Feasibility of Monitoring the Strength of HTGR Core Support Graphite--Part III

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SUMMARY

Methods are being developed to monitor, in-situ, the strength changes of graphite core-support components in a High-Temperature Gas-Cooled Reactor (HTGR). The results reported herein pertain to the development of techniques for monitoring the core-support blocks; the PGX graphite used in these studies is the grade used for the core-support blocks of the Fort St. Vrain HTGR, and is coarser-grained than the grades used in our previous investigations. The through-transmission ultrasonic velocity technique, developed for monitoring strength of the core-support posts, is not suitable for use on the core-support blocks. Eddy-current and ultrasonic backscattering techniques have been shown to be capable of measuring the density-depth profile in oxidized PGX and, combined with a correlation of strength versus density, could yield an estimate of the strength-depth profile of in-service HTGR core support blocks. Correlations of strength versus density and other properties, and progress on the development of the eddy-current and ultrasonic backscattering techniques are reported herein.

PREFACE

A review (BNWL-B-359), funded by the U.S. Atomic Energy Commission, established the theoretical feasibility of non-destructively monitoring the strength, in situ, of the core-support components of a High Temperature Gas-Cooled Reactor (HTGR). This document is the third of a series reporting the experimental verification of feasibility and refinement of the techniques for in-service monitoring. The work reported in Part I (BNWL-2239), sponsored by the U.S. Energy Research and Development Administration, established a direct correlation between the compressive strength of oxidized graphite cylinders and the velocity of ultrasonic waves transmitted diametrically through the cylinders; for this geometry, with this grade of graphite, the correlation is practically independent of the oxidation-depth profile. The work reported in Part II (NUREG/CR-0995; PNL-3126), sponsored by the U.S. Nuclear Regulatory Commission, extended the investigation to other grades of graphite, prototypic of those which might be used for the core-support posts in an advanced HTGR; it also established the need for obtaining density-depth profiles for components of non-cylindrical geometry, and for some grades of graphite even if the geometry is cylindrical. This report (Part II) extends the investigation to non-destructive techniques for measuring the density-depth profile in a graphite grade, prototypic of HTGR core-support blocks.

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LIST OF SYMBOLS AND UNITS

- E = Elastic Moduli (E_e = Young's Modulus, E_s = Shear Modulus) in gigapascals ($1 \text{ GP}_a = 1.4504 \times 10^5 \text{ psi}$).
- ρ = Density (ρ_0 = unoxidized density) in megagrams per cubic meter ($1 \text{ Mg/m}^3 = 1 \text{ gm/cm}^3$).
- S = Compressive Strength (S_0 = unoxidized strength) in megapascals ($1 \text{ MP}_a = 145.04 \text{ psi}$).
- σ = Poisson's ratio.
- V = Ultrasonic velocity (V_ℓ = velocity of the longitudinal wave, V_s = velocity of the shear wave) in meters per second ($1 \text{ m/s} = 3.2808 \text{ ft/sec}$).

INTRODUCTION

The underlying principles and basic design of the High-Temperature Gas-Cooled Reactor (HTGR) have been discussed by Fortescue et al. (1960). Walker and Johnson (1969) and Dahlberg et al. (1969) give a more detailed description of the Fort St. Vrain HTGR (FSVR). The FSVR core is composed of vertical stacks of hexagonal graphite blocks, which serve both as neutron moderator and as containment for the fuel. The columns of fueled blocks rest on graphite core support blocks. The support blocks are, in turn, supported on graphite posts. The helium exits from the core through channels in the core support blocks and circulates between the core support posts on its way to the heat exchangers.

Provision is made in the design to continuously purify a fraction of the circulating coolant, so as to remove any oxidizing impurity gases. Such gases can be introduced during normal refueling or maintenance operations from an ingress of air or water and from other sources. During normal operations the concentrations of the oxidizing impurity gases, primarily water vapor and carbon dioxide, are maintained at a very low (a few parts per million) concentration; however, certain abnormal conditions, such as failure of a heat-exchanger tube, can introduce much larger concentrations of reactive gases.

Data on the chemical impurities in the coolant gas during the start-up of the FSVR have been reported by Montgomery and Burnette (1976, 1977), Montgomery and Baldwin (1977), and Baldwin and Wolfer (1978). These data and similar data from other graphite-moderated gas-cooled High-Temperature Reactors (HTR), such as Dragon and Peach Bottom, have shown that the gas compositions cannot be accurately predicted (Carlyle and Kinsey 1969; Chapman 1970; Burnette et al. 1971). More importantly, however, the effects of the oxidizing gases on the core support structure cannot be accurately predicted from current knowledge.

