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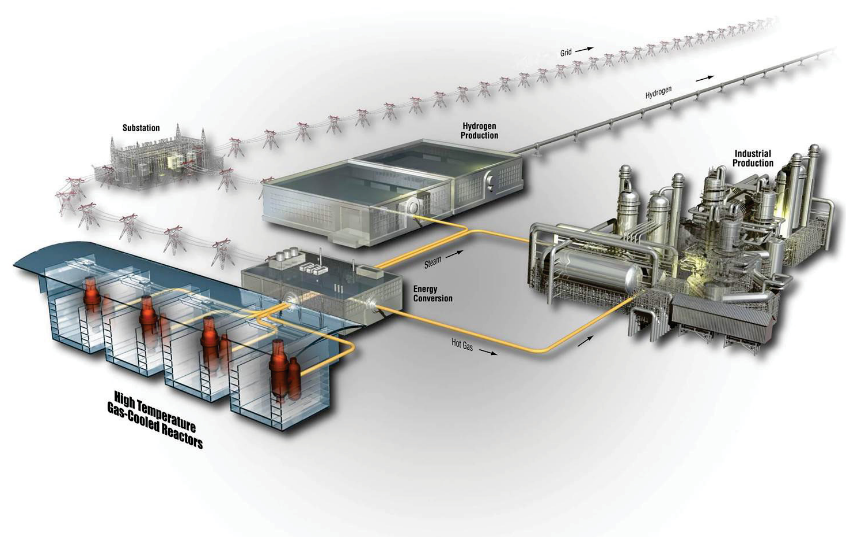
Data Report on the Newest Batch of PCEA Graphite for the VHTR Baseline Graphite Characterization Program

M. C. Carroll, D. L. Cottle,
and D. T. Rohrbaugh

August 2016



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Data Report on the Newest Batch of PCEA Graphite for the VHTR Baseline Graphite Characterization Program

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ABSTRACT

This is a summary of the data assembled following completion of physical and mechanical properties testing on the first billet from the newest batch of PCEA graphite from GrafTech. The report largely details a comparison of mechanical and physical properties from the first billet of extruded PCEA nuclear-grade graphite with the property data from this billet from the new batch. Testing has largely been completed on three of the billets from the original batch of PCEA, with data distributions for those billets exhibiting a much wider range of values when compared to the property distributions from other grades. A higher propensity for extremely low values or specimens that broke while machining or handling was also characteristic of the billets from the first batch, owing to unusually large fissures or disparate flaws in the billets in an as-manufactured state.

Coordination with GrafTech prior to placing the order for a second batch of PCEA included discussions on these large disparate flaws and how to prevent them during the manufacturing process. This report provides a comparison of the observed data distributions from properties measured in the first billet from the new batch of PCEA with those observed in the original batch, in order that an evaluation of tighter control of the manufacturing process and the outcome of these controls on final properties can be ascertained.

Additionally, this billet of PCEA is the first billet to formally include measurements from two alternate test techniques that will become part of the Baseline Graphite Characterization database: the three-point bend test on sub-sized cylinders and the Brazilian disc splitting tensile strength test. As the program moves forward, property distributions from these two tests will be obtained with specimen geometries that match specimen geometries being used in the irradiated Advanced Graphite Creep (AGC) program. This will allow a more thorough evaluation of both the utility of the test and expected variability in properties when using those approaches on the constrained geometries of specimens irradiated in the Advanced Test Reactor as part of the AGC experiment.

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Table 1. The listed Weibull parameter values for each of the properties plotted via two-parameter Weibull cumulative distribution functions. Units apply to the scale parameter (characteristic value). 15

ACRONYMS

AGC	Advanced Graphite Creep experiment
ART	Advanced Reactor Technologies
ASTM	Brand name for ASTM International, formerly known as the American Society for Testing and Materials
INL	Idaho National Laboratory
NDMAS	Nuclear Data Management and Analysis System
PCEA	Specific grade designation for medium-grained extruded grade of graphite produced by GrafTech
VHTR	Very High Temperature Reactor

Data Report on the Newest Batch of PCEA Graphite for the VHTR Baseline Graphite Characterization Program

1. INTRODUCTION

Detailed interim property comparisons between nuclear graphite grades being comprehensively evaluated as part of the Very High Temperature Reactor (VHTR) Baseline Graphite Characterization program indicated that the lone extruded grade under evaluation, PCEA from GrafTech, had consistently wider spreads in respective data distributions across the range of properties being quantified. Additionally, there existed a higher propensity for low or "zero" mechanical property values owing to large fissures in the as-manufactured billets that resulted in test coupons or specimens breaking prior to completing the machining process in preparation for testing per ASTM International standards. For specimens that reached the standardized test phase and provided property data to the database, the example pooled data sets[1] for tensile strength (Figure 1) and flexural strength (Figure 2) demonstrate the wider range of values, which is reflected by the lower slope in the Weibull cumulative density distribution.

