 on either side of the joint. Figure 23 shows a typical good "in-process" joint area. While the good joint areas should be as impermeable as the P-03, these shrinkage cracks would leak severely and would probably not be clogged by the colloidal silica impregnant treatment because of their large size.

Permeability Tests on Cemented Joints

Permeability measurements were made on the P-514 and the "in-process" joints in both the impregnated and unimpregnated condition. The specimens used for these tests were machined so that the cemented joint would be in the center of the specimen and would extend through the thickness of the specimen. This specimen was 1.10" OD by .500" in thickness. Control samples of isostatically molded P-03 and the cemented samples were subjected to 50 psi, 100 psi, 150 psi,

and 200 psi of hydrogen. The leak rates of these samples are summarized in Figure 24. Note that these data include the impregnated cemented joints and control P-03 samples as well. While impregnation with colloidal silica, according to WANL PS294545 (Rev. 3), does reduce the leak rate of the cemented samples, their leak rate is higher than the impregnated P-03 control samples. The best impregnated joint was an in-process sample with cracks; however, the most consistent samples were the P-514 impregnated joints. These tests indicate that with "in-process" joints a cemented cylinder made from isostatically molded P-03 can be produced having low permeability similar to that of the uncemented P-03.

Cementing of 6" Diameter P-03 Staved Cylinders

Four 6" diameter cemented cylinders were produced by cementing 36 staves together with P-514 cement. The 10 degree staves were machined thicker than the finished barrel thickness. Figure 25 shows an array of the staves before they were cemented together. The extra thickness of the staves provides more joint area so that machining of the cemented barrel could be done while the cement was in the as-cured state. Also, it provided more support for the cemented joint than the finished size stave and permitted the cemented assembly to be free standing. The surfaces of the staves were roughened with 200 grit energy paper prior to cementing. Subsequently, the P-514 cement was applied to the staves in generous quantities. Each pre-wetted stave was stacked in place until the assembly was complete. Since pressure is required during the curing of this cement, two hose clamps were used to extrude the excess cement and to support the uncured stave cylinder. The cured assembly is shown in Figure 26. The excess cement left on the surfaces of the barrel was deliberate, so that capillary attraction of the cement for itself could not produce a void or a recess at the joint interfaces during the curing process. The cured assembly was machined to its final size shown in Figure 27. After this, the assembly was subjected to the carbonization and graphitization thermal cycles previously described.

These cemented cylinders, together with the monolithic P-03 cylinders and unbonded stave cylinders, were subjected to a series of tests by the WANL Engineering Mechanics Department. These tests involved pressurizing the exterior of the cylinder hydrostatically so that a comparison

of strength of the different types of barrels could be made. Other data such as the deflection before failure and the mode of failure were observed in these tests. These data will be reported later.

DISCUSSION

The P-514 joint is currently considered more practical than the "in-process" joint for fabrication of the staved barrel or inner reflector. Although the "in-process" joint is considerably stronger, and when free from cracks is less permeable than the P-514 joint, the "in-process" method requires additional development before it can be recommended for full scale hardware production. Small laboratory samples did not show the cracks radiating from the "in-process" joint; however, they did appear on the 8" diameter cemented samples. It is suggested that these cracks could be eliminated by varying the heat treating cycles or by pre-treating the surfaces with binder and then curing the treated adherends prior to cementing and final curing of the cemented assembly. The latter method is not comparable with the process presently used in the manufacturing of P-03, since the curing and carbonization are done in one continuous thermal cycle.

In spite of this problem in scaling-up of the "in-process" method, this type of joint exhibits the highest tensile and flexural strength recorded in this laboratory for a graphitized carbonaceous cement joint. The failure of these joints has been in the graphite adherend rather than in the joint. These observations apply to tests at 2500°C as well as room temperature. The permeability of the impregnated "in-process" joint is almost as low as the impregnated P-03. This is true, however, only when the cracks are absent in the "in-process" joint, since the colloidal silica impregnant apparently cannot fill these cracks completely. It is necessary to mention that the "in-process" joint is very low in porosity in comparison to the P-514 joint. This can explain the low permeability of the impregnated "in-process" joints when cracks are not present in the sample. Although the P-514 joint is not as outstanding as the best "in-process" joint, it is reasonably reproducible. The average tensile strength of a P-03 sample cemented with P-514 cement is 3070 psi, whereas the average ring flexural strength of this type joint is 2955 psi. Its permeability to hydrogen after impregnation is less than impregnated H4LM graphite. These characteristics of the P-514 joint justify its use in the fabrication of the 6" diameter cemented stave cylinders.