It is well known that the strength of graphite is rapidly reduced as a result of oxidation. It is also well known that this reduction in strength can be correlated with changes in density and changes in the elastic moduli. In previous investigations, Morgan and Becker (1977, 1979) have shown that the changes in strength can be monitored by measuring the velocity at which an ultrasonic wave travels through the graphite. From theoretical considerations (Morgan and Becker 1974), one can expect the strength (S) to be related to the ultrasonic velocity (V) by an equation of the form:

$$S \sim KV^\alpha \quad (1)$$

where K is nearly constant over a wide range of densities, and α equals approximately 2.0. Morgan and Becker (1979) have tested the relationship shown in Equation 1 by oxidizing samples of four grades of fine-grained graphites, typical of materials which might be used for HTGR core support posts, in a mixture of CO_2 and CO . A good correlation was obtained for each of the graphites,

including Union Carbide^(a) grade ATJ which was used for the core support posts in FSVR.^(b) However, the results also indicated that for most applications one would need to measure the oxidation profile as a function of depth into the graphite.

The work reported herein extends the investigation to Union Carbide grade PGX, which was used for the core support blocks in FSVR,^(b) and to the investigation of other nondestructive testing techniques for obtaining the oxidation profile.

CONCLUSIONS

- Both the complicated geometry of the HTGR core-support blocks and the rapid attenuation of ultrasonic signals in oxidized PGX graphite severely limit the usefulness of ultrasonic velocity techniques for in-service monitoring the core-support blocks.
- A very good correlation between compressive strength and density was obtained for oxidized PGX graphite, similar to the correlations obtained previously with the finer-grained graphites.
- Eddy-current techniques can be used to obtain the density profile near the surface of the blocks and, if the oxidation profile is a continuous analytical function of depth from the surface, this density profile could be extrapolated to obtain a measure of the strength-depth profile in the blocks.
- Under some conditions, the near-surface profile is not indicative of the profile at greater depth. In fact, under some conditions, the oxidation rates at some distance into the block can exceed the rate near the surface.
- Ultrasonic backscattering techniques can be used to obtain the density profile at greater depth and, thus, provide a more reliable estimate of the strength-depth profile.
- Work is continuing to develop both the eddy-current and ultrasonic backscattering techniques to the state where they can be applied to in-service monitoring of the strength changes of HTGR core-support blocks.

(a) Union Carbide Corporation, New York, NY 10017.

(b) "Fort St. Vrain Nuclear Generating Station", Public Service of Colorado, Docket 50267.

GRAPHITE CHARACTERIZATION METHODS

Four nondestructive evaluation methods have been, or are currently being, evaluated for their ability to assess graphite oxidation. These are ultrasonic velocity of both the shear- and longitudinal-waves, ultrasonic surface-wave velocities, ultrasonic backscatter analysis, and electrical conductance from eddy-current measurements. A description of each method follows.

THROUGH-TRANSMISSION ULTRASONIC VELOCITIES

All work on through transmission ultrasonic velocity tests was conducted using apparatus described in an earlier report (Morgan and Becker 1979). This device employed several transmitter-receiver pairs to measure the velocity of sound passing through the graphite cylinders as shown in Figures 1 and 2. Good correlation of sonic velocity with bulk oxidation has been achieved in various grades of graphite. However, emphasis on use of this technique has decreased for two major reasons:

1. Locating a pair of transmitter-receiver sensors at meaningful positions in the Ft. St. Vrain Core Support Block and accurately gaging the physical distance between them for velocity measurements is not a practical option. These limitations are due to the support block geometry and the capability of the fuel handling machine.

FIGURE 1. Ultrasonic Velocity of the Longitudinal Wave
(T = transmitter, R = receiver)

FIGURE 2. Ultrasonic Velocity of the Shear Wave
(T = transmitter, R = receiver)

2. Ultrasonic coupling between sensors and the graphite poses a technical problem of some difficulty for field applications. If a very thin plastic couplc member such as those used for lab work is employed, a substantial force must be exerted on the sensor. This poses a risk of fracturing oxidized or reduced strength graphite as evidenced by our laboratory work. A second coupling option is the use of RTV® or silicone elastomer products. Low coupling forces may be used but the velocity measurements can be subject to large error due to the flexibility of these products. The length of the coupling path will vary as a function of the applied force, resulting in the introduction of error to the velocity measurements.

ULTRASONIC SURFACE WAVE VELOCITIES

An evaluation of surface or Raleigh wave testing was conducted to determine the applicability for profiling near-surface oxidation. The intent was to use several test frequencies to obtain velocity data from different depths of the material. Our findings are as follows:

1. Surface wave velocity is dependent on the degree of oxidation.
2. Graphite oxidized beyond mild weight loss levels (1 to 3%) will not sustain surface wave propagation.

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These findings lead to the conclusion that, although the technique is useful at very low oxidation levels, surface wave testing is not a viable technique for quantizing the degree of graphite oxidation to be expected in an HTGR.

The apparatus used for this evaluation is shown in Figure 3. A large piezoelectric transmitting transducer is angulated to the shear critical angle and is excited with a tone burst. Test frequencies ranging from 100 to 800 kHz were used for the evaluation. Ultrasonic coupling was provided by coupling members fabricated from RTV 118® silicone. A pair of broadband receiving transducers were placed into the surface wave beam at a one-inch separation, and the difference in arrival time of the surface energy was used to calculate surface wave velocity.

