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

W. C. Morgan
F. L. Becker

August 1979

Prepared for
the U.S. Nuclear Regulatory Commission

Pacific Northwest Laboratory
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FEASIBILITY OF MONITORING THE STRENGTH OF
HTGR CORE SUPPORT GRAPHITE - PART II

W. C. Morgan
F. L. Becker

August 1979

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FEASIBILITY OF MONITORING THE STRENGTH OF
HIGH CORE SUPPORT GRAPHITE - PART II

W. C. Hootan
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August 1972

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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. Correlations have been established between the velocity of an ultrasonic pulse and the compressive strength of four different grades of graphite. For some grades of graphite, one or more of the correlations are practically independent of oxidation profile in samples having cylindrical geometry (as in the core support posts). For other grades of graphite, and for other sample geometries, the oxidation-depth profile must be known in order to reliably predict the effect of oxidation on compressive strength.

ABSTRACT

The results reported herein establish the technical feasibility of a method for monitoring the strength of HIC core support structures in situ. Correlations have been established between the velocity of an ultrasonic pulse and the compressive strength of four different grades of graphite, for some grades of graphite, one or more of the correlations are practically independent of oxidation profile in samples having cylindrical geometry (as in the case of support posts). For other grades of graphite, and for other sample geometries, the oxidation profile must be known in order to reliably predict the effect of oxidation on compressive strength.

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LIST OF SYMBOLS AND UNITS

- E = Elastic Moduli (E_e = Young's Modulus, E_s = Shear Modulus) in Gigapascals (1 GPa = 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 = mean of unoxidized strength) in Megapascals (1 MPa = 145.04 psi).
- $\pm\sigma$ = Plus or minus one standard deviation.
- V = Ultrasonic velocity (V_l = velocity of the longitudinal wave, V_s = velocity of the shear wave) in meters per second (1 m/s = 3.2808 ft/sec).
- AME = "As Manufactured" End; "As Manufactured" denotes that the surface has not been machined after graphitization.

LIST OF SYMBOLS AND UNITS

- E = Elastic Modulus (E_s = Young's Modulus, E_v = Shear Modulus) in
Gigapascals (1 GPa = 1.4504×10^9 psi).
- ρ = Density (ρ_s = unoxidized density) in megagrams per cubic meter
(1 Mg/m³ = 1 g/cm³).
- σ_c = Compressive Strength (σ_{c0} = mean of unoxidized strength) in
megapascals (1 MPa = 145.04 psi).
- σ = Plus or minus one standard deviation.
- V = Ultrasonic velocity (V_s = velocity of the longitudinal wave, V_v =
velocity of the shear wave) in meters per second (1 m/s = 3.2808
ft/sec).
- ME = "As Manufactured" (ME) "As Manufactured" denotes that the surface
has not been machined after graphitization.

INTRODUCTION

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

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

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 .

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 obtained in our original investigation,⁽²⁾ using Stackpole 2020 graphite, are consistent with a value of $\gamma \approx 1.0$ ($\beta \approx 2.0$) for changes occurring during oxidation.

SELECTION AND PREPARATION OF SAMPLES

Stackpole^(a) graphite, grade 2020, was selected for our original investigation 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 log identifications and sampling schemes, used in the first investigation, are shown in reference 2.

(a) Stackpole Carbon Co., St. Marys, Pa. 15857

In addition to the remainder of Log II, additional Logs (Airco-Speer^(a) grade S1090, Union Carbide^(b) grade ATJ, and Pure Carbon Co.^(c) grade P3W) were obtained for use in these studies. These particular grades were chosen to encompass a wide range of starting materials and manufacturing variables. Many of these variables are considered proprietary by the graphite companies; but, in general, they include differences in coke types and sources, coke particle size, formation by molding or isostatic pressing, number of impregnations, and final graphitization temperatures. The variables are mentioned here only to indicate that the methods which we have used should be applicable to a wide variety of graphites, not just to the four grades used in this study.

Sampling schemes for the logs are shown in Appendix A. A number of samples were not used in the present study, but were archived for future reference.

All samples were machined to 3-inch diameter. Those which were used to determine the unoxidized properties were 6 inches long; the samples which were to be oxidized were 8 inches long. Oxidized samples were reduced to 6 inches long prior to the measurements of their properties.^(d)

ULTRASONIC VELOCITY MEASUREMENTS

EQUIPMENT

A through-transmission dry-pressure coupling technique was employed for the measurement of ultrasonic velocities. Specialized equipment developed for this program includes the test fixture and ultrasonic transducers. The test fixture and the transducers are shown in Figure 1. This fixture is basically a clamping frame which allows the six-transducer assembly to be pressure

(a) Airco Speer Carbon-Graphite, St. Marys, Pa. 15857

(b) Union Carbide Corporation New York, NY 10017

(c) Pure Carbon Co., St. Marys, Pa. 15857

(d) Compressive strength and density for these and all other samples were measured in accordance with ASTM Methods C-695⁽³⁾ and C-559⁽⁴⁾, respectively; the equipment and methods for measurement of ultrasonic velocity are described in the following section.

FIGURE 1. Ultrasonic Testing Fixture

coupled to the surface. The fixture was designed for three-inch diameter samples, six to eight inches long, with flat ends. Clamping pressure for diametral measurements was supplied by the vertical screw in the center of the frame. Axial coupling pressure is supplied by the bolt at the upper right corner of the frame. Shims of appropriate thickness are used to accommodate samples of varying length.

The fixture proved convenient and provided repeatable measurements at fixed locations, which was the objective of the design. The only recommended improvement would be to use a screw device similar to the diametral unit for the axial-coupling force. This would minimize the time required to make measurements on samples of different lengths.

Six transducer assemblies are used for the measurements; four for diametral and two for axial measurements. The diametral units are spaced two inches apart (each 1 inch from the center of the bar). The upper two units are transmitters, while the lower units are receivers. Each of the diametral units contain one longitudinal-wave and two shear-wave transducer elements. The shear-wave elements are polarized parallel and perpendicular to the axis of the sample. The transducer elements are mounted on an aluminum coupling shoe or standoff. The face of the aluminum shoe is contoured to the sample to provide uniform coupling pressure. The transducer assemblies for axial velocity measurements, along the centerline of the sample, are similar to the diametral transducer assemblies, except that they contain only two elements. Figure 2 shows a view of a typical transducer assembly. These units contain one longitudinal-wave and one shear-wave element polarized parallel to the long axis of the transducer assembly, which is vertical when installed in the fixture. Measurements in other directions are achieved by rotating the sample.

