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

by
W. C. Morgan and F. L. Becker

March 1977

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ABSTRACT

The results reported herein establish the technical feasibility of a method for monitoring the strength of HTGR core support structures in situ. A correlation has been established between changes in the velocity of an ultrasonic pulse and changes in strength which occur during oxidation; moreover, that correlation is practically independent of oxidation profile in the posts. In addition, other correlations have been developed which allow very precise predictions of the unoxidized strength from measurements of sonic velocity and density.

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LIST OF SYMBOLS AND UNITS

E = Elastic Moduli (E_e = Young's Modulus, E_s = Shear Modulus) in gigapascals (IGPa = 1.4504×10^5 psi).

ρ = Density (ρ_o = unoxidized density) in megagrams per cubic meter ($1 \text{ Mg/m}^3 = 1 \text{ gm/cm}^3$).

S = Compressive Strength (S_o = 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 latest designs for High Temperature Gas-cooled Reactors (HTGR) call for the support of the reactor core (fuel, moderator, and burnable poison) to be provided by graphite columns. These core support posts will be exposed to a high-temperature mixture of helium and (minute quantities of) reactive impurity gases. Exact conditions of exposure and concentrations of the reactive gases are not known, cannot be measured, and may vary significantly from post to post. Thus, although a very large safety factor was provided in the design of the posts, there is some concern that the loss of strength caused by oxidation could seriously reduce this safety factor.

The authors have previously reviewed⁽¹⁾ the theoretical and experimentally-determined relationships between sonic velocity, elastic moduli, and strength for graphite samples in various conditions, including samples which had been oxidized. The Griffith^(a) equations, relating elastic moduli and strength for brittle solids, have often been applied to carbons and graphites and were assumed to apply, at least in a general way, to the high-strength and high-density graphites which might be considered for the core support posts in a large HTGR. ATJ graphite,^(b) which was used for the core support posts in the Fort Saint Vrain Reactor (FSVR), is typical of this class of graphites and Stackpole 2020,^(c) which was evidently^(d) the primary candidate for use as the core support posts for the large HTGR designs, is also typical of this class of graphites.

We showed that, from theoretical considerations, one could expect to obtain a relationship between strength (S) and ultrasonic velocity (V) of the form:

$$S \sim (E/\sigma)^\gamma \sim K \left(\frac{\rho}{\sigma}\right)^\gamma V^{2\gamma} \sim K' V^\beta \quad (1)$$

(a) Griffith modified⁽³⁾ his original equations⁽²⁾ but did not publish the derivation of the modified equations; Sih and Liebowitz⁽⁴⁾ have, however, shown that the modified form is the correct derivation.

(b) National Carbon Company.

(c) Stackpole Carbon Company.

(d) From discussions with various personnel at the General Atomic Company.

where: E is Young's or Shear Modulus
 σ is Poisson's ratio
 γ is an unknown constant
 K and K' are nearly constant
 ρ is the density, and
 β is equal or greater than 2γ .

The Griffith equations, based on a constant strain energy at fracture, lead to a theoretical value of $\gamma = 0.5$ ($\beta \geq 1.0$) for isotropic solids that are homogeneous and brittle; however, from our review of the experimental data we concluded that γ should be approximately unity ($\beta \geq 2.0$), corresponding to a constant strain at fracture, for oxidation-induced changes in polycrystalline graphites.

The results reported herein are consistent with a value of $\gamma \approx 2.0$ for unoxidized samples and $\gamma \approx 1.0$ for changes occurring during oxidation; this is discussed in more detail in Appendix A.

SELECTION AND PREPARATION OF SAMPLES

Stackpole graphite, grade 2020, was selected because it was a leading contender for use as the core support posts for the large HTGR. We, therefore, purchased two as-graphitized logs of 2020, "off-the-shelf," from Stackpole. The bar identifications and sampling schemes are shown in Figures 1, 2, and 3.

In order to verify the radial isotropy of the material, nine 1-inch diameter samples were machined from a section through the center of bar I, as shown in Figure 2. The results shown in Table 1 indicate that the strength, density, and ultrasonic velocity^(e) is quite uniform for this material. A rank correlation test also indicates, however, that a weak negative correlation may exist between strength and ultrasonic velocity as measured by longitudinal waves through the sample diameter.

Figures 2 and 3 show the sampling schemes for the 1-inch and 3-inch diameter samples. The 1-inch diameter samples, which were to be oxidized, and all 3-inch diameter samples were machined to a length of 4 inches and

e. Compressive strength and density for these and all other samples were measured in accordance with ASTM Methods C-695⁽⁵⁾ and C-559,⁽⁶⁾ respectively; the equipment and methods for measurement of ultrasonic velocity are described in the following section.

LOG I (BF 34 #41)									
A	B	C	D	E	F	G	H	J	
4"	1"	5"	9"	1-1/2"	14"	9"	14"	14-1/2"	
NDT TEST BLOCK		CHEM ANALYSIS 1" DIA COMPRESS STRENGTH	~ 3" DIA x 8"	DENSITY PROFILE	8" DIA x 12"	~ 3" DIA x 8"	8" DIA x 12"		

LOG II (89970 B-F880 #219)					
II A	II B	II C	II D	II E	
4"	1, 2, 3, 4 10"	1, 2, 3, 4 10"	1, 2, 3, 4 10"	1, 2, 3, 4 10"	
END WASTE NDT TEST BLOCK	3" DIA x 8"	3" DIA x 8"	3" DIA x 8"	3" DIA x 8"	

FIGURE 1. Sampling Schemes for Logs I and II.

FIGURE 2. Sampling Scheme for 1-inch Diameter Samples (Section I-C).

FIGURE 3. Sampling Scheme for 3-inch Diameter Samples (Sections I-G, II-B, II-C, II-D, and II-E).