FIGURE 3. Ultrasonic Surface Wave Apparatus

ULTRASONIC BACKSCATTER ANALYSIS

Work has been initiated on the application of ultrasonic backscatter methods to the characterization of graphite. Backscatter testing (Goebbels 1980) has been used with high success in other materials for: 1) distinguishing small ultrasonic reflectors from grain boundary scattering noise, 2) measuring grain size, and 3) detecting regions of inhomogeneities such as porosity, inclusions and microcracks. The technique draws its success from statistical separation of desired signals from noise in the received pulse-echo waveform. This is performed by spatial waveform averaging, i.e., summation of waveforms obtained after making small increments in sensor position. Signal averaging with changes in drive frequency or sensor angle also produce good results.

Present indications are that the technique shows promise for detecting and/or quantizing both near-surface and deep oxidation. Work has been completed on determining a suitable couplant for in-service inspection. The ultrasonic properties of a variety of silicone elastomers and rubbers were evaluated and two high-temperature products were selected for future use. These are General Electric RTV31[®] silicone rubber and Dow Corning 3120[®] silicone rubber. The ultrasonic properties we measured in these products are listed in Table 1.

TABLE 1. Properties of Silicone Rubbers Selected for In-Service Ultrasonic Couplants

<u>Product</u>	<u>Attenuation at 2.25 MHz, dB/mm</u>	<u>Ultrasonic Velocity, m/s</u>
RTV31	2.25	910
3120	1.78	910

Current work is centered upon preparing to correlate backscatter data with actual oxidation in graphite. Waveforms will be digitized and averaged through the use of a Biomation transient digitizer and a DEC 1103 minicomputer system.

EDDY-CURRENT MEASUREMENTS

A strong correlation has been established between eddy-current data and graphite density and/or oxidation and substantial work has been conducted to optimize methods for profiling near-surface oxidation. The electrical conductivity of the graphite is detected by the eddy-current method and used as a measure of oxidation. The depth of penetration can be controlled by selection of test frequency, permitting profile or oxidation versus depth, to be determined through use of several test frequencies. The technique should be useful up to a depth of 3.8 to 5.0 mm, depending on the size of the search coil used.

The present status of this work is that a computer algorithm is under development for estimating the oxidation profile as a function of eddy-current data.

Illustrations of one of the probes used for obtaining laboratory data are shown in Figures 4 and 5. This probe consists of 15 turns of wire on each end of a 19-mm diameter nylon rod. One coil is used for sensing graphite properties and the other is used as a reference coil for temperature compensation. The probe was interfaced to a conventional impedance-plane eddy-current instrument. This instrument generates outputs corresponding to resistance and reactance parameters of the search coil. These parameters are in part a function of the electromagnetic loading of the coil caused by induced eddy currents in the graphite. Consequently, the parameters may be analyzed to interpret electromagnetic properties of the graphite, e.g., its electrical conductivity.

A variety of measurements have been acquired using thin plates of graphite which have been oxidized to various degrees. This permits various oxidation profiles to be simulated by the plate stacking order. Test frequencies of 4 MHz, 1 MHz and 100 kHz were selected for the data acquisition

FIGURE 4. Eddy-Current Probe Used for Measuring Graphite Properties

FIGURE 5. Close-Up of the Eddy-Current Probe Shown in Figure 4

because of their resultant penetration depths. The 4-MHz test is essentially a surface test except for very low conductivity graphites, while the other frequencies penetrate to correspondingly deeper levels in the graphite.

Our results have shown that a high degree of sophistication may be required to develop an algorithm which can estimate oxidation profiles. Analog computer methods which have been widely used for other multifrequency data combinations do not appear applicable to this problem. As a result, we have started development of an algorithm using large scale computing facilities. Programs called EDDY and ZFIT have been implemented on the Brookhaven CDC-7600 computer. EDDY calculates resistance and reactance of a coil located above a conducting plane when coil geometry, driving frequency, and layer conductivities, thicknesses, and permeabilities are specified. Program EDDY is based on theory and codes developed by C. V. Dodd et al. (1973) at Oak Ridge National Laboratory. The program has been embedded in a parameter fitting routine MINUIT, available on the Brookhaven computer. The resulting program ZFIT searches for values of a specified list of graphite parameters to fit a set of impedance measurements. This corresponds to a form of inversion of the EDDY program in which resistance and reactance at several frequencies are used to estimate material properties versus depth. The fitting program makes possible the inversion which would be impossible or very difficult by other approaches.

Further work will be conducted on the algorithm to verify that it is a viable approach for estimating near surface oxidation profiles, and to determine the simplest form of the algorithm which could be implemented on portable field equipment.