The assembled measurement system is shown in Figure 3. The system consists of the fixture and transducer assemblies; a Metrotek MP 215 pulser and MR 101 receiver, Tektronics 184 Time Mark Generator, an eight position switch for selecting the eight pairs of transducer elements and a Hewlett Packard 1722A oscilloscope. The time mark generator provides system synchronization, as well as 5 μ sec time marks. Transit times through the sample are measured using the calibrated delay of the oscilloscope as measured relative to the

FIGURE 2. Typical Ultrasonic Transducer Assembly

FIGURE 3. Typical Ultrasonic Test System

nearest 5 μ sec time mark. This provides a time resolution of 0.01 μ sec which is approximately 0.01% of the transit time.

COUPLING

As stated previously, dry-pressure coupling of the ultrasonic energy to the sample is employed. This is the only suitable method for in-reactor measurements because a liquid couplant could contaminate the graphite. The efficiency of coupling increases with pressure up to an asymptotic limit past which further increase in pressure has little effect. Experiments, with the transducers used in our system, established this limit at approximately 3.5 MPa. This pressure produced negligible deformation of the sample. The aluminum coupling shoes used on these transducers are an improvement over the point contact units used previously;⁽²⁾ the shoes produce far less surface damage, a more controlled beam shape, and give better polarization of the shear waves.

The use of aluminum as a coupling material is not necessarily optimum for this application. Materials such as graphite, ceramics or high temperature plastics which have an acoustic impedance (product of velocity and density) closer to that of graphite could produce higher acoustic transmissions.

It should be noted (as can be seen in Figure 1) that the measurements were made with the sample in a plastic bag. This thin layer of material had negligible effect on the time measurement or the coupling efficiency. However, it simplified handling of the samples while protecting the sample from contamination.

MEASUREMENT AND CALIBRATION

Since these measurements were made over a period of one year, it was important to assure the long-term stability of the measurement system. This was accomplished by calibration of the oscilloscope and time mark generator to traceable primary standards. A reference graphite sample was also measured at the beginning and end of each workday. The same positions on the sample were measured each time. The maximum standard deviation of these measurements was 0.6%. No significant change was noted as a function of time.

Velocity measurements were performed on each sample before and after oxidation. The measurement fixture and diametral transducer assemblies provide two longitudinal-wave measurements which are spaced one inch either side of the center of the sample. Shear-wave velocities are measured in the same locations and provide two measurements with shear-wave polarization parallel, and two perpendicular, to the sample axis. The sample is then rotated 90° and the measurements are repeated. Axial measurements (along the sample centerline) are performed in the same manner.

The velocities tabulated in this report are the average values for each set of measurements. The diametral shear-wave velocities are averages of eight measurements and the longitudinal-wave velocities are averages of four measurements; in the axial direction, two measurements each were obtained for both the shear-wave and longitudinal-wave velocities.

To verify the applicability of the averaging technique, 64 diametral shear-wave measurements were made at four points along the standard sample (II-C-7, S1090) at eight rotational positions from 0 to 180° . These measurements demonstrated two factors of importance: 1) the average of measurements at one lengthwise position for the eight different rotations was within 0.1% of the average of 39 calibration measurements performed over a year; 2) the overall average of the 65 measurements was within 0.24% of the average of the measurements taken in the standard manner (two lengthwise positions at 0° and 90°). These measurements demonstrate that even though the point-to-point variation of velocity is high (standard deviation of 0.6%) the averaging techniques employed yield a representative measure of sample properties.

Based on the factors of instrumentation accuracy (long-term measurement repeatability and accuracy of the averaging techniques) the accuracy of the measurements is better than $\pm 1.0\%$ for all measurements.

Additional development work, which would be required before actual in-service inspection of the posts could be undertaken, is outlined in Appendix B.

RESULTS

GENERAL

All samples were oxidized at 875°C in a mixture of 90% CO₂, 10% CO. During the oxidation runs, the samples were in a vertical position with sufficient space between them (and sufficient gas flow) to insure that the oxidation conditions were quite uniform on all samples. Sacrificial slabs of graphite were used to shield both ends of the samples during oxidation; in addition, the samples were originally machined to a length of 8 inches, then reduced to 6 inches after oxidation, to insure that uniform oxidation occurred over the test sections. Additional information on the oxidation runs is given in Appendix C.

Rank correlation tests were made, using the post-oxidation data, to determine the degree of correlation between compressive strength and the other measured properties. Table 1 lists the minimum rank correlation coefficients which would indicate that, for the number of samples tested, there would be a 95% or a 99% probability that a correlation exists between the two properties. Table 2 shows the rank correlation coefficients which were obtained from ranking strength versus density and versus the diametral measurements of sonic velocity of the longitudinal and shear waves. Rank correlation tests were also made on the axial measurements of sonic velocity of the longitudinal and shear waves; however, it would be nearly impossible to make axial velocity measurements in-reactor. Moreover, the correlations of compressive strength versus axial velocities were not as good as the correlations of strength versus diametral shear-wave velocities.

TABLE 1. Parameters for Rank Correlation of Oxidized Samples

Number of Samples:	13	9	8	7	5
R(95) ^(a)	0.533	0.632	0.714	0.750	1.0
R(99) ^(b)	0.663	0.765	0.853	0.893	(c)

- (a) R(95) = Rank Correlation Coefficient for 95 percent probability that a correlation exists between the two properties.
 (b) R(99) = Same as (a), except: 99 percent.
 (c) Sample population is too small to determine R(99).

TABLE 2. Coefficients for Rank Correlation of Oxidized Samples

Graphite	Number of Samples	Property		
		ρ	V_L	V_S
Stackpole 2020	13	0.727	0.659	0.775
Stackpole 2020	9 ^(a)	0.704	0.550	0.667
Airco-Speer S1090	8	0.976	0.500	0.786 ^(b)
Union Carbide ATJ	8	0.881	0.452	0.738
Pure Carbon Co. P3W	5	1.000	1.000	1.000

- (a) Non-purified samples only
 (b) Sample III-A-2 not included (see text).