TABLE 1. Radial Variation in Properties

<u>Sample No.</u> ^(a)	<u>Density</u> ^(b) (Mg/m ³)	<u>V_l</u> ^(c) (m/s)	<u>Strength</u> ^(d) (MPa)
45	1.796	2522	89.58
35	1.802	2520	85.15
25	1.799	2505	85.94
15	1.793	2538	80.80
5	1.788	2515	86.69
55	1.794	2496	86.54
65	1.800	2523	86.52
75	1.798	2520	87.14
85	1.800	2546	86.46
Ave:	1.797	2521	86.09

a. See figure 2.

b. Density in megagrams per cubic meter.

c. Velocity of the longitudinal ultrasonic pulse, measured through the diameter of these samples (in meters per second).

d. Compressive strength in megapascals.

8 inches, respectively; all samples were subsequently machined to a length of two times their diameter prior to measurement of compressive strength. Figure 4 is a photograph of the typical samples prior to oxidation.

Each sample was placed in a separate, numbered plastic bag so that they were traceable to their original position in the bar and to protect them from surface contamination, which might alter the oxidation characteristics. Property measurements for the remaining 1-inch diameter samples are shown in Table 2; Table 3 lists properties of the 3-inch diameter samples. The longitudinal, shear, and near-surface shear wave velocities were measured by the time-of-flight technique. Velocities were measured through the diameter, through the length, and axially near the surface.

ULTRASONIC VELOCITY MEASUREMENTS

EQUIPMENT

A block diagram of the system is shown in Figure 5. An ultrasonic pulser (high voltage spike output) is used to drive the transmit transducer (T). The ultrasonic wave produced, either longitudinal or shear, travels through the sample and is detected by the receiving transducer (R). The electrical signal generated by the receiver is amplified and the transit time is detected by the zero crossover detector. The zero crossover detector produces a stop pulse to the counter at the first zero crossing of the R.F. wave form after the enable gate. The enable gate is a delayed synchronized pulse which is manually adjusted by observing the monitoring oscilloscope. The enable gate is adjusted to trigger at the same position on the received waveform for each measurement (see Figure 6). The counter start pulse is provided directly from the pulse synchronizer. The total transit time is then read from the counter.

The measured total time is not the actual transit time through the sample; there is a propagation delay through the two transducers and a time lag for the first zero crossover. The measurement is corrected for these delay times by measuring the propagation time between the transducers with no sample present. This time is then subtracted from each measurement on the sample. The velocity is then calculated based on the measured distance and corrected transit time. A photograph of the measurement system is shown in Figure 7.

FIGURE 4. Photograph of Typical Samples

TABLE 2. Property Measurements on 1-inch Diameter Samples

Sample No.	Axial Measurements						Diam. V_L (m/s)
	V_L (m/s)	V_s (m/s)	Poisson's Ratio	Density (Mg/m ³)	E_e (GPa)	E_s (GPa)	
1	2384	1572	0.1147	1.804	9.945	4.460	2519
2	2402	1576	0.1221	1.804	10.052	4.479	2519
3	2406	1575	0.1248	1.800	10.050	4.468	2506
4	2428	1570	0.1408	1.797	10.102	4.428	2507
6	2402	1577	0.1209	1.800	10.041	4.479	2523
7	2413	1579	0.1261	1.800	10.101	4.485	2508
8	2414	1583	0.1227	1.803	10.149	4.520	2513
12	2446	1573	0.1476	1.800	10.217	4.451	2522
13	2416	1573	0.1319	1.798	10.072	4.450	2491
14	2404	1575	0.1242	1.796	10.012	4.453	2486
16	2403	1570	0.1277	1.800	10.002	4.435	2512
17	2422	1599	0.1137	1.802	10.260	4.606	2501
18	2418	1583	0.1245	1.800	10.148	4.512	2527
22	2448	1569	0.1516	1.799	10.198	4.428	2497
23	2411	1574	0.1289	1.800	10.066	4.458	2489
24	2431	1578	0.1359	1.802	10.193	4.487	2499
26	2420	1579	0.1291	1.803	10.153	4.496	2531
27	2425	1581	0.1300	1.804	10.195	4.511	2527
28	2414	1587	0.1196	1.798	10.137	4.527	2514
33	2368	1564	0.1130	1.799	9.793	4.399	2494
34	2403	1568	0.1291	1.804	10.017	4.436	2523
36	2423	1582	0.1284	1.806	10.203	4.521	2515
37	2427	1587	0.1264	1.808	10.260	4.554	2526
52	2396	1575	0.1200	1.805	10.027	4.476	2526
53	2405	1573	0.1260	1.798	10.025	4.451	2502
54	2426	1577	0.1346	1.799	10.147	4.472	2497

TABLE 2. (cont'd)

Sample No.	Axial Measurements						Diam. V_L (m/s)
	V_L (m/s)	V_S (m/s)	Poisson's Ratio	Density (Mg/m ³)	E_e (GPa)	E_s (GPa)	
56	2426	1581	0.1309	1.800	10.176	4.499	2501
57	2416	1581	0.1256	1.800	10.126	4.498	2533
58	2479	1586	0.1534	1.803	10.460	4.535	2530
62	2389	1577	0.1140	1.805	10.002	4.489	2527
63	2418	1581	0.1261	1.802	10.149	4.507	2527
64	2443	1584	0.1371	1.806	10.311	4.534	2533
66	2409	1582	0.1206	1.799	10.096	4.505	2515
67	2408	1577	0.1249	1.801	10.075	4.478	2531
68	2408	1580	0.1222	1.801	10.085	4.493	2504
73	2409	1584	0.1193	1.810	10.162	4.539	2537
74	2413	1579	0.1260	1.803	10.119	4.493	2517
76	2393	1585	0.1093	1.802	10.046	4.528	2516
77	2477	1582	0.1552	1.803	10.428	4.513	2537