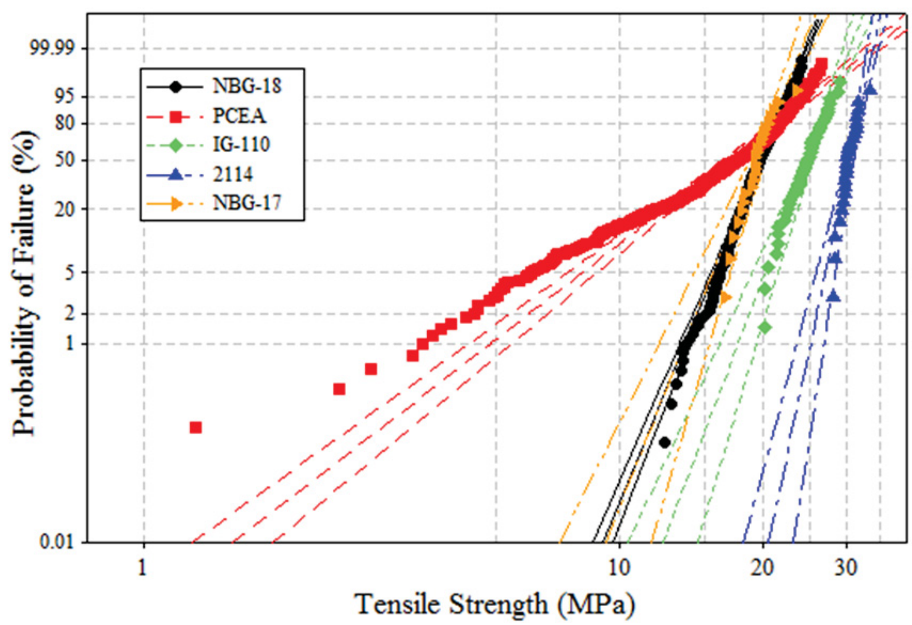


Figure 1. Tensile strength distributions for different candidate grades of graphite evaluated as part of the VHTR program. The extruded PCEA graphite exhibits the widest range of values (variability) along with the largest number of data points at low values. The dotted lines represent the 95% confidence bands for a given distribution.

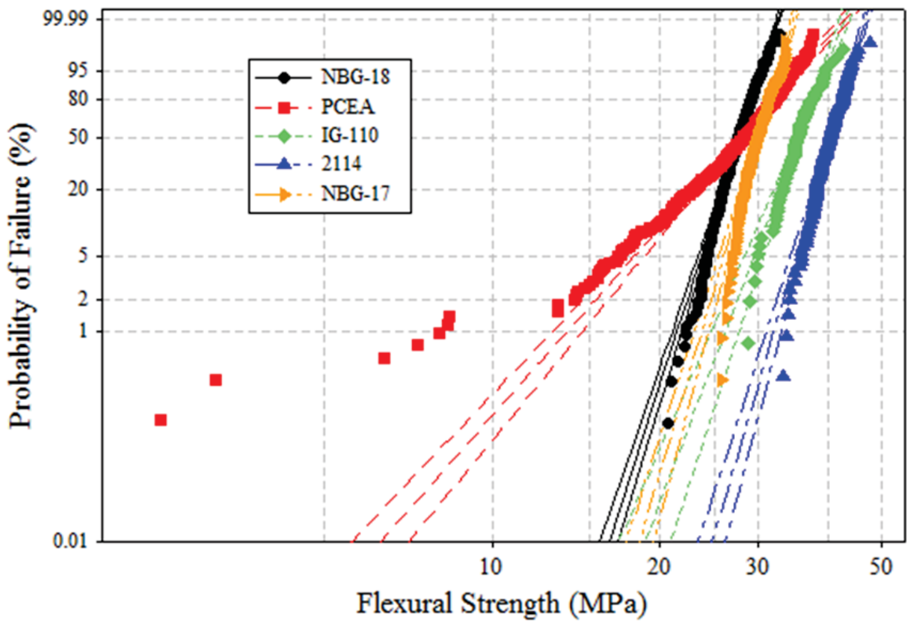


Figure 2. Flexural strength distributions for different candidate grades of graphite evaluated as part of the VHTR program. As with the tensile distributions, the extruded PCEA graphite exhibits the widest range of values (variability) along with the largest number of data points at low values.

Also evident from these example distributions is the higher propensity for very low individual property measurements. A number of discrete data points fall well below the values that would be predicted by the 95% confidence bands (dotted lines) for a consistent distribution, a clear indication that disparate flaws are present in the population of PCEA specimens extracted from the original billets. In some cases, the largest disparate flaws were present in the form of extended fissures that were readily visible[2,3] in the sectioned billets (Figure 3).

Figure 3. Sub-blocks of the original billet of PCEA contained larger disparate flaws that compromised test specimens.

As one of the goals[4] of the Baseline Graphite Characterization program is to evaluate the property variability in nuclear-grade graphites across different subsets of each grade, the variability between graphite batches must be quantified. This report provides initial results from the first billet from the newest batch of PCEA, which was produced after providing GrafTech with selected first batch results and observations. This information helped ensure that the manufacturing process parameters that might be contributing to these property distribution characteristics and flaw populations might be tightened as deemed appropriate. Comparing the results from this billet and subsequent billets from the newest batch with results obtained from the original batch will provide an indication of the success of the approaches employed by the manufacturer to alleviate the most deleterious flaws and thereby decrease the measured variability in property values, providing a basis for more predictable performance in a Very High Temperature Reactor (VHTR) environment.