The procedure for cementing the staved cylinders differs little from that which was used on the mechanical test specimens. Machining of the cemented barrels was done after the cement was cured rather than after it was graphitized. Inspection of the graphitized joints in these barrels showed most of the joints were good; however, one joint in each barrel was more porous than the others. These inferior joints were caused by the non-uniformity of the stave dimensions and subsequent non-uniformity in pressure distribution on the staves during the cure. It is expected that this large porosity can be eliminated by closer dimensional control and better pressure distribution on the stave assembly during the curing cycle. The high quality and low porosity of the majority of joints are more typical of the results to be expected with a well controlled production process for a full-scale inner reflector cylinder.

CONCLUSIONS

1. Isostatically molded P-03 graphite staves can be joined by both P-514 cement and the "in-process" method; however, the P-514 joint is preferred since it does not produce shrinkage cracks. Its average tensile strength is 3070 psi, whereas its ring flexure strength is 2955 psi at room temperature.
2. The developmental "in-process" joints are the strongest graphitized carbonaceous joints observed in this laboratory. The average tensile strength of these joints is 4900 psi and its ring flexural strength averages 7255 psi at room temperature.
3. The permeability of the impregnated "in-process" joint is very similar to impregnated P-03 graphite without a joint. This is true only when the shrinkage cracks are absent in the graphite on either side of the "in-process" joint.
4. The permeability of the impregnated P-03 with P-514 joints is higher than the "in-process" joint, but it is lower than impregnated H4LM graphite used presently for the inner reflector in the NERVA reactors.
5. A process has been demonstrated for cementing staved P-03 cylinders with P-514 cement. Although these cylinders are only 6" OD, they are approximately to scale and the results certainly warrant the production of a full size cemented stave cylinder to verify the results of this study.

RECOMMENDATIONS

1. P-514 cement should be used for cementing graphitized, isostatically molded P-03 graphite staved cylinders.
2. The "in-process" method should be developed further so that the occasional shrinkage cracks can be eliminated. When these defects are eliminated, a cemented P-03 staved cylinder can be produced by the "in-process" method. This barrel should approximate the strength and permeability to hydrogen of a solid isostatically molded monolithic P-03 barrel after impregnation with colloidal silica.

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TABLE I

THERMAL EXPANSION OF THE ISOSTATICALLY MOLDED P-03 GRAPHITE

<u>TEMPERATURE</u>		<u>RADIAL SPECIMEN 1)</u>	<u>AXIAL SPECIMEN 1), 2)</u>
(°C)	(°F)	(microinches per inch per °C) 3)	
700	1292	3.56	3.39
800	1472	3.68	3.54
900	1652	3.81	3.70
1000	1832	3.94	3.85
1100	2012	4.08	4.02
1200	2192	4.23	4.18
1300	2372	4.38	4.35
1400	2552	4.54	4.52
1500	2732	4.70	4.69
1600	2912	4.87	4.86
1700	3092	5.05	5.04
1800	3272	5.23	5.22
1900	3452	5.42	5.40
2000	3632	5.62	5.59
2100	3812	5.82	5.78
2200	3992	6.02	5.97
2300	4172	6.24	6.16
2400	4352	6.46	6.36
2500	4532	6.68	6.56
Specimen density (g/cc):		1.830	1.831

1) The given values represent average coefficient of linear thermal expansion between 23°C (73°F) and the indicated temperatures.

2) The radial specimen expanded slightly (0.0005 inch per inch) when heated between 1900 and 2500°C; the total time above 1900°C was 240 minutes.

3) In order to convert to microinches per inch per degree Fahrenheit, divide the given values by 1.800.