TABLE 3. Property Measurements on 3-inch Diameter Samples

Sample No.	Diametral Measurements						Axial Measurements				
	V_L (m/s)	V_S (m/s)	Poisson's Ratio	Density (Mg/m ³)	E_e (GPa)	E_s (GPa)	V_L (m/s)	V_S (m/s)	Poisson's Ratio	E_e (GPa)	E_s (GPa)
II-B-2	2484	1617	0.1319	1.806	10.695	4.725	2419	1587	0.1222	10.209	4.548
II-B-4	2523	1605	0.1603	1.800	10.754	4.634	2376	1569	0.1129	9.869	4.434
II-D-2	2481	1609	0.1375	1.791	10.540	4.634	2403	1586	0.1140	10.038	4.505
II-D-4	2439	1595	0.1267	1.782	10.214	4.533	2398	1576	0.1201	9.913	4.425
II-E-2	2454	1599	0.1311	1.788	10.340	4.571	2405	1586	0.1149	10.035	4.500
II-E-4	2438	1590	0.1303	1.784	10.189	4.507	2399	1587	0.1111	9.985	4.493
II-D-1	2467	1603	0.1346	1.787	10.420	4.592	2400	1584	0.1143	9.988	4.482
II-D-3	2466	1606	0.1321	1.789	10.445	4.613	2413	1585	0.1202	10.072	4.496
II-C-2	2466	1603	0.1341	1.800	10.488	4.624	2414	1578	0.1266	10.104	4.484
II-C-3	2455	1604	0.1279	1.789	10.377	4.600	2399	1583	0.1148	9.992	4.481
II-C-4	2487	1593	0.1517	1.780	10.409	4.519	2369	1571	0.1070	9.723	4.392
I-G-1	2477	1597	0.1440	1.798	10.498	4.588	2411	1580	0.1242	10.088	4.486
I-G-2	2485	1618	0.1319	1.809	10.728	4.739	2418	1592	0.1177	10.243	4.582
I-G-3	2482	1617	0.1313	1.810	10.710	4.734	2404	1594	0.1071	10.177	4.597
I-G-4	2510	1611	0.1496	1.804	10.770	4.684	2411	1597	0.1086	10.206	4.603

FIGURE 5. Block Diagram of Ultrasonic Velocity Measurement System

FIGURE 6. Typical Oscilloscope Output

FIGURE 7. Photograph of Measurement System

LONGITUDINAL WAVE MEASUREMENTS

Measurement of longitudinal velocities are made using the system shown in Figures 5 and 7 and the transducers shown in Figure 8. These transducers are conventional longitudinal units with the addition of a rubber coupling layer on the front face. The rubber layer provides ultrasonic coupling to the graphite sample without the use of a liquid couplant. (Residual liquid couplant would be deleterious to the graphite during HTGR conditions.) The rubber coupling layer is less effective than a liquid couplant; however, we were able to transmit sufficient energy through these layers to perform the measurements over a path length of 12 inches, the length of the longest sample.

The transducers shown in Figure 8 have a resonant frequency of 2.25 MHz. Transducers with frequencies down to 0.5 MHz were also used for measurements of longitudinal velocities over path lengths exceeding 8 inches in unoxidized material and 6 or more inches in highly oxidized material. Oxidation increases the ultrasonic attenuation of the graphite which necessitates the use of lower frequency transducers. Highly oxidized surface layers also degrade the coupling efficiency of the rubber coupling layer.

The rubber coupling material used in these tests would not be suitable at HTGR shutdown temperatures; however, suitable materials are available for this purpose. The remainder of the transducer would be satisfactory for the measurements under HTGR shutdown conditions.

SHEAR WAVE MEASUREMENTS

Transducers used for shear velocity measurements are shown in Figure 9. These transducers require no couplant and are pressure coupled to the test piece. Pressures of 10 to 20 lbs. are sufficient to produce maximum coupling on graphite.

The construction of these transducers is quite simple. A shear transducer element is bonded to a 0.25 inch diameter steel rod. The end of the rod is tapered to yield a contact point of approximately 0.125 inch. (For harder materials, such as steel, sharp contact points are required.) The polarization axis of the receiving and transmitting transducers are aligned parallel when testing the graphite samples.

FIGURE 8. Transducers for Longitudinal Wave Velocities

FIGURE 9. Transducer for Shear Wave Velocities

Graphite crystals are highly anisotropic and, since shear waves are polarized (displacements are perpendicular to the propagation direction), the measured shear wave velocities vary as function of the polarization axis of the shear wave in relation to a preferred orientation of crystallites which is developed in the log during the manufacturing process. In addition, there can be localized variations in preferred orientation, especially with small samples. In this program, we have elected to use the average velocity of two orthogonally polarized shear waves. For measurements through the diameter, shear waves are polarized parallel to the rod axis and measured at a minimum of three lengthwise positions and a minimum of two equally spaced azimuthal positions. The shear wave polarization of the transducers are then rotated 90° and the measurements repeated. The average of these readings are used to characterize the specimen. Measurements through the length of the sample are made in two orthogonal polarization directions at the center and a minimum of three positions at one-half the sample radius. The measurements are averaged to determine the preoxidation properties of the sample.

It should be noted that the two polarization orientations may not yield maximum or minimum shear wave velocities; however, the technique provides a repeatable method for characterizing the changes in material properties due to oxidation.

NEAR-SURFACE SHEAR WAVE MEASUREMENTS

The near-surface shear velocity along the length of the sample was also measured using the shear wave probes described above. Since these probes are approximately a point source, substantial energy is transmitted parallel to the surface. The probes are mounted in a fixture as shown in Figure 10, with a probe separation of 4 inches. Shear waves are polarized parallel to the axis of the sample and penetrate to an effective depth of less than 0.5 inches.