PREPARATION OF SAMPLES

Union Carbide^(a) grade PGX is a molded graphite, with medium grain size, available in large diameter logs. We acquired about one-eighth of a 1.14-m (45-in.)-diameter by 1.83-m (8-ft)-long log from General Atomic.^(b)

As shown in Appendix A, we machined samples having three different geometries for use with the four techniques. These sample geometries are:

- Large cylinders, 76 mm (3 in.) in diameter, used to obtain data for direct comparison with our previous data (on strength, density, longitudinal velocity, and compressive strength, both before and after oxidation) for 4 other grades of graphite. Cylinders which were used to obtain the unoxidized properties were 152 mm (6 in.) long. Samples to be oxidized were 203 mm (8 in.) long, but were reduced to 152 mm long after oxidation and prior to measurement of their properties.
- Large blocks, 115 x 165 x 190 mm (4.5 x 6.5 x 7.5 in.), used for testing the ultrasonic surface wave, ultrasonic back scattering, and eddy-current techniques. The blocks were given a plasma sprayed coating of ZrO₂ prior to oxidation; this coating retarded, but did not eliminate oxidation of the underlining graphite.
- Thin plates, 1.25 mm (0.050 in.) thick and 76 mm (3 in.) square, were oxidized to various densities and used to simulate oxidation gradients for development of the eddy-current techniques.

RESULTS

The principle results obtained during this investigation are discussed below under the primary heading which designates the identifies the sample geometry. Additional information on the oxidation rate of PGX is presented in Appendix B.

LARGE CYLINDERS

In our previous investigations, we concentrated on the development of techniques that would be suitable for monitoring the strength changes of the core support posts. The graphite grades that we examined have finer grain size and higher strength than PGX. Thus, it was important to determine if the techniques that we had developed were applicable to PGX, and to provide a basis for comparison. The majority of the previous work had been performed on 76-mm (3-in.)-diameter by 152-mm (6-in.)-long cylinders, before or after oxidation

(a) Union Carbide Corporation, New York, NY 10017.

(b) General Atomic Company, San Diego, CA 92138.

at 875°C in a mixture of 80% CO₂, 20% CO; therefore, we chose to use the above geometry and oxidation conditions for one series of tests on PGX.

In our previous investigations, we established that there is a very high correlation between the compressive strength and certain properties. The general equation for this correlation can be written as:

$$S = S_0 (P/P_0)^\alpha \quad (2)$$

where S = the measured compressive strength of a sample.

S₀ = the average strength of unoxidized samples.

P = the measured value of the property^(a) for the sample.

P₀ = the average value of the property as measured on unoxidized samples, and

α = a constant which varies with the grade of graphite and the specific property.

Table 2 lists the properties measured on unoxidized samples of PGX graphite and Table 3 shows the properties measured on other samples of PGX prior to their oxidation at 875°C in a mixture of 80% CO₂ and 20% CO. Table 4 lists the same properties, plus their compressive strength, measured after various amounts of oxidation. Table 5 shows the results of Rank Correlation Tests for all 21, oxidized and unoxidized samples. The differences in the absolute values of the correlation parameter, R, can be disregarded, because in all cases the probability of a correlation far exceeds 99%.

It is interesting to note that neither unoxidized sample BA-7 or oxidized sample BA-1, which had unusually low densities (and, thus low values for their other properties), do not present anomalies in either the Rank Correlation Tests or in the Least Squares fit of the data to Equation 2. Table 6 shows the calculated unoxidized compressive strength, and the correlation coefficients obtained by least squares analysis of the data, as per Equation 2.

We had assumed (and to the best of our knowledge all previous investigators have assumed) that the oxidation rate would decrease as a continuous analytic function of distance from the surface.^(b) However, visual

(a) The best correlations have been obtained for average sample density, and for the diametral ultrasonic velocities; however, very good correlations have been established for the axial ultrasonic velocities, and for the elastic and shear moduli, measured both in the diametral and axial directions.

(b) This assumption seems logical based on the fact that the concentration of the oxidant will decrease and the concentration of the oxidation products will increase with increasing penetration depth.

TABLE 2. Property Measurements on Unoxidized PGX Cylinders

Sample No.	Strength (MPa)	Density (Mg/m ³)	Diametral Measurements		Axial Measurements	
			V_L (km/s)	V_S (km/s)	V_L (km/s)	V_S (km/s)
AC-3	39.20	1.749	2.096	1.392	1.909	1.345
AC-4	38.94	1.748	2.092	1.387	1.901	1.337
AB-11	38.87	1.751	2.103	1.395	1.912	1.345
BA-6	38.65	1.754	2.126	1.406	1.910	1.352
AC-6	38.55	1.749	2.108	1.397	1.915	1.345
BA-17	38.35	1.752	2.110	1.402	1.893	1.342
BA-4	37.99	1.746	2.101	1.383	1.896	1.345
AC-5	37.75	1.747	2.088	1.384	1.904	1.336
AC-2	36.78	1.747	2.106	1.393	1.909	1.340
BA-15	36.09	1.746	2.104	1.398	1.886	1.342
BA-2	35.88	1.733	2.081	1.384	1.890	1.335
BA-7	29.10	1.690	1.972	1.311	1.763	1.265
Average:	37.18	1.743	2.091	1.386	1.891	1.336
$\pm 1\sigma$:	2.78	0.013	0.039	0.025	0.041	0.023
AB-22	34.14	1.750	2.023	1.377	2.067	1.412