TABLE 2

STRESS VS. STRAIN RELATIONSHIP FOR ISOSTATICALLY MOLDED
P-03 GRAPHITE IN TENSION AT ROOM TEMPERATURE

APPLIED STRESS (psi)	STRAIN, RADIAL SPECIMENS ¹⁾				STRAIN, AXIAL SPECIMENS ¹⁾			
	Low	Average	High	Tangent Modulus ²⁾ ($\times 10^6$ psi)	Low	Average	High	Tangent Modulus ²⁾ ($\times 10^6$ psi)
	(mils per inch)				(mils per inch)			
0	0	0	0	--	0	0	0	--
500	0.184	0.250	0.288	1.99	0.184	0.223	0.268	2.23
1000	0.436	0.531	0.628	1.78	0.440	0.508	0.552	1.76
1500	0.708	0.815	0.945	1.76	0.720	0.795	0.853	1.75
2000	1.012	1.122	1.277	1.63	1.064	1.095	1.160	1.66
2500	1.312	1.455	1.632	1.50	1.324	1.421	1.500	1.79
3000	1.632	1.786	1.990	1.50	1.630	1.736	1.845	1.59
3500	1.949	2.116	2.370	1.52	1.940	2.069	2.190	1.50
4000	2.285	2.395 (5)	2.580	1.79	2.280	2.405	2.570	1.49
4500	2.602	2.724 (3)	2.930	1.52	2.610	2.755	2.925	1.42

NOTES: ¹⁾ Averaged for six specimens (log No. 1), unless shown otherwise in parentheses.

²⁾ Calculated from the average strain values for the indicated 500-psi stress increments.

TABLE 3
TENSILE TEST RESULTS ON THE ISOSTATICALLY MOLDED P-03 GRAPHITE*

TEST STOCK	TEST TEMPERATURE		TENSILE STRENGTH	SPECIMEN ELONGATION	NO. OF FRACTURES
	(°C)	(°F)	(psi)	(mils)	
Sub-size, (7"D x 21"L)	RT	RT	4080, 4140, 4290, 4400 (Av. 4230)	< 1	1 or 2
Log No. 3 (7"D x 72"L)	RT	RT	4870, 4910, 5150, 5740, 5760, 6360 (Av. 5465)	< 1	1 or 2
Sub-size	1000	1830	5050	< 1	--
Log No. 3	1000	1830	6985	0.8	2
Sub-size	1400	2550	6330	< 1	2
Log No. 3	1400	2550	7850	0.8	2
Sub-size	1800	3270	6390	---	2
Log No. 3	1800	3270	8995	1.8	3
Sub-size	2000	3630	6800	---	2
Log No. 3	2000	3630	8755	1.9	2
	2000	3630	9080	2.0	2
	2000	3630	9565	2.5	3
	2000	3630	7640**	0.5	1
Sub-size	2200	3990	7950	3.0	3
Log No. 3	2200	3990	10,345	3.8	3
Sub-size	2400	4350	8450	10.5	1
Sub-size	2500	4530	7440	11.7	1
Log No. 3	2500	4530	8455	9.3	2
Sub-size	2600	4710	6420	18	1
Sub-size	2800	5070	5180	33	1

*Axial specimens only.

**Previously tested in cyclic fatigue at 2000°C (3630°F).

TABLE 4

SUMMARY OF COMPRESSION TESTS ON THE ISOSTATICALLY
MOLDED P-03 GRAPHITE AT ROOM TEMPERATURE

LOG NUMBER	SPECIMEN ORIENTATION	SPECIMEN DENSITY			COMPRESSIVE STRENGTH VALUES ⁴⁾ (psi)
		Low (g/cc)	Average ³⁾ (g/cc)	High (g/cc)	
1	Axial ¹⁾	1.813	1.815 (4)	1.818	13890, 14550, (15500), 16470, 17100
		1.831	1.835 (5)	1.826	15950, 16480, (<u>16680</u>), 16860, 17630
		1.831	1.835 (8)	1.838	15300, 15410, 17330, (17480), 17590, 17760, 18180, 18920, <u>19240</u>
	Radial ¹⁾	---	1.819 (2)	---	17770, (18080), 18380
		1.821	1.825 (4)	1.828	14740, (<u>17270</u>), 17690, 18250, 18330
		1.831	1.832 (5)	1.834	17500, <u>17920</u> , 18330, (<u>18430</u>), 19140, 19190
3	Axial ²⁾	1.828	1.830 (6)	1.832	20020, 20160, 20190, (<u>20600</u>), 20870, 20980, 21250
		1.833	1.835 (7)	1.838	20030, 20400, 20770, (<u>20900</u>), 21040 21110, 21400, 21400
	Sub-size stock (7"D x 21"L) Axial ²⁾	1.824	1.847 (6)	1.846	18150, 20050, 20150, (<u>20300</u>), 20500, 21280, 21350