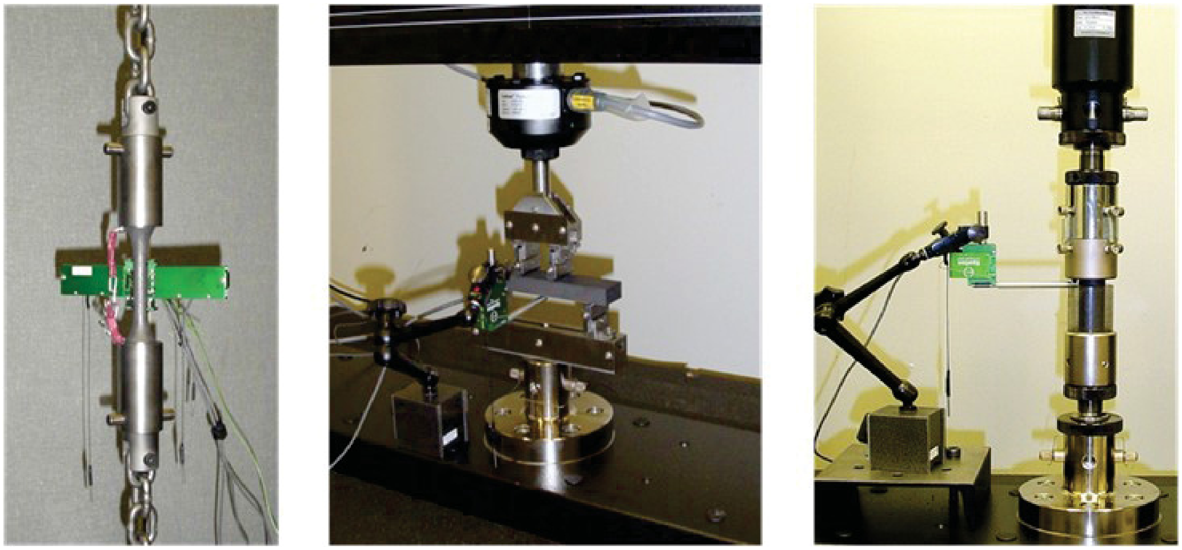
Data from the first billet of PCEA from the newest batch is a compilation of 16 individual physical and mechanical properties taken from 128 tensile, 128 flexural, and 152 compression specimens extracted from randomly-selected sub-sections of PCEA billet XPC01D3-36. Following receipt and detailed dimensional checks, the specimens provided 1,290 individual property data results to describe both the characteristics and inherent variability in this billet of nuclear-grade graphite from the lone extruded grade being evaluated as a VHTR candidate grade.

2. EXPERIMENTAL PROCEDURES

2.1 Physical and Mechanical Properties Testing

The physical and mechanical properties being reported for PCEA graphite are based upon the systematic evaluation of specimens machined to the specific guidelines of the published standards from ASTM International. Tensile testing (Figure 4a) is performed in accordance with ASTM C749-08,[5] flexural testing (Figure 4b) via ASTM C651-10,[6] and compressive testing (Figure 4c) via ASTM C695-91.[7]

As was reported in INL/EXT-13-30011,[2] the relatively simple shapes of flexural (rectangular bars) and compressive specimens (right circular cylinders) make them ideal for the non-destructive evaluation of elastic material constants, such as dynamic Young's modulus and shear modulus values, which can be obtained through measurements of resonant frequency (ASTM C747-93)[8] and sonic velocity (ASTM C769-09).[9] Additionally, the respective geometries of those sample types render accurate geometry/volume measurements relatively straightforward, which allows for the reporting of material density per ASTM C559-90.[10] These evaluations are performed on specimens prior to mechanical testing, allowing the individual position information of each specimen to be utilized in order to describe multiple properties from within a single billet. Moving forward, two additional tests are being reported as part of the Baseline graphite characterization program for all billets: the three-point bend test on sub-sized AGC geometry cylinders, as reported in INL/EXT-15-36044,[11] provides a large data population from which to compare three-point bend testing on irradiated specimens, and the Brazilian disc splitting tensile strength test,[12] which is ideal for testing the small button-sized specimens used for measuring thermal conductivity of AGC specimens via laser flash analysis. Each of these two alternate tests is presented as trend data for comparison purposes.



(a) (b) (c)

Figure 4. Examples of the ASTM-based configurations for (a) tensile, (b) flexural, and (c) compression testing.

2.2 Property Interpretation

The preferred method for illustrating statistical distributions of data in the Baseline Graphite Characterization program is the cumulative distribution function *F(x)* developed by Weibull,[13] which takes the following form:

$$F(x) = 1 - e^{-\left(\frac{x}{\beta}\right)^m}$$

In this two-parameter relationship, x is the individual measured property, β is the associated scale parameter below which 63.2% of the values from the measured data set fall, and m is the shape parameter describing the slope of the cumulative function. When applied specifically to strength values, the shape parameter m is also commonly referred to as the Weibull modulus, while the scale parameter, being indicative of the representative magnitude of the value being measured in the cumulative distribution, is also referred to as the characteristic value. Comparisons of data pools using different distribution types have consistently pointed to the Weibull-based distribution as the best-fit distribution type for nuclear-grade graphite properties.[3] Each of the two parameters in this distribution, β and m, will be the basis for the comparative evaluations of distributions in this report that comprise the basis for the Discussion section as well as for the introductory graphite grade comparison shown in Figures 1 and 2. At present, the two-parameter Weibull relationship is the only relationship defined by ASTM International for the interpretation of graphite strength behavior. ASTM Standard D7846-12[14] provides detailed information on the application of the two-parameter Weibull distribution. Presenting the data as cumulative distributions is not only illustrative of the character of the overall pooled data sets via the best-fit function (Weibull), but is also germane to the evolution of graphite property analysis with respect to present efforts by the subcommittee on Manufactured Carbon and Graphite Products in ASTM International. Based on preliminary results gathered in this program, there is a movement to reconsider the specifications on graphite in terms of minimum values and instead present required or desired graphite properties in terms of the parameters that define a two-parameter Weibull distribution.[15]

3. DISCUSSION

3.1 Physical Properties

An initial evaluation of the property variability within a selected subset or population of nuclear-grade graphite specimens is often readily available through an observation of the measured density values. As has been reported in earlier work, the baseline graphite characterization program utilizes the simple cylindrical and square bar geometries of the compressive and tensile specimens, respectively, for accurate measurements of both dimensions and mass per ASTM 559-90. This initial comparison of the density data (Figure 5) reveals a much tighter distribution of measured data values throughout the billet of PCEA from the new batch (henceforth PCEA 2) when plotted alongside the pooled data from the three billets evaluated from the first batch (PCEA 1). It is expected that other measured properties will show a similar low level of variability when compared to PCEA data from the first batch. Further discussion on this topic will follow in the section on Mechanical Properties.