FIGURE 10, Mounting Fixture for Near-surface Shear Wave Velocity Measurements

This measurement was used to qualitatively characterize the inhomogeneity of the oxidation profile with depth. Where the oxidation was uniform through the diameter, little difference was noted between the near-surface velocity and that through the diameter. Where the oxidation was principally near the surface, the velocity changes of the near-surface wave were as much as 23 times greater than that through the diameter. Further investigation will be required to evaluate the effectiveness of this measurement technique in determining the actual oxidation profiles.

MEASUREMENT ACCURACY

The accuracy of this measurement technique depends primarily on the following:

1. Stability of the zero crossover detector.
2. Consistency of coupling pressure.
3. The accuracy of the counter.

The accuracy of the counter (HP 5327B) far exceeds the requirements of this measurement. The counter, in the period average mode, has a calibrated accuracy of 0.001%. The limiting factors are the zero crossover detector and the coupling pressure. Evaluation of the system on a sample of fused quartz of known velocity indicated a system accuracy of 0.2%. Measurements on an unoxidized sample of graphite showed the average of ten measurements on the sample could be repeated within 0.3%. A portion of this repeatability error is due to the point to point variations in the graphite velocity. Based on the above, it is expected that the velocity differences (before and after oxidation) are accurate to within $\pm 0.5\%$.

Improvements in system performance can be achieved by:

1. Fabrication of transducers (longitudinal and shear) shaped to fit the curvature of the parts to be tested. This would increase signal amplitude and the signal to noise ratio.
2. The use of rectangular rather than circular contact areas for the shear probes. This would give improved polarization control of the shear wave measurement.
3. Fabrication of more suitable testing fixtures to provide consistent incident points on the sample and uniform coupling pressures.

RESULTS

STRENGTH-MODULUS RELATIONSHIPS

Compressive strength was measured on four of the 3-inch diameter samples and various relationships between strength, Young's modulus, shear modulus, Poisson's ratio, and density were examined; the results are shown in Table 4. It can be seen that the Griffith relationship, $S \sim \sqrt{E/\sigma}$, would not provide a good correlation of these data. The best correlations appear to be of the form $S \sim E^2 \rho^2$.^(f) However, attempts to correlate the strength of the 1-inch diameter samples using this relationship led to large deviations between measured and predicted strength. An increase in the deviation of individual strength, as well as the observed increase in mean strength, is to be expected as sample size is decreased; but, in the present case, the increased deviation was uncomfortably large.

In order to further examine this phenomenon, strengths were obtained on two additional 3-inch diameter samples; a computer program, for obtaining a linear correlation of three variables, was written and a parametric study was made of the correlation of strength with density, moduli, and Poisson's ratio. The results, shown in Table 5, confirm the conclusions drawn from the results in Table 4 for the 3-inch diameter samples and shows that good correlations of the strength can be made with the moduli and Poisson's ratio. It also shows that the best correlation for the 1-inch samples is quite different than those obtained for the 3-inch samples. It should be noted, however, that the ability to obtain a good correlation for a small number of samples does not assure that the correlation will apply to other samples; this is especially so when the properties are not independent variables (in the present case, none of the properties are independent variables).

From purely theoretical grounds (see Appendix A), one would expect a correlation of the general form $S \sim \rho^\alpha E^\beta \sigma^{-\gamma}$ with all exponents equal or greater than zero; using this as a criterion, only five of the twelve

(f) This subject is discussed further in Appendix A.

TABLE 4. Strength-Modulus Relationships

Young's Moduli

Sample No.	Strength MPa	Diametral Measurements			Axial Measurements		
		$S\left(\frac{\sigma}{E}\right)^{\frac{1}{2}}$	S/E	S/E^2	$S\left(\frac{\sigma}{E}\right)^{\frac{1}{2}}$	S/E	S/E^2
II-B-2	77.83	8.643	7.277	0.6804	8.515	7.624	0.7468
II-B-4	72.06	8.798	6.701	0.6231	7.707	7.302	0.7399
II-D-2	73.86	8.436	7.008	0.6649	7.871	7.358	0.7330
II-E-2	<u>72.60</u>	<u>8.175</u>	<u>7.021</u>	<u>0.6790</u>	<u>7.769</u>	<u>7.235</u>	<u>0.7209</u>
Ave	74.09	8.513	7.002	0.6618	7.965	7.380	0.7351
$\pm 1\sigma$ (a)	± 2.61	± 0.270	± 0.236	± 0.0268	± 0.373	± 0.170	± 0.0110
$\pm \delta$ (%)	$\pm 3.52\%$	$\pm 3.17\%$	$\pm 3.37\%$	$\pm 4.04\%$	$\pm 4.68\%$	$\pm 2.31\%$	$\pm 1.50\%$
							± 0.0051
							$\pm 0.69\%$

22 Shear Moduli

II-B-2	77.83	13.00	16.47	3.486	3.448	12.76	17.11	3.763	3.721
II-B-4	72.06	13.40	15.55	3.356	3.341	11.50	16.25	3.665	3.649
II-D-2	73.86	12.72	15.94	3.440	3.459	11.75	16.40	3.639	3.660
II-E-2	72.60	12.30	15.88	3.475	3.506	11.60	16.13	3.585	3.617
Ave.	74.09	12.86	15.96	3.439	3.439	11.90	16.47	3.663	3.662
$\pm 1\sigma$	± 2.61	± 0.463	± 0.381	± 0.059	± 0.070	± 0.58	± 0.44	± 0.075	± 0.044
$\pm 1\delta(\%)$	$\pm 3.52\%$	$\pm 3.61\%$	$\pm 2.39\%$	$\pm 1.71\%$	$\pm 2.03\%$	$\pm 4.88\%$	$\pm 2.67\%$	$\pm 2.03\%$	$\pm 1.19\%$

(a) Plus or minus one standard deviation.