NOTE: Sample AB-22 was cut with its long axis transverse to the long axis of the other samples; thus, neither the diametral no axial velocities are directly comparable with the values measured on the other samples.

examination of sectioned samples of oxidized PGX indicates that there is an area of increased porosity below the surface. Physical measurements of density confirm the visual indications that there was an increase in oxidation rate with penetration depth. Figure 6 shows a optical view of a 25-mm thick disk, cut from the end of an oxidized 76-mm diameter sample; and Figure 7 shows the density profile obtained by physical measurements on another PGX sample.

A review of the literature indicates that this inverse oxidation phenomenon has been observed by Walker et al. (1968)^(a) at Pennsylvania State

(a) See especially p. 366 of the reference.

TABLE 3. Pre-Oxidation Measurements on PGX Cylinders

Sample No.	Density (Mg/m ³)	Diametral Velocities		Axial Velocities	
		V_{ℓ} (km/s)	V_s (km/s)	V_{ℓ} (km/s)	V_s (km/s)
BA-3	1.744	2.089	1.390	1.905	1.346
BA-5	1.745	2.097	1.392	1.902	1.348
BA-16	1.746	2.104	1.396	1.901	1.348
BA-10	1.747	2.095	1.392	1.902	1.348
BA-11	1.747	2.105	1.397	1.900	1.347
BA-1	1.684	1.950	1.303	1.768	1.255
BA-8	1.739	2.081	1.387	1.901	1.345
BA-12	1.754	2.117	1.409	1.920	1.357
BA-14	1.741	2.092	1.391	1.890	1.342
Average:	1.739	2.081	1.384	1.888	1.337
$\pm 1\sigma$:	0.021	0.050	0.031	0.046	0.031

TABLE 4. Property Measurements on Oxidized PGX Cylinders

Sample No.	Strength (MPa)	Density (Mg/m ³)	Diametral Measurements		Axial Measurements	
			V_{ℓ} (km/s)	V_s (km/s)	V_{ℓ} (km/s)	V_s (km/s)
BA-3	31.67	1.709	1.933	1.297	1.762	1.250
BA-5	29.33	1.676	1.869	1.250	1.690	1.202
BA-16	23.02	1.642	1.851	1.235	1.665	1.251
BA-10	22.96	1.568	1.360	1.025	1.631	1.089
BA-11	21.29	1.581	1.390	1.041	1.668	1.110
BA-1	18.51	1.561	1.579	1.079	1.329	1.054
BA-8	18.44	1.544	1.243	1.007	1.628	1.094
BA-12	17.48	1.512	1.387	1.031	1.594	1.069
BA-14	16.11	1.511	1.305	0.958	1.547	1.032
Average:	22.09	1.589	1.546	1.103	1.613	1.128

TABLE 5. Rank Correlation Tests on PGX Cylinders

Sample No.	Strength	Density	Δ^2	Diametral Measurements				Axial Measurements			
				V_L	Δ^2	V_S	Δ^2	V_L	Δ^2	V_S	Δ^2
AC-3	1	4.5	12.25	8	49	7	36	4.5	12.25	3.5	6.25
AC-4	2	6	16	9	49	8	36	7	25	9	49
AB-11	3	3	0	6	9	5	4	2	1	3.5	0.25
BA-6	4	1	9	1	9	1	9	3	1	1	9
AC-6	5	4.5	0.25	3	4	4	1	1	16	3.5	2.25
BA-17	6	2	16	2	16	2	4	9	9	6.5	0.25
BA-4	7	9.5	6.25	7	0	11	16	8	1	3.5	12.25
AC-5	8	7.5	0.25	10	4	9.5	2.25	6	1	10	4
AC-2	9	7.5	6.25	4	25	6	9	4.5	20.25	8	1
BA-15	10	9.5	0.25	5	25	3	49	11	1	6.5	12.25
BA-2	11	11	0	11	0	9.5	2.25	10	1	11	0
BA-3 ^(a)	12	12	0	13	1	13	1	13	1	14	4
BA-5 ^(a)	13	14	1	14	1	14	1	14	1	15	4
BA-7	14	13	1	12	4	12	4	12	4	12	4
BA-16 ^(a)	15	15	0	15	0	15	0	16	1	13	4
BA-10 ^(a)	16	17	1	19	9	19	9	17	1	18	4
BA-11 ^(a)	17	16	1	17	0	17	0	15	4	16	1
BA-1 ^(a)	18	18	0	16	4	16	4	21	9	20	4
BA-8 ^(a)	19	19	0	21	4	20	1	18	1	17	4
BA-12 ^(a)	20	20	0	18	4	18	4	19	1	19	1
BA-14 ^(a)	21	21	0	20	1	21	0	20	1	21	0
$\Sigma \Delta^2$:			70.5		220		192.5		112.5		126.5
$R = 1 - \frac{6\Sigma \Delta^2}{n(n^2 - 1)}$:			0.954		0.857		0.875		0.927		0.918

(a) Measurements after oxidation.