The diametral shear-wave velocities seem to be more consistently correlatable with compressive strength than do the diametral longitudinal-wave velocities; therefore, the tabulations and data plots are in terms of $S/\bar{S}_0$ versus $V_s/\bar{V}_s$; i.e., the plots correspond to the simple relationship:

$$S/\bar{S}_0 = (V_s/\bar{V}_s)^\beta \quad (2)$$

where: S = compressive strength of an oxidized sample,
 $\bar{S}_0$ = average strength of the unoxidized samples,
 V_s = velocity of the diametral shear-wave,
 $\bar{V}_s$ = average shear-wave velocity of the unoxidized samples, and
 β = a constant.

The plots adequately illustrate that a reasonable correlation exists between compressive strength and sonic velocities for all four grades of graphite; results of a slightly more sophisticated correlation are reported in the Discussion.

STACKPOLE 2020

Table 3 combines the results from our latest measurements (on samples H-1 to H-4) with previous results obtained on unoxidized samples from Log No. II of Stackpole grade 2020 graphite.

TABLE 3. Property Measurements on Unoxidized Stackpole 2020

Sample No.	Strength (MPa)	Density (Mg/m ³)	Diametral Measurements		Axial Measurements	
			V _ℓ (m/s)	V _s (m/s)	V _ℓ (m/s)	V _s (m/s)
II-B-2	77.83	1.806	2485	1617	2419	1587
II-H-2	74.96	1.789	2545	1632	2327	1572
II-D-2	73.86	1.791	2481	1609	2403	1586
II-H-4	73.55	1.788	2526	1625	2329	1586
II-H-3	73.25	1.790	2510	1634	2363	1591
II-E-4	72.66	1.784	2438	1590	2399	1587
II-E-2	72.60	1.788	2454	1599	2405	1586
II-D-4	72.26	1.782	2439	1595	2398	1576
II-B-4	72.06	1.800	2523	1605	2376	1569
II-H-1	68.09	1.785	2515	1634	2307	1570
Average:	73.11	1.790	2492	1614	2373	1581
+1σ:	2.45	0.007	42	17	39	8

Stackpole 2020 seems to be very homogeneous (except, possibly, for variations in impurity concentrations); however, there are variations in properties within a log, and between different logs. The variations of properties along the length of Log II are shown in Table 4 (see Appendix A, Figures A-1 and A-5, for the sampling scheme used on Log No. II). The average density and average diametral sonic velocities of the samples from section G of Log I fall midway between the averages obtained on the samples from sections B and C of Log II.

There is also a variation of properties with radial position in each section of the log; samples B-1, C-1, D-1, etc., are from the same quarter-section. Thus one can compare measurements on sample pairs (e.g., H-1 and H-3, or H-2 and H-4) to determine if there is a consistent difference between samples from different quarter-sections of the log. Of the 79 pairs of measurements which could be compared for sections B through H, in only 19 cases was value of the property higher for sample 4 than for sample 2 or

TABLE 4. Variation of Properties Within Log No. II, Stackpole 2020

Section No.	Density	Diametral Sonic Velocity	
		V_L	V_S
B	1.804	2493	1616
C	1.791	2470	1600
D	1.787	2463	1603
E	1.786	2453	1596
F	1.785	2385*	1578*
G	1.791	2504	1616
H	1.788	2524	1631

* After purification, (see text).

higher for sample 1 than for sample 3; moreover, in all except 6 cases where the value of a measurement for sample 4 or 1 exceeds the value for sample 2 or 3, respectively, this "reverse difference" is less than 0.5 percent. The only "reverse differences" which exceed 1% are the two sets of diametrically measured longitudinal-wave velocities for the "B" section, and two of the remaining four cases (with reverse difference between 0.5 and 1.0%) occur in the corresponding samples from section "C." The velocities are:

II-B-2 2484	II-B-3 2465
II-B-4 2523	II-B-1 2501
II-C-2 2466	II-C-3 2455
II-C-4 2487	II-C-1 2474

These, and other observations provide a strong basis for concluding that the variations in properties are not random; instead, the variations in properties follow a definite pattern which develops in the log during the manufacturing process.

Table 5 lists the pre-oxidation measurements on samples from Logs I and II; the data on all samples except F-1 to F-4 were from our prior investigation, reported in Reference 2. Samples F-1 to F-4 were purified at

POCO Graphite, Inc. Ash content was not measured on these samples; but, POCO claims typical purities of less than 20 ppm ash after purification (the typical ash content of Stackpole 2020 is about 1500 ppm). Evidently, some of the impurity atoms were acting as dislocation pinning centers, because the purification produced a small, but significant, reduction in the sonic velocities of these samples. There does not appear to be a significant difference between non-purified samples from Logs I and II; and rank correlation tests of the results indicate that inclusion of the results from Log I increases the correlation (of strength versus diametral shear-wave velocity) above the probability level obtained when only the results from samples of Log II were considered. Therefore they were treated as a single lot of samples with an average strength of 73.11 MPa and an average diametral shear-wave velocity of 1614 m/s. The purified (II-F-) samples were assumed to have the same average strength as the non-purified samples, but to have an average diametral shear-wave velocity of 1578 m/s, as listed in Table 5.