TABLE 5. Correlations of Strength with Properties

Sample Size and Direction of Measurements	Correlation Equations	Maximum Difference (Percent)	Percent of Variance Removed
3 inch Diametral Measurements	$S = 0.6109 E_e^{1.69} \sigma^{-0.412}$	-1.4	91
	$S = 3.875 E_s^{1.67} \sigma^{-0.198}$	-1.4	91
3 inch Axial Measurements	$S = 0.3424 \rho^{1.84} E_e^{1.87}$	1.4	92
	$S = 1.338 \rho^{2.41} E_s^{1.74}$	-1.9	79
1 inch Axial Measurements	$S = 0.0412 \rho^{11.8} E_s^{0.455} \text{ (a)}$	-5.1	17

(a) Although the general form of this correlation appears physically reasonable, the dependence on density (ρ) seems unreasonably large.

correlations would appear physically reasonable; these are: The correlations with moduli and Poisson's ratio measured through the diameter of the 3-inch samples, the correlations with density and moduli measured along the axis of the 3-inch samples, and the density-shear modulus correlation for the 1-inch samples (the latter correlation does not, however, remove much of the variance).

One additional parametric study was carried out; correlations were obtained using the assumptions that $\alpha=\beta$ or $\beta=\gamma$. It should not be assumed that either approximation is correct; however, the results shown in Table 6 indicate that the correlations are substantially improved for the measurements made through the sample diameters. In contrast, none of the correlations for the axial 3-inch measurements, and only the $\beta=\gamma$ correlations for the 1-inch samples, meet the a priori criterion mentioned above.

The results suggest, however, that with additional data over a greater range of variations in properties (as might be expected during long-term production), that the best results might be obtained from a linear correlation of all four variables (or by treating ultrasonic velocity as the fourth variable). Limitations on time and funding did not allow us to pursue this further. The above correlations confirm the feasibility of nondestructively determining compressive strength from measurements of ultrasonic velocity and density.

RESULTS AFTER OXIDATION: 3-INCH SAMPLES

Based on the results of our review of the existing experimental data, we concluded that one might be expected to obtain a correlation between the change in strength and the change in ultrasonic velocity of the general form:

$$(S/S_0) = (V/V_0)^\beta, \text{ with } \beta \approx 2.0. \quad (2)$$

However, the data was very sparse and there was possibility that differences in oxidation gradient might effect the correlation (i.e., that a measure of the oxidation profile within the posts might be required if we were to obtain a valid correlation). Therefore, ultrasonic velocities were measured:

1) at several radial positions parallel to the axis of the samples, 2) through

TABLE 6. Other Strength-Property Relationships

Sample Size and Direction of Measurement	Correlation Equations	Maximum Difference (Percent)	Percent Variance Removed
3 inch Diametral Measurements	$S = 0.6439 (\rho E_e)^{1.35} \sigma^{-0.388}$	-1.2	93
	$S = 2.821 (\rho E_s)^{1.34} \sigma^{-0.217}$	-1.2	93
	$S = 0.9699 \rho^{5.16} (E_e/\sigma)^{0.303} \text{ (a)}$	0.9	95
	$S = 1.322 \rho^{5.28} (E_s/\sigma)^{0.266} \text{ (a)}$	0.9	95
1 inch Axial Measurements	$S = 0.3404 \rho^{7.36} (E_e/\sigma)^{0.262} \text{ (a)}$	-5.4	44
	$S = 0.4432 \rho^{7.42} (E_s/\sigma)^{0.239} \text{ (a)}$	-5.4	45

(a) Although the general form of this correlation appears physically reasonable, the dependence on density (ρ) seems unreasonably large.

the diameter at several positions, and 3) in a thin layer near the sample surface.

A computer program was written to calculate the oxidation rate profiles in cylindrical samples and a parametric study was conducted to determine the effect of temperature (reaction rate) and gas composition on the oxidation profiles. Conditions were then selected to provide quite different oxidation gradients, without causing significant differences in oxidation rate along the length of individual samples. After oxidation, 1 inch was removed from each end of the samples to reduce the effects of inhomogeneous oxidation; the densities and ultrasonic velocities shown in Tables 7 and 8 were measured after the reduction in length. Table 8 shows the differences in changes of the shear wave velocity at the surface to the changes along the axis of the oxidized 3-inch diameter samples. The ratio of 1.2 for sample I-G-4 indicates that the oxidation took place almost homogeneously throughout this sample; in contrast, oxidation of sample II-C-2 occurred primarily at the surface.

The strength after oxidation was calculated in the following way:

a) pre-oxidation strength (S_0) was estimated using one of the correlation equations shown in Tables 5 and 6; b) the exponent β was calculated from equation 2 using the measured strength and the average change^(g) in ultrasonic velocity; and c) the average value of β , for all nine samples, was then used to calculate the oxidized strength.

Table 9 shows the results for the best case, where the correlation of strength with density and Young's Modulus ($S_0 = 0.3424\rho^{1.84}E_e^{1.87}$) was used to calculate the original strength. For this correlation, the largest difference between calculated and measured strength is 1.85 MPa (Sample No. I-G-4). The largest difference in any of the 8 correlations is 3.86 MPa (6.40% of the measured strength of sample I-G-4) for the calculation using Young's Modulus and Poisson's ratio, measured through the sample diameter ($S_0 = 0.6109E_e^{1.69}\sigma^{-0.412}$).

g. The average $(v/v_0) = \text{exponential of } [\ln (v/v_0)_s + \ln (v/v_0)_e]/2$ as measured through the sample diameters; this "average" is, however, seldom different than the simple average:

$$[(v/v_0)_s + (v/v_0)_e]/2$$

TABLE 7. Properties Measured After Oxidation

Sample No.	Diam. Meas.		Poisson's Ratio	Density (Mg/m ³)	E _e (GPa)	E _s (GPa)
	V _l (m/s)	V _s (m/s)				
II-D-1	2219	1491	0.0884	1.691	8.185	3.760
II-D-3	2221	1482	0.0982	1.676	8.088	3.683
II-C-2	2195	1426	0.1352	1.663	7.674	3.380
II-C-3	2194	1443	0.1193	1.666	7.763	3.468
II-C-4	2234	1475	0.1136	1.685	8.168	3.667
I-G-1	2042	1337	0.1246	1.624	6.532	2.904
I-G-2	2115	1384	0.1252	1.663	7.169	3.186
I-G-3	2138	1369	0.1524	1.665	7.193	3.121
I-G-4	2212	1433	0.1388	1.711	7.997	3.510