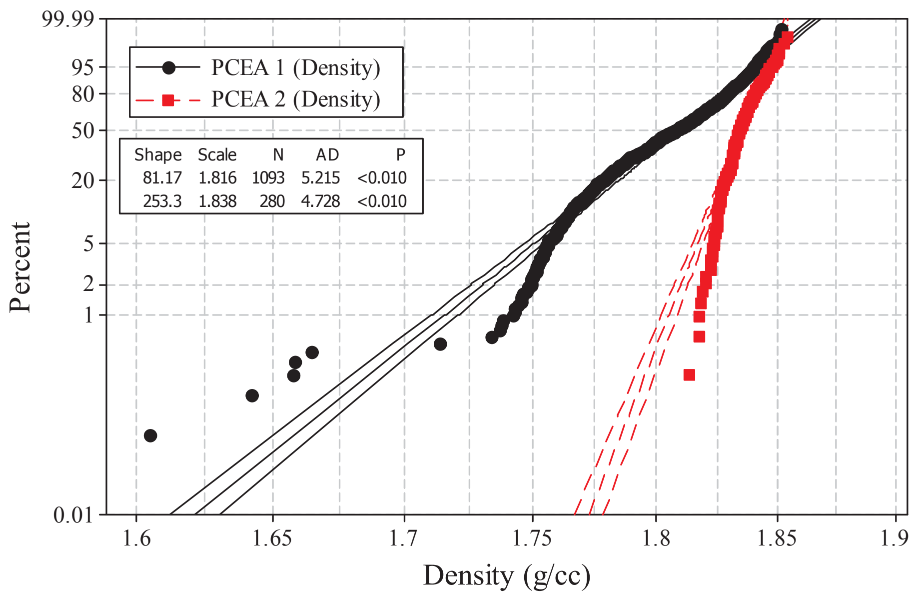


Figure 5. Density distributions from the original of batch PCEA (PCEA 1) and the billet from the newest batch (PCEA 2) reveal a much narrower spread of measured specimen density values from the new batch.

One of the key components of the Baseline Graphite Characterization continues to be the validation of test techniques on specimens from the AGC experiment that are limited to what might be considered in some tests to be a compromising geometry. One of these tests is the evaluation of dynamic Young's modulus via measurements of sonic resonance. Ideally, a specimen utilizing this technique has a large enough length-to-cross-section ratio to enable consistent resonance between support nodes after impulse excitation. An example of an ideal geometry is the square bars machined for flexural testing, each of which is subjected to dynamic Young's modulus evaluations as part of the non-destructive physical properties protocol carried out prior to final mechanical testing. Evaluating a cross-section of the compressive specimens, which are machined to match the specified dimensions of the AGC specimens, allows a direct comparison of the validity of the test on specimens with small length-to-cross-section ratios. Figure 6 provides an example of this comparison, with the cylindrical specimens not only showing an extremely good agreement between distribution characteristic values, but also a matching shape parameter that indicates a representative level of variability in the data as confirmed by the larger population of tested flexure bars.

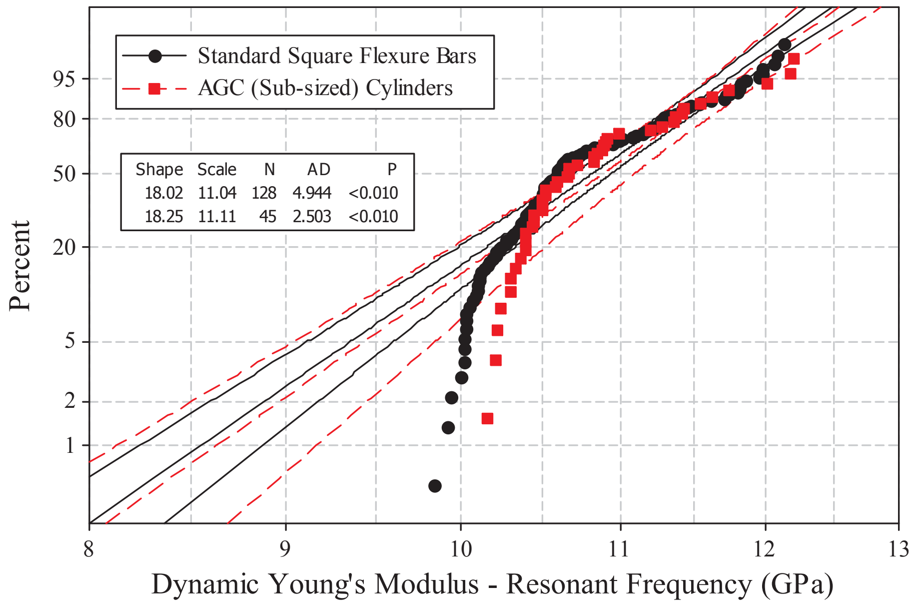


Figure 6. Dynamic Young’s modulus values collected via resonant frequency shows excellent agreement between “ideal” long square-bar geometries and the smaller AGC cylindrical geometry.