TABLE 5. Pre-Oxidation Measurements on Stackpole 2020

Sample No.	Density (Mg/m ³)	Diametral Velocities		Axial Velocities		Notes
		V _L (m/s)	V _S (m/s)	V _L (m/s)	V _S (m/s)	
I-G-4	1.804	2510	1611	2411	1597	Log No. I
I-G-2	1.809	2485	1618	2418	1592	
I-G-3	1.810	2482	1617	2404	1594	
I-G-1	1.798	2477	1597	2411	1580	
Average:	1.805	2488	1611	2411	1591	
+1σ:	+0.006	+15	+10	+6	+7	
II-D-1	1.787	2467	1603	2400	1584	Log No. II Non-purified
II-D-3	1.789	2466	1606	2413	1585	
II-C-2	1.789	2455	1604	2399	1583	
II-C-3	1.789	2455	1604	2399	1583	
II-C-4	1.780	2487	1593	2369	1571	
Average:	1.789	2468	1602	2399	1580	
+1σ:	+0.007	+12	+5	+18	+10	
II-F-1	1.781	2356	1563	2309	1548	Log No. II Purified
II-F-2	1.786	2400	1579	2309	1549	
II-F-3	1.785	2383	1588	2325	1544	
II-F-4	1.789	2399	1582	2329	1567	
Average:	1.785	2385	1578	2318	1552	
+1σ:	+0.003	+21	+11	+11	+11	

Table 6 lists the properties measured after oxidation, and Figure 4 shows the correlations between relative strength and relative diametral shear-wave velocity. Because of the limited number of samples, it is not clear whether the difference in the two correlations is due to the purification process, or to other differences; however, it appears most likely that it is due to the removal of the impurities. The slopes of the two least square lines corresponds to $\beta = 2.19$ for the non-purified samples and $\beta = 4.10$ for the purified samples.

TABLE 6. Property Measurements on Oxidized Stackpole 2020

Sample No.	Strength (MPa)	Density (Mg/m ³)	Diametral Measurements				Axial Measurements	
			V_L (m/s)	V_S (m/s)	S/S_0	$V_S/V_S^{(a)}$	V_L (m/s)	V_S (m/s)
II-F-1	64.98	1.737	2314	1529	0.889	0.969(b)	2263	1520
I-G-4	60.34	1.711	2212	1433	0.825	0.888	2123	1421
II-D-1	59.74	1.691	2219	1491	0.817	0.924	2280	1525
II-F-2	59.44	1.715	2307	1522	0.813	0.965(b)	2240	1508
II-D-3	59.23	1.676	2221	1482	0.810	0.918	2309	1552
II-F-4	57.74	1.661	2191	1457	0.790	0.923(b)	2181	1470
II-F-3	57.35	1.701	2262	1504	0.784	0.953(b)	2235	1518
II-C-2	47.26	1.663	2195	1426	0.783	0.884	2351	1492
II-C-3	56.28	1.666	2194	1443	0.770	0.895	2344	1519
I-G-2	55.02	1.663	2115	1384	0.753	0.857	2080	1374
II-C-4	53.62	1.685	2234	1475	0.733	0.914	2292	1491
I-G-3	53.36	1.665	2138	1369	0.730	0.848	2097	1403
I-G-1	47.70	1.624	2042	1337	0.652	0.828	2027	1342
Average:	55.84	1.672	2174	1427	0.764	0.884	2211	1458

(a) $\bar{V}_S = 1614$ m/s

(b) $\bar{V}_S = 1578$ m/s

We expected to see a large reduction in oxidation rate for the purified samples; but, the rates were approximately the same as we had previously observed for non-purified samples. The only difference which we observed was that the surfaces of the purified samples appeared to have been homogeneously oxidized, whereas the surfaces of the non-purified samples normally contained "wormhole" regions (areas where oxidation opened large pores). Appendix C contains a more detailed comparison of the relative oxidation rates.

FIGURE 4. Strength versus Velocity for Stackpole 2020

AIRCO-SPEER S1090

Table 7 lists the property measurements on unoxidized Airco-Speer grade S1090 graphite. Table 8 lists the pre-oxidation measurements made on Airco-Speer S1090 samples after purification by POCO Graphite, Inc.; the reduction in the sonic velocities, due to removal of impurities, is much larger than it was for the Stackpole 2020 samples. Table 9 lists the results obtained on the oxidized samples and Figure 5 shows the correlation of strength versus shear-wave velocity. The slope corresponds to $\beta = 2.51$, with $\bar{V}_s = 1632$ m/s for samples III-A-1 and III-A-12, which were not purified prior to oxidation and $\bar{V}_s = 1569$ m/s for the purified samples.

The velocities of the diametral shear-wave and the axial longitudinal wave of sample III-A-11 were greater after oxidation than they were prior to oxidation. Therefore, V_s for this sample was not used in the correlation; however, the point is shown in Figure 5.

TABLE 7. Property Measurements on Unoxidized Airco-Speer S1090

Sample No.	Strength (MPa)	Density (Mg/m ³)	Diametral Measurements		Axial Measurements	
			V_L (m/s)	V_S (m/s)	V_L (m/s)	V_S (m/s)
III-B-10	62.60	1.815	2508	1672	2361	1623
III-C-11	58.11	1.779	2494	1640	2226	1555
III-C-9	57.25	1.758	2465	1625	2436	1557
III-C-2	57.23	1.792	2457	1639	2317	1577
III-B-8	56.54	1.757	2490	1620	2149	1553
III-C-4	56.45	1.761	2496	1618	2248	1577
III-B-5	55.19	1.757	2468	1617	2186	1569
III-B-3	55.01	1.779	2463	1627	2248	1561
Average:	57.30	1.775	2480	1632	2271	1572
$\pm 1\sigma$:	± 2.38	± 0.021	± 19	± 18	± 95	± 23

Purification appeared to have very little effect on the oxidation rates; this is in agreement with the observations on Stackpole 2020 samples (see Appendix C).

TABLE 8. Pre-oxidation Measurements on Purified Airco-Speer S1090

Purified Sample No.	Density (Mg/m ³)	Diametral Measurements		Axial Measurements	
		V_L (m/s)	V_S (m/s)	V_L (m/s)	V_S (m/s)
III-A-10	1.815	2380	1586	2219	1539
III-A-2	1.798	2325	1553	2312	1579
III-A-11	1.787	2344	1569	2209	1533
III-A-8	1.746	2374	1561	2078	1487
III-A-3	1.786	2338	1573	2159	1528
III-A-9	1.755	2371	1555	2107	1497
III-A-4	1.758	2385	1589	2147	1509
III-A-5	<u>1.760</u>	<u>2373</u>	<u>1569</u>	<u>2186</u>	<u>1510</u>
Average:	1.776	2361	1569	2177	1523
<u>+1σ</u> :	<u>+0.024</u>	<u>+22</u>	<u>+13</u>	<u>+73</u>	<u>+29</u>