TABLE 6. Calculated Compressive Strengths(a)

<u>Property</u>	<u>Calculated S_o MPa</u>	<u>α</u>
Density	36.9 ± 1.3	5.66
<u>Diametral Measurements</u>		
Longitudinal velocity	36.2 ± 2.8	1.54
Shear-wave velocity	36.7 ± 2.1	2.21
Elastic modulus	35.9 ± 3.0	0.62
Shear modulus	36.8 ± 2.0	0.93
<u>Axial Measurements</u>		
Longitudinal velocity	36.1 ± 2.7	2.94
Shear-wave velocity	36.9 ± 2.2	3.08
Elastic modulus	36.0 ± 2.6	1.04
Shear modulus	36.0 ± 1.9	1.22

(a) Measured compressive strength is 37.2 ± 2.8 MPa (see Table 2).

University and by others at Oak Ridge National Laboratory (ORNL).(a) Moreover this phenomenon is not isolated to PGX graphite, nor is it isolated to samples oxidized in a mixture of CO_2 and CO . Because of the potential significance of this phenomenon to the oxidation behavior of graphite in an operating HTGR, we have performed additional work to investigate this phenomenon.

Disks (76 mm in dia, 25 mm thick), which had been cut from the ends of oxidized cylinders (Morgan and Becker 1979), were ashed in air at $700^\circ C$. Table 7 shows the elemental composition of the ash; the analysis was made by X-ray Photoelectron Spectroscopy; Morgan and Thomas (1981) have published details of the analysis and visual observations of the ash. It was not possible to determine which of the impurity elements are responsible for the inverse oxidation phenomenon; however, the catalytic activity of many impurities is known (McKee 1980) to be strongly dependent on the chemical state of the impurity element.

It seems most likely that the increased oxidation rate in the interior of these samples resulted from a change in oxidation state of one or more of the impurity elements as a result of the change in the CO/CO_2 ratio with distance from the surface (Morgan and Thomas 1982). The possibility that this phenomenon might occur in the reactor core-support components cannot be ruled out at the

(a) Conversations with W. P. Eatherly (ORNL).

PGX, SA. NO. VII-BA-12

$$\rho_0 = 1.754$$

$$\rho = 1.512$$

$$\Delta\rho = 13.8\%$$

FIGURE 6. Sectioned Sample of Oxidized PGX Graphite

present time. Thus, it will be necessary to obtain oxidation depth profiles rather than merely extrapolating the near-surface oxidation profiles.

FIGURE 7. Density Versus Oxidation Depth for a 3.0-in. Diameter Sample

TABLE 7. Elemental Composition of Graphite Ash

Sample No:	II-D-3	II-F-3	III-A-12	III-A-10	V-A-7	VI-A-4	VII-BA-3
Graphite Grade: (a)	2020	2020-P	S1090	S1090-P	ATJ	P3W	PGX
Total Ash, ppm:	970	32	1030	17	880	2410	3820
Element	Concentration, atomic percent						
Si	18.5	5.5	9.4	3.9	13.3	33.5	1.1
Ca	18.8	1.9	15.4	1.9	23.6	3.8	6.6
Ba	1.0	--	0.4	--	1.5	0.2	0.5
Fe	--	0.6	--	1.5	--	2.7	3.0
Sn	0.1	--	0.1	--	0.1	0.1	0.1
P	2.8	--	1.0	--	2.4	0.8	0.6
K	1.3	3.0	0.8	--	0.8	0.9	2.4
Al	1.3	--	--	--	--	--	2.3
S	0.8	--	14.6	--	0.8	--	5.7
Ti	0.3	--	--	--	--	--	0.9
Na	--	0.3	--	0.3	--	--	1.2
Ni	--	1.8	--	3.3	--	--	--
V	--	11.6	--	8.1	--	--	--
Cl	--	1.0	--	2.0	--	--	--
Pb	--	0.1	--	0.1	--	--	--
O	51.5	45.1	55.8	42.8	51.5	53.4	49.7
C	3.5	29.2	2.4	36.1	6.0	4.5	5.4

(a) Samples which had been purified by POCO Graphite, Inc., were designated 2020-P and S1090-P.

LARGE BLOCKS

The large (115- x 165- x 190-mm) blocks were intended for use in developing the eddy-current techniques for measuring the near-surface oxidation profile; therefore, we wanted to obtain a large surface area with a fairly uniform oxidation gradient into the block. In order to increase the amount of surface area which was uniformly oxidized (i.e., to minimize the edge-effects), we plasma sprayed a thin coating of ZrO_2 on bottom surface and four sides of the blocks, leaving a 165- x 190-mm uncoated surface to be exposed to the oxidizing gasses. This coating adhered well to the graphite, but was somewhat porous, and did not completely eliminate oxidation of the underlying graphite.