TABLE 8. Changes for Oxidized 3 inch Diameter Samples

Sample No.	ρ/ρ_o (a)	Changes in Velocity							Ratio (b)	
		Through Diam. $(v/v_o)_L$ $(v/v_o)_S$	Axial $(v/v_o)_L$ $(v/v_o)_S$	Center Line $(v/v_o)_L$ $(v/v_o)_S$	"Surface" $(v/v_o)_S$					
<u>"Oxidized (c) 9 days at 900°C (d)"</u>										
II-D-1	0.9457	0.8996	0.9303	0.9500	0.9626	0.9625	0.9828	0.8357	9.55X	
II-D-3	0.9365	0.9004	0.9231	0.9569	0.9792	0.9717	0.9909	0.8355	18.08X	
<u>"Oxidized 9 days at 950°C"</u>										
II-C-2	0.9238	0.8903	0.8895	0.9739	0.9453	0.9855	0.9910	0.7911	23.21X	
II-C-3	0.9309	0.8938	0.8997	0.9771	0.9596	0.9791	0.9857	0.8092	13.34X	
II-C-4	0.9465	0.8986	0.9259	0.9681	0.9494	0.9777	0.9832	0.8260	10.36X	
<u>"Oxidized 28 1/2 days at 875°C"</u>										
I-G-1	0.9041	0.8244	0.8372	0.8405	0.8495	0.8533	0.8723	0.7783	1.74X	
I-G-2	0.9196	0.8508	0.8551	0.8602	0.8635	0.8690	0.9084	0.8117	2.06X	
I-G-3	0.9198	0.8613	0.8466	0.8728	0.8805	0.8805	0.8966	0.8132	1.81X	
I-G-4	0.9489	0.8812	0.8889	0.8808	0.8896	0.8950	0.8951	0.8716	1.22X	

(a) Density change

(b) Ratio of the change of shear wave velocity at the surface to the change measured along the central axis of the sample.

(c) All samples oxidized in mixture of 80% CO₂ plus 20% CO.

(d) Air leaked into the furnace during the first 3 1/2 days.

TABLE 9. Prediction of Strength from Density and Young's Modulus

Sample No.	Measured S (MPa)	Average $\frac{(v/v_o)}{o}$	Calculated S_o (MPa)	Calculated β	Calculated S (MPa)	Difference (MPa)
II-D-1	59.74	0.9148	72.89	2.234	59.07	0.67
II-D-3	59.23	0.9117	74.19	2.436	59.64	-0.41
II-C-2	57.26	0.8899	75.48	2.368	57.31	-0.05
II-C-3	56.28	0.8968	73.09	2.400	56.52	-0.24
II-C-4	53.62	0.9121	68.83	2.713	55.39	-1.77
I-G-1	47.70	0.8308	75.10	2.449	48.48	-0.78
I-G-2	55.02	0.8529	78.14	2.205	53.67	1.35
I-G-3	53.36	0.9539	77.28	2.345	53.23	0.13
I-G-4	60.34	0.8890	77.22	2.096	58.49	1.85

Table 10 shows the results obtained for the correlation with density and shear modulus, also measured parallel to the axis of the samples.

It should be noted that with $\beta=2.0$, the correlation of change in strength with change in ultrasonic velocity would be completely independent of oxidation profile. The observed average value of β is slightly larger than 2.0, so one can expect a small dependence on oxidation profile; however, the accuracy of the present correlations appears to be more than adequate for the proposed application.

EFFECT OF STRESS AND PRESTRESSING

A primary question which needed to be answered was whether stresses applied during oxidation, or stressing prior to oxidation, would alter the correlations of strength and ultrasonic velocity. Krefeld et al.,^(7,8) have reported that the oxidation rate can be increased as much as a factor of 5 by applying tensile stresses of the order of one-tenth of their ultimate strength. Moreover, we inferred⁽¹⁾ from their results that the loss of strength with oxidation might also be significantly increased.

The 1-inch diam. samples were randomly divided into 3 groups prior to oxidation; one set was constrained at approximately 40% of ultimate compressive strength during oxidation (see Appendix B for details), one set was stressed to approximately 20% of ultimate compressive strength prior to oxidation and the third set was oxidized without further treatment. All samples were oxidized together for 14 days at 875°C in the same CO₂/CO mixture used for the 3-inch diam. samples.

Table 11 shows the relative order of the samples in the furnace and their weight changes, prior to their reduction to 2 inches in length. The difference between the unstressed and prestressed samples is insignificant; however, the results of a "t" test indicate that there is between 80 and 90% probability of a 16% increase in oxidation rate for the stressed samples.