TABLE 9. Property Measurements on Oxidized Airco-Speer S1090

Purified Sample No.	Strength (MPa)	Density (Mg/m ³)	Diametral Measurements				Axial Measurements	
			V_L (m/s)	V_S (m/s)	S $\overline{S}_0$	V_S $\overline{V}_S$ (a)	V_L (m/s)	V_S (m/s)
III-A-10	58.43	1.804	2322	1564	1.020	0.997	2183	1618 ^(b)
III-A-2	57.05	1.772	2282	1542	0.996	0.983	2259	1530
III-A-11	56.57	1.754	2297	1588 ^(b)	0.987	1.012 ^(b)	2225 ^(b)	1525
III-A-8	52.48	1.735	2298	1527	0.916	0.973	2007	1456
III-A-3	49.93	1.721	2215	1473	0.871	0.939	2137	1466
III-A-9	49.75	1.722	2304	1505	0.868	0.959	1998	1439
III-A-4	48.85	1.698	2284	1489	0.853	0.949	2045	1435
III-A-5	<u>44.09</u>	<u>1.669</u>	<u>2212</u>	<u>1479</u>	<u>0.769</u>	<u>0.943</u>	<u>2064</u>	<u>1443</u>
Average:	52.14	1.734	2277	1511	0.910	0.963	2099	1471
III-A-12	52.57	1.767	2273	1571	0.917	0.962 ^(c)	2406	1650
III-A-1	48.87	1.780	2183	1577	0.853	0.966 ^(c)	2259	1571

(a) $\overline{V}_S = 1569$ m/s

(b) not used in average

(c) $\overline{V}_S = 1632$ m/s

FIGURE 5. Strength versus Velocity for Airco-Speer S1090

UNION CARBIDE ATJ

Table 10 lists the results obtained on unoxidized samples of Log V (Union Carbide ATJ); the results obtained on the oxidized samples are listed in Table 11 and are shown graphically in Figure 6. The slope of the line corresponds to $\beta = 4.11$, much higher than observed for any of the other graphites, except the purified samples of Stackpole 2020. This slope would make the correlation very dependent on oxidation profile and on the geometry of the graphite component (see Appendix D).

The samples of Union Carbide ATJ oxidized much more rapidly than did the samples of Stackpole 2020 or Airco-Speer S1090; moreover, the sample surfaces showed extensive, fairly homogeneous "wormholing", which indicates that impurities were catalyzing the oxidation reaction.

TABLE 10. Property Measurements on Unoxidized Union Carbide ATJ

Sample No.	Strength (MPa)	Density (Mg/m ³)	Diametral Measurements		Axial Measurements	
			V_L (m/s)	V_S (m/s)	V_L (m/s)	V_S (m/s)
V-B-4	63.64	1.713	2511	1600	2207	1547
V-B-7	63.16	1.691	2495	1598	2190	1548
V-B-1	63.15	1.702	2358	1583	2180	1525
V-B-6	62.37	1.695	2481	1600	2196	1536
V-B-8	62.10	1.701	2516	1605	2175	1534
V-B-2	61.40	1.693	2497	1607	2163	1528
V-B-3	60.25	1.687	2494	1595	2200	1526
V-B-5	<u>60.18</u>	<u>1.688</u>	<u>2472</u>	<u>1588</u>	<u>2227</u>	<u>1543</u>
Average:	62.03	1.696	2478	1597	2192	1536
<u>+1σ</u>	<u>+1.32</u>	<u>+0.009</u>	<u>+51</u>	<u>+8</u>	<u>+20</u>	<u>+9</u>

TABLE 11. Property Measurements on Oxidized Union Carbide ATJ

Sample No.	Strength (MPa)	Density (Mg/m ³)	Diametral Measurements				Axial Measurements	
			V_L (m/s)	V_S (m/s)	S/S_0	$V_S/\bar{V}_S$	V_L (m/s)	V_S (m/s)
V-A-1	47.60	1.657	2280	1475	0.767	0.924	2074	1474
V-A-2	43.40	1.659	2154	1446	0.700	0.905	2072	1418
V-A-3	42.92	1.629	2232	1463	0.692	0.916	2107	1498
V-A-8	42.92	1.626	2186	1431	0.692	0.896	2063	1440
V-A-7	40.00	1.585	2109	1383	0.645	0.866	1979	1400
V-A-4	34.72	1.555	2121	1389	0.560	0.870	2081	1476
V-A-6	31.80	1.557	2144	1396	0.513	0.874	2074	1468
V-A-5	28.29	1.579	2205	1424	0.456	0.892	2011	1435
Average:	38.96	1.606	2179	1426	0.628	0.893	2058	1451

PURE CARBON CO P3W

Table 12 lists the property measurements obtained on samples from Log VI (Pure Carbon Co. graphite grade P3W). Property measurements on the oxidized samples are listed in Table 13 and shown in Figure 7. The slope corresponds to $\beta = 2.81$, indicating a significant dependency of the correlation on oxidation profile and component geometry.

FIGURE 6. Strength versus Velocity for Union Carbide ATJ

TABLE 12. Property Measurements on Unoxidized Pure Carbon Co. P3W

Sample No.	Strength (MPa)	Density (Mg/m ³)	Diametral Measurements		Axial Measurements	
			V_L (m/s)	V_S (m/s)	V_L (m/s)	V_S (m/s)
VI-D-1	58.74	1.706	2403	1565	2150	1483
VI-D-4	58.56	1.702	2352	1561	2221	1518
VI-B-1	55.99	1.693	2336	1550	2144	1488
VI-O-3	55.98	1.700	2328	1645	2219	1507
VI-B-4	55.43	1.694	2358	1537	2190	1502
VI-O-2	55.32	1.705	2363	1559	2191	1515
VI-B-2	51.04	1.691	2343	1550	2154	1481
VI-B-3	50.92	1.691	2308	1507	2184	1495
Average:	55.25	1.698	2349	1547	2182	1499
$\pm 1 \sigma =$	± 2.95	± 0.006	± 28	± 18	± 30	± 14