NOTES:

¹⁾ Specimen size 0.75 x 0.75 x 1.50 inches.²⁾ Specimen size 0.50"D x 0.90"L.³⁾ Number of specimens is given in parentheses.⁴⁾ Average value of compressive strength is underlined in parentheses.

TABLE 5
STRESS VS. STRAIN IN ROOM-TEMPERATURE COMPRESSION OF ISOSTATICALLY MOLDED P-03 GRAPHITE

APPLIED STRESS (psi)	AXIAL SPECIMEN, 1.813 g/cc		RADIAL SPECIMEN, 1.819 g/cc		AXIAL SPECIMEN, 1.825 g/cc		RADIAL SPECIMEN, 1.825 g/cc		AXIAL SPECIMEN, 1.830 g/cc	
	MEASURED STRAIN (mils per inch)	TANGENT MODULUS ($\times 10^6$ psi)	MEASURED STRAIN (mils per inch)	TANGENT MODULUS ($\times 10^6$ psi)	MEASURED STRAIN (mils per inch)	TANGENT MODULUS ($\times 10^6$ psi)	MEASURED STRAIN (mils per inch)	TANGENT MODULUS ($\times 10^6$ psi)	MEASURED STRAIN (mils per inch)	TANGENT MODULUS ($\times 10^6$ psi)
0	0.00	---	0.00	---	0.00	---	0.00	---	0.00	---
1000	0.51	1.96	0.53	1.89	0.55	1.82	0.45	2.22	0.48	2.08
2000	1.02	1.96	1.12	1.70	1.13	1.72	0.99	1.85	0.91	2.33
3000	1.55	1.89	1.68	1.79	1.66	1.88	1.50	1.96	1.39	2.08
4000	2.17	1.61	2.33	1.53	2.28	1.61	2.08	1.72	1.87	2.08
5000	2.78	1.64	3.03	1.43	2.86	1.72	2.65	1.76	2.34	2.13
6000	3.45	1.50	3.73	1.43	3.47	1.64	3.24	1.70	2.84	2.00
7000	4.10	1.54	4.42	1.45	4.00	1.89	3.79	1.82	3.41	1.75
8000	4.81	1.41	5.14	1.39	4.58	1.72	4.34	1.82	3.97	1.79
9000	5.51	1.43	5.92	1.28	5.16	1.72	4.96	1.61	4.53	1.79
10,000	6.31	1.25	6.78	1.16	5.74	1.72	5.60	1.56	5.11	1.73
11,000	7.09	1.28	7.66	1.14	6.28	1.85	6.25	1.54	5.73	1.61
12,000	7.95	1.16	8.47	1.24	6.90	1.61	6.96	1.41	6.36	1.59
13,000	8.75	1.25	9.28	1.24	7.55	1.54	7.67	1.41	6.98	1.61
14,000	9.50	1.33	9.96	1.64	8.20	1.54	8.37	1.43	7.58	1.67
15,000			10.47	1.96	8.80	1.67	9.00	1.59	8.23	1.54
16,000			10.94	2.13	9.42	1.61	9.70	1.43	8.85	1.61
17,000			11.33	2.56			10.20	2.00	9.45	1.67
18,000									9.89	2.27
19,000									10.30	2.44
20,000									10.64	2.94
Compressive strength (psi): 14,550			17,770		16,480		18,250		21,250	
Total strain (mils per inch): 9.67			11.45		9.48		10.56		10.93	