Because of the large variation in oxidation rate, rank correlation tests were also made to determine if relative oxidation rate correlated with position of the samples in the furnace. The prestressed samples showed an inverse

TABLE 12. Prediction of Strength from Density and SHEAR Modulus

<u>Sample No.</u>	<u>Measured S (MPa)</u>	<u>Average (v/v_o)</u>	<u>Calculated S_o (MPa)</u>	<u>Calculated β</u>	<u>Calculated S' (MPa)</u>	<u>Difference (MPa)</u>
II-D-1	59.74	0.9148	72.99	2.250	59.09	0.65
II-D-3	59.23	0.9117	73.59	2.348	59.09	0.14
II-C-2	57.26	0.8899	74.33	2.237	56.36	0.90
II-C-3	56.28	0.8968	73.16	2.408	56.50	-0.22
II-C-4	53.62	0.9121	69.81	2.867	56.11	-2.49
I-G-1	47.70	0.8308	74.18	2.383	47.79	-0.09
I-G-2	55.02	0.8529	78.11	2.202	53.54	1.48
I-G-3	53.36	0.8539	78.66	2.457	54.07	-0.71
I-G-4	60.34	0.8890	78.21	2.204	59.15	1.19

TABLE 11. Relative Position in Furnace and Weight
Loss of 1 inch Diameter Samples

<u>Un-stressed</u>		<u>Pre-stressed</u>		<u>Stressed</u>	
<u>No.</u>	<u>W/Wo</u>	<u>No.</u>	<u>W/Wo</u>	<u>No.</u>	<u>W/Wo</u>
16	0.8205			2	0.7726
		23	0.7513		
18	0.8131			22	0.7761
		7	0.7780		
3	0.7578			13	0.6983
		77	0.7911		
24	0.7556			8	0.7776
		64	0.7494		
66	0.7612			54	0.7553
		52	0.7898		
68	0.8225			67	0.7104
		14	0.7778		
1	0.9231			74	0.7208
		56	0.9179		
33	0.7809			62	0.8276
		37	0.8720		
57	0.7538			27	0.7643
		58	0.7979		
63	0.7521			36	0.8079
- - - - -		- - - - -		- - - - -	
Ave	0.7941		0.8028		0.7611
$\pm 1\sigma$	0.0537		± 0.0560		± 0.0414
$\pm 1\sigma$ (%)	$\pm 6.76\%$		$\pm 6.98\%$		$\pm 5.44\%$

correlation (i.e., the lowest oxidation rate occurred at the gas-inlet end of the furnace), which does not appear reasonable; the rates of the other two sets of samples did not correlate (i.e., the test results indicate a random ordering) with furnace position.

Table 12 shows the differences in density change of the 1-inch diam. samples after reduction to 2 inches in length. Although it can be noted that the three samples with the highest oxidation rates were stressed, and the three lowest were not stressed during oxidation, it should also be noted that 4 of the 5 samples with the lowest oxidation rates came from positions immediately adjacent to the surface of the log and that the 6 highest came from interior, but not central, positions (see Figure 2).

Rank correlation tests for all three sets of samples showed a high probability of a correlation existing between radial positions of a sample in the log and its oxidation rate; however, the results also suggested that samples 16 and 56, two of the four-innermost samples, should be excluded. This is further illustrated in Table 13, which shows the results of the rank correlation tests with no distinction made between the three sets of samples. For all 29 samples, there is better than 95% probability that the oxidation rate correlates with radial position of the sample within the log; excluding samples 16 and 56 increases that probability to much more than 99%.

It appears most likely that the variation in oxidation rate is due to a variation in the concentration of catalytic impurities. A much larger variation in oxidation rate has been observed for different samples of Grade 2020, and the highest oxidation rates have been linked to increased iron impurity level.⁽⁹⁾ It should be noted, however, that 2020 is not a "nuclear grade" graphite; a nuclear grade (say 2020-N) would normally go through a purification process, which would remove the catalytic impurities without altering the physical properties.

As is further discussed in Appendix B, even the lowest oxidation rates were much higher than the oxidation rate for TSX graphite (TSX is a very pure nuclear grade graphite, with lower strength and lower density than Grade 2020).

TABLE 12. Density Changes for Oxidized 1 inch
Diameter Samples

<u>Unstressed</u>		<u>Prestressed</u>		<u>Stressed</u>	
<u>Sample Number</u>	<u>Density Change</u>	<u>Sample Number</u>	<u>Density Change</u>	<u>Sample Number</u>	<u>Density Change</u>
1	-7.2%	56	-8.0%	62	-17.4%
68	17.6	37	12.0	36	19.1
16	17.8	58	19.9	8	22.2
18	18.3	52	20.7	22	22.3
33	21.8	77	20.8	2	22.4
57	22.6	14	22.0	27	23.4
66	23.6	7	22.1	54	24.7
3	24.1	64	24.8	74	27.8
24	24.3	23	25.0	67	28.9
63	24.7			13	30.3

TABLE 13. Rank Correlation Tests for 1 inch Diameter Samples

No.	All Samples			Minus Samples #16 and 56		
	Relative Rate	Radial Position	Difference	Relative Rate	Radial Position	Difference
1	1	1	-0-	1	1	-0-
56	2	26½	24½	-	-	-
37	3	3	-0-	2	3	1
62	4	6	2	3	6	3
68	5	3	2	4	3	1
16	6	26½	20½	-	-	-
18	7	9	2	5	9	4
36	8	9	1	6	9	3
58	9	9	-0-	7	9	2
52	10	11½	1½	8	11½	3½
77	11	3	8	9	3	6
33	12	6	6	10	6	4
14	13	28½	15½	11	26½	15½
7	14	24	10	12	24	12
8	15	13	2	13	13	-0-
22	16	6	10	14	6	8
2	17	14	3	15	14	1
57	18	19½	1½	16	19½	3½
27	19	15½	3½	17	15½	1½
66	20	19½	½	18	19½	1½
3	21	25	4	19	25	6
24	22	22	-0-	20	22	2
54	23½	28½	5	21½	26½	5
63	23½	17½	6	21½	17½	4
64	25	22	3	23	22	1
23	26	17½	8½	24	17½	6½
74	27	11½	15½	25	11½	13½
67	28	15½	12½	26	15½	10½
13	29	22	7	27	22	5
			—			—
Σd^2			2207.5			1012
$R = 1 - \frac{6 \Sigma d^2}{n(n^2-1)}$			0.456			0.691

Therefore, the present results should not be interpreted as being contradictory to the results of Krefeld et al.;^(7,8) nor can they be considered definitive as to the effect of stress on the strength-ultrasonic velocity correlation for well-purified nuclear graphites. However, it can be seen from Figure 11 that (for these samples) the correlation of strength change with change in ultrasonic velocity is not affected by the changes in oxidation rate, the effects of stress during oxidation, or compressive stressing of the samples prior to oxidation.