TABLE 13. Property Measurements on Oxidized Pure Carbon Co. P3W

Sample No.	Strength (MPa)	Density (Mg/m ³)	Diametral Measurements				Axial Measurements	
			V_L (m/s)	V_S (m/s)	S S_0	V_S V_0	V_L (m/s)	V_S (m/s)
VI-A-3	52.31	1.672	2256	1507	0.947	0.974	2078	1490
VI-A-1	48.63	1.638	2176	1428	0.880	0.923	2107	1433
VI-A-4	43.61	1.587	2120	1397	0.789	0.903	2031	1405
VI-C-2	29.85	1.473	1972	1346	0.540	0.870	2186	1483
VI-A-2	24.29	1.471	1877	1203	0.440	0.778	1731	1243
Average:	39.74	1.569	2080	1376	0.719	0.889	2027	1411

FIGURE 7. Strength versus Velocity for Pure Carbon Co. P3W

Eight samples of P3W were oxidized, but only five are listed in Table 13, or shown on Figure 7; the other three samples were so porous that we were unable to obtain measurements of the sonic velocities. However, the oxidation was very homogeneous with no sign of "wormholing". Figure 8 shows sample VI-C-3, the most heavily-oxidized sample. Density of this sample was only 1.030 Mg/m^3 (61 percent of the average density measured on the unoxidized samples, see Table 12); however, the appearance of the sample surface had not been changed significantly by this large density change.

Because we couldn't obtain velocity measurements on these samples, we didn't proceed with the strength measurements. However, it can be seen (Figure 8) that the sample tended to crumble and spall during machining.

FIGURE 8. Sample VI-C-3 after 39 Percent Oxidation

DISCUSSION

It can be seen from Figures 4 through 7 that a fairly good correlation exists between compressive strength and the shear-wave velocity measured diametrically. Undoubtedly, a much better correlation could be developed using more refined analytical methods to determine the pre-oxidation strength of the samples, and correlate the change in strength with change in sonic velocity for each sample, as we did in the previous study.⁽²⁾ However, the primary goal was to show that the strength versus sonic velocity correlation method is applicable to other types of graphites, not just to Stackpole 2020; the results clearly indicate that the method is applicable to a wide range of graphites. Table 14 lists the exponents β , β' and β'' calculated using the simple relationship:

$$S/\bar{S}_0 = (V_S/\bar{V}_S)^\beta = (V_L/\bar{V}_L)^{\beta'} = (\rho/\bar{\rho}_0)^{\beta''} \quad (3)$$

where: S is the compressive strength of an oxidized sample,
 V_L is its diametral longitudinal-wave velocity,
 V_S is its diametral shear-wave velocity, and
 ρ is its density; $\bar{S}_0$, $\bar{V}_L$, $\bar{V}_S$ and $\bar{\rho}_0$ are the corresponding average properties measured on unoxidized samples.

TABLE 14. Exponents Calculated Using Equation 3

Graphite		β	β'	β''
Stackpole 2020	Non-Purified	2.19	1.97	3.95
Stackpole 2020	Purified	4.10	3.97	4.05
Airco-Speer S1090	Purified	2.51	2.60	4.08
Union Carbide ATJ		4.11	3.62	8.52
Pure Carbon Co. P3W		2.81	2.71	4.16

Because of the higher values of β , compared with the value for the non-purified Stackpole 2020 samples, oxidation-depth profiles would be required in order to use the correlations to monitor the strength of core support posts. This is especially true for the Union Carbide ATJ, which is the graphite used for the FSVR core support posts. A brief discussion of the effect of the oxidation depth profile for different geometries is given in Appendix D.

In the simple analytic method, used to obtain the correlations shown in Figures 4 through 7 and the exponents listed in Table 14, the least squares lines were forced through the origins and it was necessary to make several assumptions in selection of the values used for the origins.

A somewhat more sophisticated approach has been used to develop correlations which are not constrained at the origins and, thus, avoid the question of selecting the origins. Assume a correlation of the form

$$S = K P^{\alpha} \quad (4)$$

where:

S is the compressive strength,

P is a measurable property (density or sonic velocity), and

K and α are constants for both oxidized and non-oxidized samples.

Equation 4 can then be rewritten in the form

$$\ln S = \ln S_r + \alpha \ln (P/P_r) \quad (5)$$

where the S_r and P_r are reference values of the strength and property.

A least squares analysis, using an arbitrary value for P_r , yields S_r and α without the constraints which were used in the preceeding simple analytic method.

We have selected the reference values of density and sonic velocities as the measured average values for the sets of non-oxidized samples and obtained the correlations shown in Table 15.

TABLE 15. Calculated Compressive Strengths and Correlation Coefficients

Graphite	Measured $S_o \pm 1\sigma$	Calculated From:					
		Density $S_r \pm 1\sigma$	α	Diametral Velocities			
				Longitudinal		Shear	
				$S_r \pm 1\sigma$	α	$S_r \pm 1\sigma$	α
	(MPa)	(MPa)		(MPa)		(MPa)	
Stackpole 2020 (Non-purified)		73.1 $\pm$ 2.5	4.30	72.7 $\pm$ 3.7	2.00	72.3 $\pm$ 3.9	2.11
Stackpole 2020 (Bar II only)	73.1 $\pm$ 2.5 ^(a)	73.0 $\pm$ 2.7	4.17	72.6 $\pm$ 3.7	2.08	72.3 $\pm$ 3.7	2.50
Stackpole 2020 (all samples)		73.0 $\pm$ 2.5	4.20	72.6 $\pm$ 3.5	1.99	71.8 $\pm$ 3.8	2.12
Airco-Speer S1090(Non-purified)	57.3 $\pm$ 2.4	55.9 $\pm$ 3.2	2.43	57.3 $\pm$ 1.8	1.21	57.2 $\pm$ 1.3	3.30
Airco-Speer S1090(All samples)		56.1 $\pm$ 2.5	3.42	57.8 $\pm$ 3.0	1.32	57.6 $\pm$ 2.7	1.77
Union Carbide ATJ	62.0 $\pm$ 1.3	60.4 $\pm$ 3.5	7.78	61.1 $\pm$ 5.2	3.47	62.0 $\pm$ 3.7	4.22
Pure Carbon Co. P3W	55.3 $\pm$ 3.0	55.8 $\pm$ 2.4	4.94	56.4 $\pm$ 3.0	3.44	55.8 $\pm$ 3.0	3.34

(a) Non-purified samples only.