In order to develop different oxidation profiles, the blocks were oxidized at different temperatures in CO₂-CO, as shown in Table 8. After oxidation, a 30-mm-thick slab was cut from one end of each block and the oxidation patterns, noted in Table 8, were observed. A second slab (115 x 165 x 15 mm) was cut from the block and a sample (15 x 15 x 115 mm) was machined from the center portion of the slab. The density profile in this small sample was obtained by incrementally reducing its length and measuring the weight loss for each increment. The resultant density profiles are shown in Figures 8 through 10. The positions from which these samples were taken were chosen such that they would be most representative of the uniformly oxidized material in the block.

TABLE 8. Data on Oxidation of PGX Blocks

Block No.	Oxidation Temperature (°C)	Weight Loss (a) (%)	Oxidation Atmosphere (% by Volume) (b)	Condition of the Cross-sectioned Surface
AA-4	875	10.5	80% CO ₂ /20% CO	Broad band of oxidation pitting, from ~10 to 40 mm below surface.
AA-1	925	10.6	80% CO ₂ /20% CO	Narrow band of pitting, from ~6 to 12 mm below surface.
AA-3	975	7.5	94% CO ₂ /6% CO	Very narrow band of pitting, from ~3 to 5 mm below surface.
AA-2	1025	5.0	94% CO ₂ /6% CO	Anomalous oxidation pattern; not sectioned (see text).
AB-3	1025	12	94% CO ₂ /6% CO	Reduced to granular powder to a depth of ~3 to 5 mm below surface; not sectioned.

(a) See Appendix B for oxidation rate.

(b) Composition of gas entering the furnace. At 875°C, about 1% of the CO₂ reacted with the graphite sample; at 925°C and 975°C about 4% reacted; at 1025°C about 11% reacted.

The surface of Block AB-3 appeared solid, except in a few patches where 27 grams of particles had sluffed off (the block was oxidized standing on end in the furnace); however, the slightest pressure would dislodge large patches of granular particles. Block AA-2 had what appeared to be a flow line running diagonally the length of the block; that is, a "pitch-rich" line which had been produced during the mixing, or the molding operation, when one part of the mixture flowed relative to an adjacent part of the mixture. This line was quite visible optically, and eddy-current measurements indicated that there was an abrupt change in density at this line. After oxidation, eddy-current measurements showed an even larger demarcation in density at the line and a

FIGURE 8. Density-Depth Profile for Block Oxidized at 875°C

FIGURE 9. Density-Depth Profile for Block Oxidized at 925°C

FIGURE 10. Density-Depth Profile for Block Oxidized at 1025°C

superimposed gradient, which was apparently caused by a gradient in catalytic impurities within the denser part of the block. The corner farthest from the flow line was very friable.

The other blocks seemed to be of fairly uniform density (as indicated by the eddy-current measurements) prior to oxidation. After oxidation, Block AA-3 had a side-to-side gradient in density with a small patch (roughly 20 to 40 mm across) of medium density toward the edge having the lowest density. Eddy-current measurements indicated that Block AA-1 and half of Block AA-4 had been quite uniformly oxidized; the other half of Block AA-4 had a decreasing density with distance from the center and, particularly, toward one corner.

The observed density profiles lend strong support to the existence of the inverse oxidation phenomenon, discussed in the previous section. The results also indicate that PGX (or, at least, this log of PGX) is a highly variable material and that large differences in oxidation rate can be expected within the FSVR core-support blocks.

THIN PLATES

The thin (1.25-mm-thick) plates were prepared to serve as density standards to aid in the development of the algorithm used to obtain density profiles from the eddy-current data. By stacking plates of different densities, one can obtain a stepwise simulation of any density profile, and one can directly determine the contribution of different layers to the overall response of the equipment. A total of 37 plates were cut from piece AB-12; 31 of these plates were oxidized in 80% CO₂/20% CO at 875°C so as to obtain series from 0.2 to over 20% oxidation.

A wide variety of oxidation profiles were simulated by stacks of these plates and eddy-current data were obtained at three frequencies, as discussed on pages 6 and 8. Actual calibration of the equipment will require preparation of thicker samples; however, use of the thin plates to simulate oxidation profiles has been a definite aid in developing the eddy-current technique and in perfecting the data processing algorithm ZFIT.

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APPENDIX A

SAMPLING SCHEMES AND PREPARATION OF SAMPLES

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SAMPLING SCHEMES AND PREPARATION OF SAMPLES

A one-eighth section of a PGX log (General Atomic Company Log No. 7637-57; Union Carbide Corp. Log No. 36K5) was obtained from General Atomic. Figure A.1 shows a perspective view of this section and shows the location of the slabs cut from this section. Figures A.2, and A.3 show the locations from which the samples were cut from the slabs. Other details of sample preparation are given in Table A.1.