Dynamic Young’s modulus via measurements of resonant frequency is only one accepted method for measuring the elastic properties of nuclear-grade graphites. Extracting the stress-strain relationship from the tensile test curve is another method of evaluating Young’s modulus, and the measurement of sound wave speed through sonic velocity measurements will, in theory, provide a value representing the same characteristic. However, as past evaluations have shown, the measurement techniques are prone to provide differing values—attributable in principle to the area or volume of elastic interaction that provides the measured response during the test. Past data has consistently shown that modulus measurements using dynamic techniques (resonant frequency and sonic velocity) have higher characteristic values for their respective distributions than those measured via the standard tensile test. Additionally, values measured via sonic velocity are consistently higher than those measured via sonic resonance. This relationship continues to hold true with the values measured in the billet of PCEA under study in this report, with the same relationship between elastic modulus values. Figure 7 shows the relative distributions with resonant frequency modulus values being higher than elastic modulus values from tensile testing and sonic velocity values being highest of all from a property distribution standpoint.

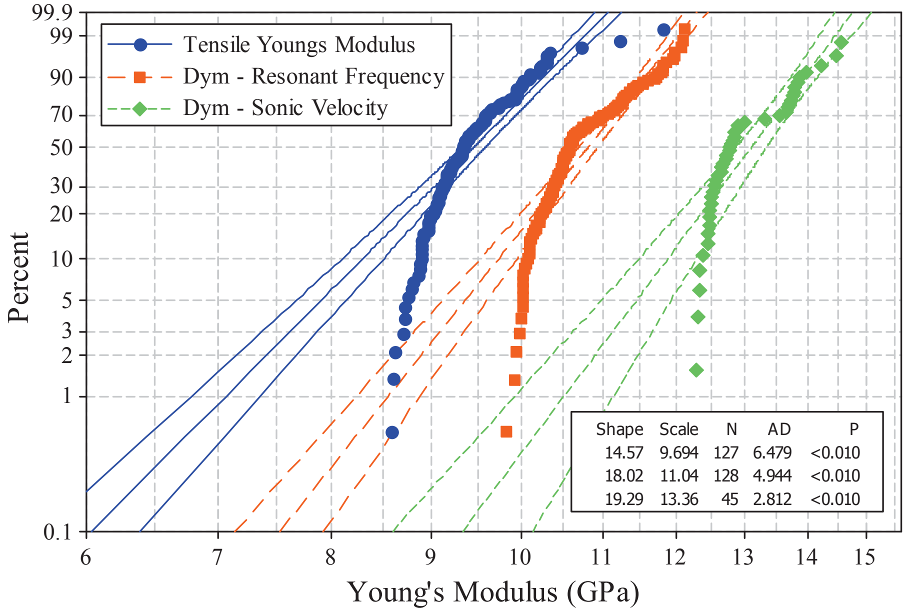


Figure 7. Young's modulus values from the same PCEA 2 billet quantified dynamically via sonic velocity, dynamically via resonant frequency, and from the stress-strain relationship in standard tensile testing.

3.2 Mechanical Properties

3.2.1 Tensile Testing

Results of the tensile tests from the first billet exhibit a similar relationship to that seen with the density reported in Section 3.1. The data from this first billet of PCEA shows less variability than the pooled data from the first batch of PCEA (Figure 8), qualitatively demonstrated by the shape parameter, or Weibull modulus value, of 6.12 for the new-batch PCEA and 3.69 for the pooled tensile data from the first billet. The scale parameters, or characteristic values, are slightly higher for the new PCEA versus the original-batch PCEA, at 19.74 and 18.59 MPa, respectively. With the pooled data from the first batch representing a much larger set of data than that compiled from the new billet of PCEA, consideration must be given to the possibility that the differences in variability are attributable to the increased chance of low outlying values in the larger dataset rather than a true reduction in variability for the new-batch PCEA. The pooled data from the original batch comprised 484 tensile test strength results, while the compilation of data from the new PCEA totaled 127 tensile strength values. Figure 9 shows that a possible difference based solely on an artifact of larger pooled data sets is not the case. In actuality, the distribution shape from the new PCEA shows the same relative drop-off at the lower values even when compared with individual billets, rather than a pooled set, of PCEA from the first batch.