TABLE 6
SUMMARY OF FLEXURE TEST RESULTS ON THE
ISOSTATICALLY MOLDED P-03 GRAPHITE

MATERIAL AND ORIENTATION	SPECIMEN SIZE (inches)	SPECIMEN DENSITY			FLEXURE STRENGTH VALUES (psi)
		Low (g/cc)	Average (g/cc)	High (g/cc)	
Log No. 1, Axial	0.75 x 0.75 x 5.00	1.831	1.851 (4)	1.818	5450, 5675, (5860), 6070, 6250, 5650, 6080, 6120, (6182), 6385, 6430, 6430 6050, 6065, 6250, (6526), 6580, 6665, 6775, 7300
		1.821	1.824 (6)	1.826	
		1.831	1.834 (7)	1.838	
Log No. 1, Radial	0.75 x 0.75 x 5.00	1.818	1.819 (2)	1.819	6060, (6310), 6560 5385, 5920, (6205), 6420, 6490, 6810 6400, 6480, (6726), 6790, 6820, 7140
		1.821	1.824 (5)	1.828	
		1.831	1.832 (5)	1.834	
Log No. 3, Axial	0.44 x 0.44 x 3.50	1.839	1.842 (6)	1.846	6445, 6520, (7190), 7270, 7700, 8015
		---	1.851	---	
Sub-scale	0.44 x 0.44 x 3.50	1.839	1.845 (6)	1.853	5760, 6080, 6250, (6265), 6450, 6460, 6590

FIGURE 1

APPEARANCE OF THE ISOSTATICALLY MOLDED P-03 LOGS. THE BOTTOM LOG (NO. 2) HAS INCURRED CONSIDERABLE DAMAGE IN BAKING, WHILE THE TOP LOG (NO. 1) WAS RELATIVELY SOUND.

(100 x)

(400 x)

0 0.2 0.5 1.0
MILLIMETERS

0 0.1 0.2
MILLIMETERS

FIGURE 2 MICROSTRUCTURE OF THE ISOSTATICALLY MOLDED P-03 GRAPHITE, SHOWING
TYPICAL VIEW OF COKE GRAINS (LIGHT PHASE) AND PORES (DARK PHASE)
UNDER BRIGHT-FIELD ILLUMINATION.

FIGURE 3 THERMAL EXPANSION OF ISOSTATICALLY MOLDED P-03 GRAPHITE, COMPARING THE RESULTS FROM LOG NO. 1 WITH THOSE OBTAINED PREVIOUSLY ON SMALL DEVELOPMENTAL STOCK. THE INSERT SHOWS THAT THERMAL EXPANSION AT HIGH TEMPERATURES IS AFFECTED LESS BY SPECIMEN ORIENTATION THAN BY ITS DENSITY

SUMMARY OF ELECTRICAL RESISTIVITY DATE FOR ISOSTATIC P-03 GRAPHITE,
PLOTTED AS A FUNCTION OF SPECIMEN DENSITY

THERMAL CONDUCTIVITY OF THE ISOSTATICALLY MOLDED P-03, TOGETHER WITH COMPARISON DATA FOR THE CONVENTIONALLY FABRICATED GRAPHITES

FIGURE 5

A) TYPE 609B073

B) TYPE L-2

605884A

GRAPHITE TENSILE SPECIMENS

STRESS VS STRAIN CURVE FOR ISOSTATICALLY MOLDED P-03 GRAPHITE IN TENSION, AVERAGED FOR SIX SPECIMENS EACH FROM THE AXIAL AND RADIAL ORIENTATIONS. EXTREME VALUES FOR THE ENTIRE GROUP ARE ALSO PRESENTED TO DEMONSTRATE THE RANGE OF VARIATION.

FIGURE 7

TANGENT YOUNG'S MODULUS AS A FUNCTION OF APPLIED STRESS IN TENSION

STRESS VS STRAIN RELATIONSHIP IN TENSION AS A FUNCTION OF P-03 GRAPHITE'S STRENGTH. THE WEAKEST SPECIMEN HAS A CONSISTENTLY HIGHEST DEFORMATION AT ALL STRESS LEVELS.

TENSILE STRENGTH OF ISOSTATICALLY MOLDED P-03 GRAPHITE
AS A FUNCTION OF TEMPERATURE, AS DETERMINED ON AXIAL
SPECIMENS

FIGURE 10

PERMANENT ELONGATION OF THE THREAD-LOADED L-2 SPECIMENS
WHEN PULLED TO FRACTURE AT A UNIFORM STRAIN RATE.