Similar correlations were also obtained for the sonic velocities measured axially and for the corresponding elastic and shear moduli. In general, standard deviations for correlations with the axial measurements were approximately twice as large as for the diametral measurements; the correlations with the moduli generally had slightly lower standard deviations than did those for the velocities and the derived α -coefficients were much smaller (due to the inclusion of the density dependence in the moduli).

Two important observations from the results presented in Table 15 are 1) the calculated strengths are not significantly more uncertain than are the measured strengths of the non-oxidized samples, and 2) purification of the samples may have reduced the strengths in proportion to the reduction in their moduli. This last observation implies that the results of the simple correlation method may be somewhat in error and that the method used to obtain the correlation coefficients shown in Table 15 is to be preferred for future analyses.

Similar correlations were also obtained for the same velocity measures
across and for the corresponding elastic and shear moduli. In general, stan-
dard deviations for correlations with the axial measurements were approximately
twice as large as for the lateral measurements. The correlations with the
axial measurements had slightly lower standard deviations than with the lateral
measurements and the derived coefficients were much smaller (due to the in-
crease of the density dependence in the model).

Two important observations from the results presented in Table 1 are
if the calculated strengths are not significantly more uncertain than are the
measured strengths of the low-oxidized samples, and (2) prediction of the
strengths may have reduced the strength in prediction to the reduction in their
moduli. The last observation implies that the results of the simple correla-
tion method may be somewhat in error and that the method used to obtain the
correlation coefficients shown in Table 1 is to be preferred for future

analysis.

CONCLUSIONS

The primary conclusions from these results can be summarized as follows:

- 1) These results demonstrate that sonic velocity can be used to monitor the strength of oxidized samples for a wide range of fine-grained graphites.
- 2) The strengths calculated for the oxidized samples have about the same uncertainty as the measured strengths of unoxidized samples.
- 3) Purification of samples may reduce their strength in proportion to the change in elastic moduli.
- 4) There appears to be a significant difference between the different graphites in their rate of change of strength with change in sonic velocity (a factor of 2x for the exponent, β , was observed for the graphites investigated in this study).
- 5) For some grades of graphite, and for other than cylindrical geometries, measurement of the oxidation depth-profile will also be required to predict the load-bearing ability of a component.
- 6) Purification of a graphite can reduce the sonic velocity, but may not lead to a large reduction in oxidation rate.
- 7) There is a large difference between the relative rates of oxidation as a function of burn-off for the different graphites.

CONCLUSIONS

- The primary conclusions from these results can be summarized as follows:
- 1) These results demonstrate that sonic velocity can be used to monitor the strength of oxidized samples for a wide range of time-graded oxidation.
 - 2) The strengths calculated for the oxidized samples have about the same accuracy as the measured strengths of unoxidized samples.
 - 3) Partial oxidation of samples may reduce their strength in proportion to the change in elastic modulus.
 - 4) There appears to be a significant difference between the different grades of test in their rate of change of strength with change in wave velocity (a factor of 2x for the oxidized, 2x was observed for the graphitic - investigated in this study).
 - 5) For some grades of graphite, and for other than dry, indirect measurement of the oxidation depth-profile will also be required to pre-dict the load-bearing ability of a component.
 - 6) Partial oxidation of a graphite can reduce the sonic velocity, but may not lead to a large reduction in oxidation rate.
 - 7) There is a large difference between the relative rates of oxidation as a function of pore size for the different grades.

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APPENDIX A

SAMPLE SELECTION AND NUMBER DESIGNATION

Figures A-1 through A-4 show perspective views of the graphite logs. A line was scribed lengthwise of each log; this line was then used as a reference to locate each sample's original position in the log.

Samples were labeled with 1) a Roman numeral assigned to the logs in the sequence of their arrival date; 2) a letter to designate the sequential slab within the log; and 3) the sample position within the slab. The general sample positions are indicated on the Figures A-1 to A-4 and are shown in more detail in Figures A-5 through A-7.

FIGURE A-1. Sampling Scheme for Log II (Stackpole 2020)

FIGURE A-2. Sampling Scheme for Log III (Airco-Speer S1090)

FIGURE A-3. Sampling Scheme for Log V (Union Carbide ATJ)

FIGURE A-4. Sampling Scheme for Log VI (Pure Carbon Co. P3W)

FIGURE A-5. Sampling Positions Within Log II (Stackpole 2020) and Log VI (Pure Carbon Co. P3W)

FIGURE A-6. Sampling Positions Within Log III (Airco-Speer S1090)

FIGURE A-7. Sampling Positions Within Log V (Union Carbide ATJ)

FIGURE A-1 Sampling Positions Within Lot V (Union Carbide 807)

APPENDIX B

FURTHER DEVELOPMENTAL WORK

In order to perform these measurements in-reactor, three areas must be considered; they are 1) access, 2) the mechanical delivery or positioning system, and 3) the transducer assembly. It is anticipated, that for the inspection of core support posts, an access port of approximately six inches diameter would be required in the core support block.

The positioning system would be located on the upper surface of the block. An articulated arm would be inserted through the access port and then raised to 90° . The articulated arm would need to rotate 0 to 360° , elevate in the vertical axis, and extend to reach the post to be inspected.

A self-aligning transducer assembly mounted on the arm would clamp on to the post. The diameter of the post as well as the longitudinal-wave and shear-wave time-of-flight through the post would be measured. Measurements would be performed at three to five different elevations for comparisons and averaging. The transducer assemblies which have been developed are suitable for in-reactor measurements up to 500°F . Materials are available for higher temperature service if necessary.

Measurements on core support blocks would be performed between adjacent coolant channels by the through-transmission technique. The design of the testing fixture would depend on the configuration of the core support blocks to be tested.

APPENDIX B

EXPERIMENTAL APPARATUS

In order to perform these measurements, the apparatus, shown in Figure 1, was constructed. It consists of three main parts: (1) the mechanical delivery and positioning system, and (2) the transport assembly. It is anticipated that for the installation of some support post, an access port of approximately six inches diameter would be required in the support block.

The positioning system would be located on the upper surface of the block. An access port would be inserted through the access port and then raised to 90°. The access port would need to rotate 0 to 360° clockwise in the vertical axis, and extend to reach the post to be installed.