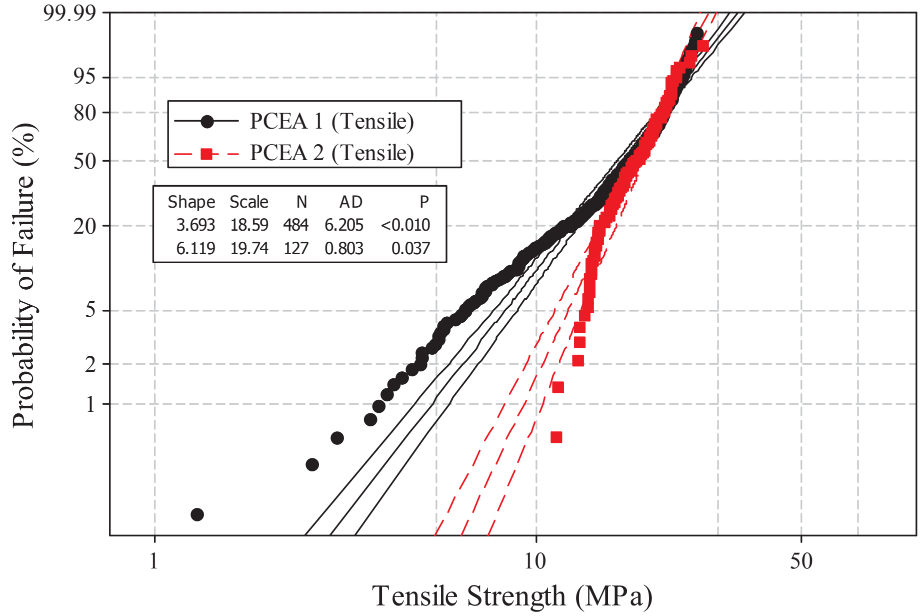


Figure 8. Tensile data from the pooled data from the original batch of PCEA and the billet from the new batch shows a much steeper slope (tighter distribution of values) for the new PCEA. The characteristic strength values are slightly higher for the new PCEA, but still reasonably similar.

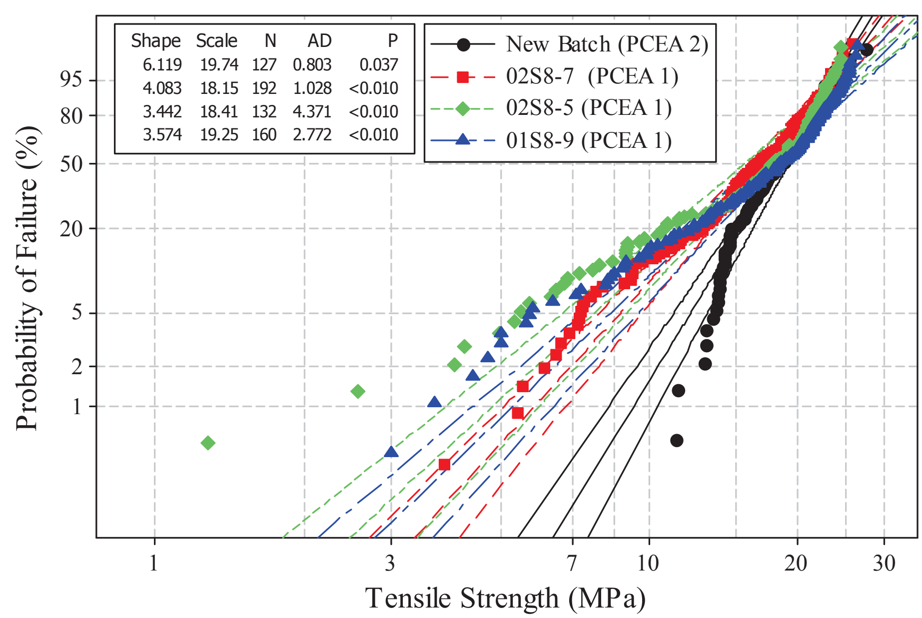


Figure 9. Further breakdown of the PCEA from the original batch shows that the difference in the shape parameter between the batches of PCEA was not an artifact of pooling the data. Each of the original-batch PCEA shows a much wider spread of data at the lower end of the distribution than is exhibited by the new PCEA billet.

This slight decrease in variability in tensile properties is also reflected in the tensile strain distributions (Figure 10), in which the characteristic strain value for all measured strain-at-failure data points is similar, at 0.308% for new-batch PCEA and 0.306% for original-batch PCEA. The shape parameter is not substantially higher for the strain values, but the distribution slope is still clearly steeper for new-batch PCEA.

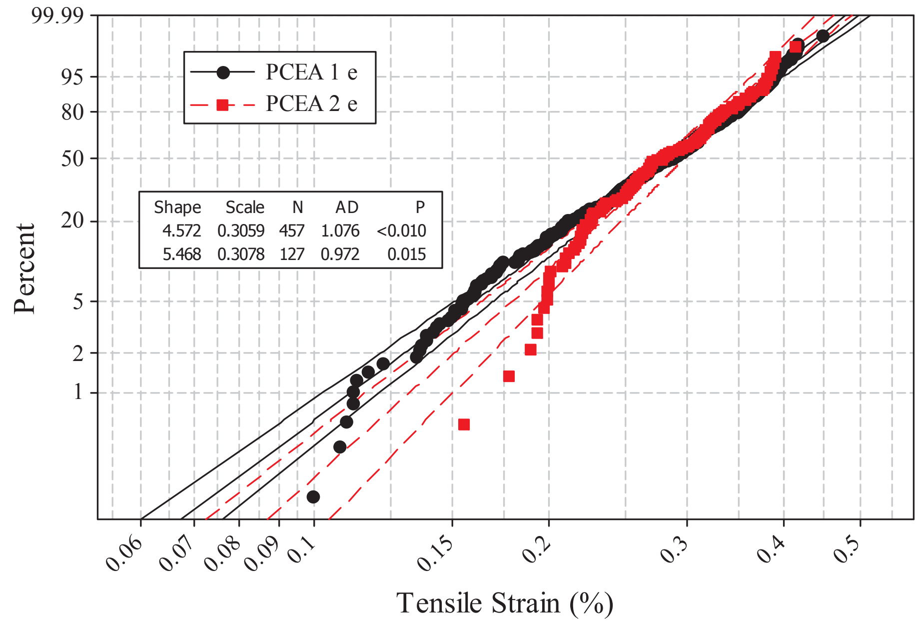


Figure 10. Tensile strain data of PCEA 1 and PCEA 2 reveals similar behavior to the strength results from tensile testing, although the characteristic values are almost identical.