CONCLUSIONS

- These results indicate that it is technically feasible to monitor loss of strength of the core support structure of an operating HTGR.
- Several excellent correlations were developed to nondestructively predict the compressive strength of unoxidized 3 inch diameter samples of Stackpole grade 2020 graphite.
- The results, which were obtained from linear correlations of three variables, suggest that even better relationships could be developed from a linear correlation of four variables (strength, density, Young's or shear modulus, and Poisson's ratio; or with ultrasonic velocities considered as independent variables, rather than only using them to calculate the moduli and poisson's ratio).
- Although good correlations were also developed for 1-inch diameter samples, the increased dependence of compressive strength on flaw distribution and localized variations in structure result in more variability and reduced precision in the predictions.
- Excellent predictions of strength after oxidation were obtained using the unoxidized strength correlations and changes in ultrasonic velocities, measured through the diameters of the 3-inch diameter samples.
- Although the results obtained on the 1-inch diameter samples are not considered definitive, it appears that the correlations are not affected by catalytic oxidation, by compressive stressing of samples prior to oxidation (proof-testing of HTGR Core-Support Posts), or by compressive stresses applied during oxidation.

- The form of the correlation between the changes in strength and the changes in ultrasonic velocities is such that it is also practically insensitive to changes in oxidation profile; thus, this correlation method should be readily adaptable to prediction of the strength changes of HTGR Core-Support Posts.

FIGURE 11. Strength Versus Relative Velocity for Oxidized 1-inch Diameter Samples

APPENDIX A

Effect of Porosity

Porous solids may generally be divided into two different classifications; these are:

- (1) A continuous solid phase containing discrete pores (or bubbles), or
- (2) A continuous pore phase containing particles which are bonded together at points of contact.

The dependence of elastic properties on porosity has been derived (a) by MacKenzie⁽¹⁰⁾ for the continuous solid-phase model, and (b) by Kerner⁽¹¹⁾ for the continuous pore-phase model. Both models yield a very large dependence of elastic moduli on porosity, with the continuous pore-phase model predicting the largest effect. Most brittle materials exhibit a dependency which is intermediate between the predictions, and Euler⁽¹²⁾ has shown how the two models can be combined to yield a better fit to experimental data.

Similar theoretical analyses for the dependence of strength on porosity are not, however, available; empirical expressions for the variation of strength with porosity of various ceramic materials⁽¹³⁻¹⁵⁾ show that 10% change in porosity will produce about a 50% change in strength. For example, Ryschkewitsch⁽¹³⁾ reported that 10 percent change in density produced a 50 percent change in the compressive strength of sintered Al_2O_3 and ZrO_2 ; this dependence was also confirmed by Duckworth⁽¹⁴⁾ for silicate porcelain and is the "rule-of-thumb" which emanated from pioneering work of Griggs⁽¹⁶⁾ on the strength of graphite after oxidation.

It can be seen from Figure A-1 that this general "rule-of-thumb" applies equally well to Stackpole 2020; the curve was drawn from the average breaking load of the unoxidized samples on the basis that 10% weight loss would result in a 50% reduction in breaking load (the straight lines merely serve to bound the data points.)

The studies reported in references 13 to 15 covered a range of relative densities from about 97% to less than 50% (the relative density of Stackpole 2020 is about 80%). The calculated change in elastic moduli between 80 and 70% relative density (12.5% in bulk density) is almost 40%; using the empirical correlations: $(S/S_0) = (\rho/\rho_0)^\alpha$, $(E/E_0) = (\rho/\rho_0)^\beta$, and $(S/S_0) = (E/E_0)^\lambda$, $\lambda = \alpha/\beta$. The "rule-of-thumb" gives $\alpha = 6.58$, Euler's model gives $\beta = 3.78$, and $\lambda = 1.74$.

The exponents on E, for the "physically reasonable" correlations in Table 5 range from 1.67 to 1.87, in excellent agreement with the value of 1.74 determined above.

FIGURE A-1. Breaking Load Versus Weight Loss for Oxidized 1-inch Diameter Samples

APPENDIX B

STRESS DURING OXIDATION

Figure B-1 shows the configuration of the sample holders which were used to apply compressive stress on 1-inch diameter samples during oxidation. The sample holders were machined from National Carbon Company grade TSX graphite, which is a fine-grained, high purity nuclear graphite. Sample ends and the interior contact surfaces of the holders were machined smooth and parallel; firm pressure was required to slide the samples into the holders at room temperature.

The coefficients of thermal expansion between 25 and 875°C (the oxidation temperature) are approximately $1.63 \times 10^{-6}/^{\circ}\text{C}$ for TSX parallel to the axis of extrusion, and $5.42 \times 10^{-6}/^{\circ}\text{C}$ for the log of Stackpole 2020 from which the 1-inch diameter samples were cut. Thus, the total difference in thermal expansion was about 3.2×10^{-3} . Young's modulus of the samples average slightly more than 10 GPa. The Young's modulus for TSX was slightly higher and more variable; but, since the cross sectional area of the sample holders was 7.64 times the cross section of the samples, it has only a small effect on the stresses generated in the samples. A larger uncertainty in the stress-at-temperature is introduced by possible deformation of the contact surfaces; however, since gentle pressure was still required to remove some of the samples from the sample holders after oxidation, we concluded that the compressive stress in the samples was approximately 30 MPa, or approximately 35% of their ultimate compressive strength prior to oxidation.

Since strength loss during oxidation was approximately proportional to change in modulus, the samples remained at approximately 35% of ultimate compressive stress during the entire oxidation period.

FIGURE B-1. Sample Holder for Applying Stress During Oxidation

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