A self-aligning mechanism, assembly mounted on the end would allow the post. The diameter of the post as well as the longitudinal-wave and transverse-wave frequencies through the post would be measured. Measurements would be performed at three to five different frequencies for comparison and averaging. The transport assembly which have been designed are suitable for the reactor measurements up to 100 ft. diameters are available for higher frequency measurements if necessary.

Measurements on core support blocks would be performed between adjacent blocks and channels by the above-mentioned technique. The design of the testing fixture would depend on the configuration of the core support blocks to be tested.

APPENDIX C

OXIDATION RATES

A total of seven oxidation runs were made with a 90% CO₂-10% CO mixture at 875°C. No attempt was made to obtain absolute values for the oxidation rates; however, comparison of the relative oxidation rates can be made between samples which were oxidized in the same run(s). Table C1 lists samples which can be compared directly.

There are several things about the relative oxidation rates which should be noted; these include:

- 1) There is a factor of about 3 to 5 between the highest and lowest oxidation rates for samples from the same bar.
- 2) Purification seems to have little effect on the oxidation rates of Log II or Log III.
- 3) The oxidation rates are lowest for samples from Log III (purified Airco-Speer S1090); the other graphites, ranked in increasing oxidation rate, are Log II (purified Stackpole 2020), Log V (Union Carbide ATJ) and Log VI (Pure Carbon Co. P3W).
- 4) There is a factor of about 20 or 30 between the average oxidation rates of Log III and the rates of Log VI.*
- 5) All samples from Log II and sample III-A-2 lost about the same amount of weight during Run 2 as they had during Run 1; however, samples III-A-3, -4, and -5 lost about 4 times as much weight in Run 2 as in Run 1.
- 6) Samples from Logs II, III, and V lost about the same amount of weight in Run 6 as they lost in Run 5; however, samples VI-C-4 lost approximately 14 times as much weight, and samples VI-C-1 and -3 lost approximately 25 times as much weight in Run 6 as they lost in Run 5.

*Note: It appears likely that the pre-oxidation weight of sample III-A-8 was incorrectly listed; we recorded an apparent weight gain during Run 5. If the pre-oxidation weight is assumed to be in error by 10 mg, the oxidation rates during Runs 5 and 6 are comparable to those recorded for sample III-A-9. The initial density, listed in Table C-1, would then be 1.757 Mg/m³ and the fractional change would be 1.3 percent after Run 6.

We did not attempt to explain the observations mentioned above, but they do serve as an indication of the variability in rates and behavior that one can find between different samples of one grade of graphite, and between samples from different grades of graphite.

TABLE C-1. Relative Rates of Oxidation

		DENSITY			
	Sample Number	Initial (Mg/m ³)	Final (Mg/m ³)	Fractional Change (%)	Notes
<u>Runs 1 and 2</u>					
	II F-1	1.781	1.739	2.4	Purified
	F-2	1.786	1.715	4.0	Purified
	III A-2	1.798	1.772	1.4	Purified
	A-4	1.758	1.698	3.4	Purified
<u>Run 3</u>					
	VI A-1	1.695	1.638	3.4	
	A-3	1.692	1.672	1.2	
	A-4	1.695	1.587	6.4	
<u>Run 4</u>					
	V A-1	1.715	1.680	2.0	
	A-7	1.690	1.585	6.2	
	A-8	1.713	1.626	5.1	
<u>Runs 5 and 6</u>					
	III A-8	1.746(a)	1.735	0.6(a)	Purified
	VI C-1	1.694	1.109	34.5	
	C-3	1.693	1.030	39.2	
	C-4	1.692	1.209	28.5	
<u>Run 6</u>					
	V A-5	1.696	1.579	6.9	
	A-6	1.697	1.557	8.2	
<u>Run 7</u>					
	III A-1	1.808	1.780	1.5	Not Purified
	A-12	1.788	1.767	1.2	Not Purified
	A-10	1.815	1.804	0.6	Purified
	A-11	1.787	1.754	1.8	Purified
	V A-3	1.689	1.638	3.0	
	A-4	1.722	1.587	7.8	
	VI C-2	1.698	1.473	13.3	

(a) see footnote on page C-1.

APPENDIX D

OXIDATION PROFILE

A very simple model will suffice to show the importance of knowing the oxidation profile.

- 1) Consider three sample configurations: a) a solid cylinder, b) a slab, and c) a hollow cylinder. Let the path traversed by the sonic waves, (i.e., the diameter, thickness, and wall thickness, respectively) be of equal length for all samples.

If the samples are homogeneously oxidized, the measured strengths and measured sonic velocities will be equal for all sample configurations.

- 2) Let the samples be oxidized in such a manner that the graphite along one-half of the path length is unchanged, but that (for simplicity) the other one-half of the path length is composed of material with essentially zero strength.

Measured velocities will still be equal in all configurations; however:

- a) The measured strength of the solid cylinder would then be reduced to 25% of the original strength;
- b) Measured strength of the slab would be 50% of the original; and
- c) Measured strength of the hollow cylinder would range between 25% and 75% of the original strength; the actual percentage will depend on the diameter-to-wall thickness ratio and whether the oxidation was from the outside or the inside surface.

APPENDIX B

VALIDATION PROJECT

A very simple model will suffice to show the importance of knowing the oxidation profile.

- 1) Consider three sample configurations: a) a solid cylinder, b) a slab, and c) a hollow cylinder. Let the path traversed by the sonic waves, (i.e., the diameter, thickness, and wall thickness, respectively) be of equal length for all samples.

If the samples are homogeneously oxidized, the measured strength and measured sonic velocities will be equal for all sample configurations.

- 2) Let the samples be oxidized in such a manner that the gradient along one-half of the path length is unchanged, but that (for simplicity) the other one-half of the path length is composed of material with essentially zero strength.

Measured velocities will still be equal in all configurations; however:

- a) The measured strength of the solid cylinder would then be reduced to 50% of the original strength;

- b) Measured strength of the slab would be 50% of the original and

- c) Measured strength of the hollow cylinder would range between 50% and 75% of the original strength. The actual percentage will depend on the

diameter-to-wall thickness ratio and whether the oxidation was from

the outside or the inside surface.

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