ELONGATION (NON-ELASTIC COMPONENT ONLY) OF THREAD-LOADED TENSILE SPECIMEN AT HIGH TEMPERATURES WHEN TESTED IN HELIUM ATMOSPHERE. VALUES OF SPECIMEN ELONGATION DETERMINED FROM THE LOAD - VS - DEFLECTION CHART WERE CORRECTED TO COINCIDE WITH MICROMETER ELONGATION MEASUREMENTS.

FIGURE 12

COMPRESSIVE STRENGTH OF ISOSTATICALLY MOLDED P-03 GRAPHITE AT ROOM TEMPERATURE

STRESS VS STRAIN FOR THE P-03 GRAPHITE IN COMPRESSION, AS DETERMINED AT ROOM TEMPERATURE

VARIATION OF THE TANGENT YOUNG'S MODULUS WITH APPLIED STRESS IN THE ROOM-TEMPERATURE COMPRESSION TESTS OF P-03 GRAPHITE

605869A

STRESS VS STRAIN CURVE FOR P-03 IN COMPRESSION, SHOWING THE INFLUENCE OF PRELOADING. THE INSERT INDICATES THE MAGNITUDE OF PERMANENT SET.

FIGURE 16

FIGURE 17

CEMENTED TENSILE SPECIMEN

605868A

FIGURE 18

COMPARATIVE TENSILE STRENGTH OF CEMENTED P-03 GRAPHITE WITH IN-PROCESS VS. P-514 JOINT

605872A

FIGURE 19

605873A

FIGURE 20

605874A

FIGURE 21

P-514 JOINT

FIGURE 22 PHOTOMICROGRAPH OF A
GRAPHITIZED P-514 JOINT

"IN-PROCESS" JOINT

(50X)

FIGURE 23 PHOTOMICROGRAPH OF A
GRAPHITIZED "IN-PROCESS" JOINT

HYDROGEN PERMEABILITY OF UNIMPREGNATED, SILICA-IMPREGNATED, AND CEMENTED P-03 WITH IN-PROCESS OR P-514 JOINTS

APPEARANCE OF P-03 STAVES BEFORE CEMENTING

FIGURE 25

CEMENTED P-03 STAVED CYLINDER IN THE AS-CURED STAGE

FIGURE 26

APPEARANCE OF CEMENTED P-03 CYLINDER AFTER MACHINING

FIGURE 27

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6. Ref. 1, Appendix III. Grade P-03 is a fine-grained premium graphite manufactured by the Pure Carbon Company, St. Mary's, Pennsylvania.
7. The Industrial Graphite Engineering Handbook, Union Carbide Corporation, Carbon Products Division, 1964
8. Personal communication to V. Kachur from Eugene M. Woodruff of Battelle/Northwest, dated April 28, 1965. Thermal conductivity was calculated by D. E. Baker from the measurements of thermal diffusivity by G. E. Shook.
9. C. E. Moeller and D. R. Wilson, "Thermal Conductivities of Several Metals and Non-Metals From 200 to 1300°C by the Radial Heat Flow Technique", Proceedings, Third Conference on Thermal Conductivity, Volume 1, pp. 224-251, 1963.
10. Necessary facilities required for isostatically molding the scaled-up P-03 logs were provided Refractomet Division of the Universal-Cyclops Steel Corporation, Bridgeville, Pennsylvania. This equipment was limited to the maximum chamber diameter of 12 inches.
11. D. E. Baker, "Thermal Conductivity of Irradiated Graphite by a Rapid Thermal-Pulse Method", Report No. HW-SA-2874, Hanford Laboratories (now Battelle/Northwest), 1963.
12. Thermal conductivity data on the irradiated stock were provided by J. A. Begley.
13. W. Pollack, Evaluation of NRX-A2 Type Inner Reflector Properties, WANL-TME-765, (Confidential-Restricted Data), May, 1964.
14. H. C. Nicholson and L. L. France, Joining of Graphite Fuel Elements for the NRX-B Advanced Design, WANL-TME-847, (Confidential-Restricted Data), July, 1964.