3.2.2 Flexural Testing

Flexural testing comparisons between billets from the original batch and the billet from the new batch are similar in character to those differences noted from the tensile test comparisons. The characteristic values are higher for the new billet, at 30.87 MPa, than measured from the original batch, at 29.62 MPa. Most notably, the shape parameter remains measurably higher for the new batch, with a steeper slope clearly evident when comparing the cumulative distribution functions (Figure 11).

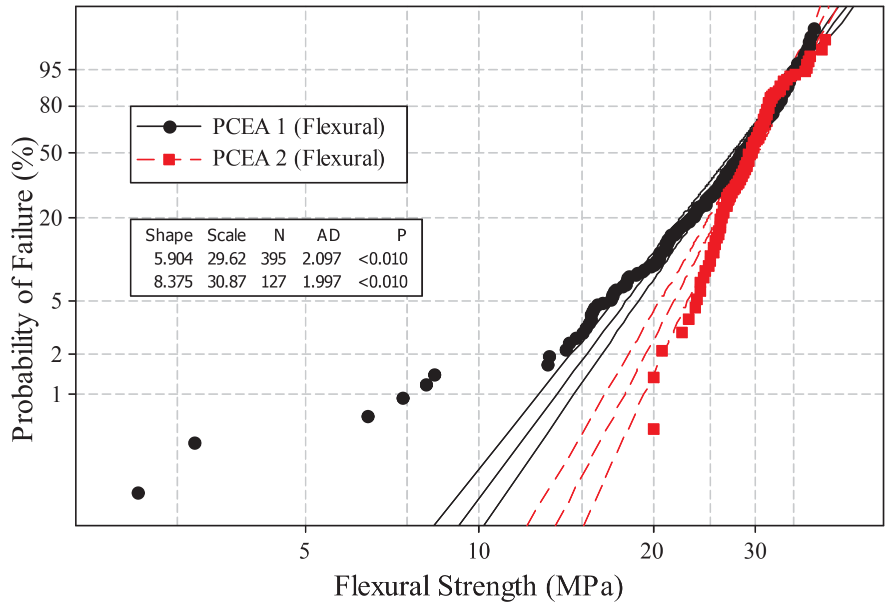


Figure 11. Flexure test comparison between batches of PCEA.

3.2.3 Compression Testing

Compression testing reveals results very similar to those seen in the other two major mechanical test types. The characteristic values are extremely similar and are actually slightly lower for the new-batch PCEA, at 67.17 MPa versus 67.25 MPa for pooled strength data from the original billet. The Weibull modulus shows an interesting trend not as clearly seen with tensile or flexural testing; while the strength values exhibit considerably lower overall variability as exhibited by the steep slope of the distribution, values from the original batch show not only a large number of values that are lower than from the new batch, but also a large number of higher strength values. While the new-batch PCEA values all fall between about 50 and 75 MPa, testing of the original batch of PCEA revealed a spread of data that ranged in compressive strength values from under 30 MPa all the way up to 80 MPa.

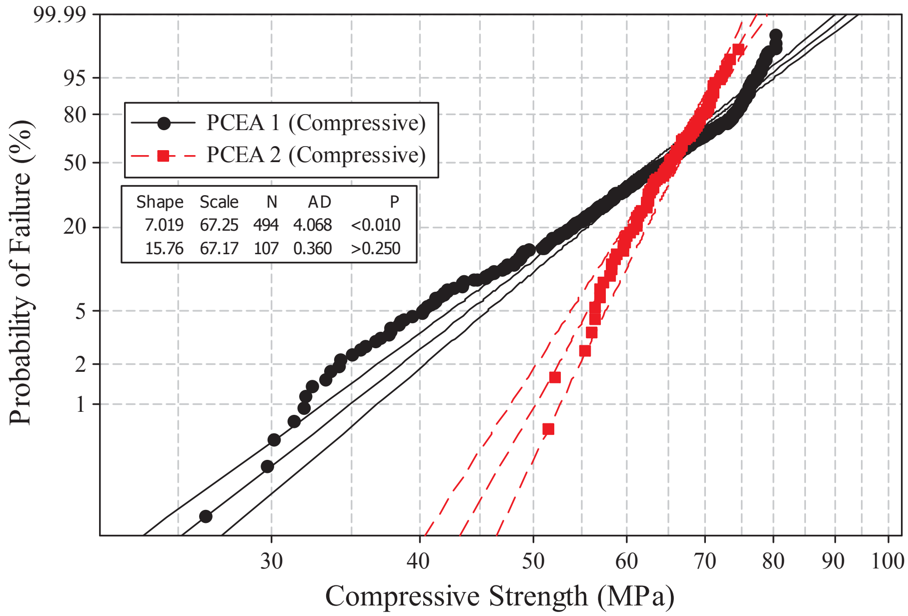


Figure 12. The distributions of compressive test strength values reveal a very similar overall characteristic value between the new batch and the original batch of PCEA, but significantly lower overall variability in the new batch billet.