FIGURE A.1. Perspective View of the Log Section

FIGURE A.2. Sampling Scheme of Section A

SIDE VIEW

END VIEW

FIGURE A.3. Sampling Scheme of Section B

TABLE A-1. Notes on Sample Preparation

Sample No.	Notes
AB-11	
AB-22	
AC-1	
AC-2	
AC-3	
AC-4	Machined into cylinders, 76 mm (3 in.) in dia by 156 mm(6 in.) long for measurements (including strength) of the unoxidized material.
AC-5	
AC-6	
BA-2	
BA-4	
BA-6	
BA-7	
BA-15	
BA-17	
BA-1	
BA-3	
BA-5	
BA-8	Machined into cylinders, 76 mm (3 in.) in dia by 203 mm (8 in.) long for pre- and post-oxidation measurements.
BA-9	
BA-10	
BA-11	
BA-12	
BA-14	
BA-16	
AA-1	Machined into 114 x 165 x 190 mm (4.5 x 6.5 x 7.5 in.) blocks; after density measurements, eight edges were machined to a 0.5 in. radius. The edges along one 165 x 190mm face was left as-machined, all other sides were sand-blasted and plasma sprayed with a coating of zirconium oxide for oxidation protection.
AA-2	
AA-3	
AA-4	
AB-3	
AB-4	
AB-12	Sliced and machined into plates 1.25 mm (0.050 in.) thick by 76 mm (3 in.) square. The surface of the plates from AB-12 is transverse to the long axis of the parent log; those from AB-21 are parallel to the long axis.
AB-21	

APPENDIX B

NOTES ON OXIDATION RATE

APPENDIX B

NOTES ON OXIDATION RATE

During the oxidation runs, it was found to be advantageous to plot the weight loss against time-at-temperature on a log-log plot, as shown Figure B.1. Once a slope was established for a given sample, the length of time required to reach a given weight loss could be easily predicted by extrapolation of the line. This correlation is based on the assumption that the equation for weight loss (ΔW) is related to oxidation time as given in Equation B.1.

$$\Delta W = K t^{\beta} \quad (B.1)$$

Where K can be thought of as a temperature-dependent reactivity term, and β accounts for the increased accessibility of the oxidant with burn-off. In our previous investigations, (Morgan and Becker 1977, 1979) differences of 2 to 3X in oxidation rates have normally been observed for 3-in. dia samples of other graphites cut from the one log and oxidized adjacent to each other in the furnace. Thus, K must be determined for each sample; this requires that ΔW be obtained at two different times (t_1 and t_2). Moreover, if the difference between t_1 and t_2 (or ΔW_1 and ΔW_2) is small, little confidence can be placed on the calculated values of β or K; therefore, we have not included three samples in Table B.1 (the largest difference $\Delta W_2 - \Delta W_1$ was 3.3%, and the largest difference $t_2 - t_1$ was 8.3%, for these samples). The values of β and K, calculated for the other 7 samples, are listed in Table B.1

The data, at 1.2 and 10.5% weight loss, from Block AA-4 yields $\beta = 1.56$ and $K = 0.047$ for the oxidation at 875°C. Using the average value of $\beta = 1.53$, the data for the blocks oxidized at the higher temperatures was extrapolated to 10% weight loss. An Arrhenius plot of the relative weight loss rate versus inverse temperature, Figure B.2, yields an activation energy of 306 kJ per mole-degree (73.2 kcal/mole-degree) for the CO_2 -C reaction for these samples.

This activation energy is considerably larger than the 229 ± 10 kJ/mole-degree reported by Eto and Growcock (1981) for samples of PGX oxidized in 2% CO_2 at slightly lower temperatures, but both activation energies are well within the range of activation energies reported by Walker et al. (1968) for graphites doped with Fe, Co, or Ni and oxidized in pure CO_2 over approximately the same temperature range which we used for the blocks.

FIGURE B.1. Weight Loss and Function of Time-at-Temperature

TABLE B.1. Calculated Rate Constants

Sample No.	Weight Loss, %			Time at Temperature, days			Rate Constant	
	ΔW_1	ΔW_2	Diff.	t_1	t_2	Diff.	β	$K^{(a)}$
BA-1	1.0	6.8	5.8	8.0	32.2	24.2	1.38	0.056
BA-11	1.2	8.7	7.5	10.0	32.2	22.2	1.69	0.025
BA-10	1.2	9.5	8.3	8.0	32.2	24.2	1.49	0.054
BA-8	1.1	10.5	9.4	8.0	35.0	27.0	1.53	0.046
BA-9 ^(a)	2.0	11.8	9.8	10.2	32.2	22.0	1.54	0.056
BA-14	1.3	12.6	11.3	10.0	42.2	32.2	1.58	0.034
BA-12	1.7	13.4	11.7	8.0	32.2	24.2	1.48	0.079
Average:							<u>1.53</u>	<u>0.050</u>

(a) Rate coefficient in percent/ $t^{1.53}$.

(b) Sample BA-9 was damaged during testing and does not appear in Tables 3 or 4.

FIGURE B.2. Arrhenius Plot for PGX Blocks

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