3.2.4 Three-point Bend Testing

The three-point bend test has recently been adopted for evaluating carbon and graphite products under ASTM Standard D7972-14.[16] The constraint on testing per the standard lies in the prescribed specimen length-to-diameter relationship, with a recommended ratio of at least 6:1. Accepting that the geometry is fixed at this non-ideal ratio for AGC specimens, adding the three-point bend test to the Baseline Graphite Characterization program is an ideal method by which to accumulate data on the test technique that is based on specimens taken from the same billet of graphite as "standard" flexure specimens tested in four-point bending. Further information on the considerations for three-point bend testing as they apply to the Baseline Graphite Characterization program (i.e. a basis in irradiated specimen testing) is provided in another report.[11]

Figure 2 is an example comparison between flexure strengths obtained through standard four-point flexure testing on rectangular bars and flexure strength from three-point bend testing on short cylinders, all from the new billet of PCEA graphite. As shown from the cumulative distributions, the four-point flexure test has considerably lower measured flexure strength using the standardized approach from ASTM C651-11, a trend that is seen across the candidate grades being evaluated.[1] While several factors may explain this significant shift in values that are based on the test geometry and fundamental flexure physics, the test approach nonetheless has considerable value in providing information on specimens that are either limited in geometry, such as the AGC specimens, and sample volumes that are otherwise limited in size and render ideal geometries impossible. The potential to carry out comparative strength evaluations on relatively simple geometries also provides an opportunity to readily assess other non-destructive physical and thermal property measurements for which simple cylinders are ideal. Continued testing in the Baseline Graphite Characterization program that can help illuminate the differences in measured values from specimens of the same grade/billet of graphite will be extremely valuable in quantifying the fundamental response behavior.

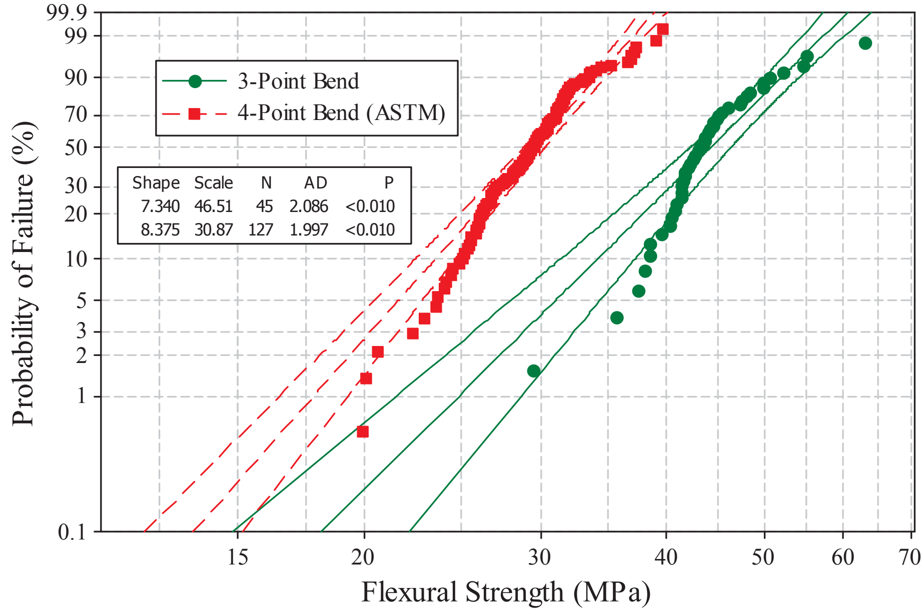


Figure 13. Data distributions from the standard four-point bend testing and three-point bend testing on sub-sized specimens reveal the propensity for much higher strength values on the specimens with non-ideal length-to-cross section ratios and cylindrical geometries.

3.2.5 Brazilian Disc Tensile Splitting Strength

A measure of tensile properties can also be ascertained from the splitting strength of disc-shaped specimens loaded on end in compression. The Brazilian disc, or splitting tensile strength, test is presently standardized as a test for measuring splitting strength in other types of materials that include rock and concrete. An adaptation of this test for quantification of splitting strength in nuclear graphite has been investigated by Tsang et al.[17] for specimens of a single fine-grained grade with a specific geometry, with an allowance for the propensity for graphite to fracture in compression at a single loading point being made by adding a radius of curvature to the upper and lower compression surfaces (Figure 14). Qualifying this approach as a standardized test method is being carried out by comparing measured tensile strengths from the Brazilian disc test, on a range of both candidate graphite grades and overall dimensions that maintain a 2:1 diameter-to-thickness ratio, to those obtained through strict application of ASTM C749-08.[5]

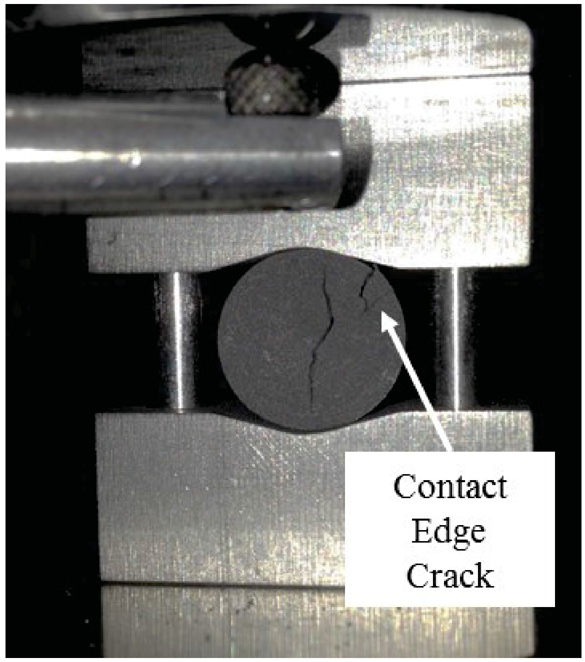
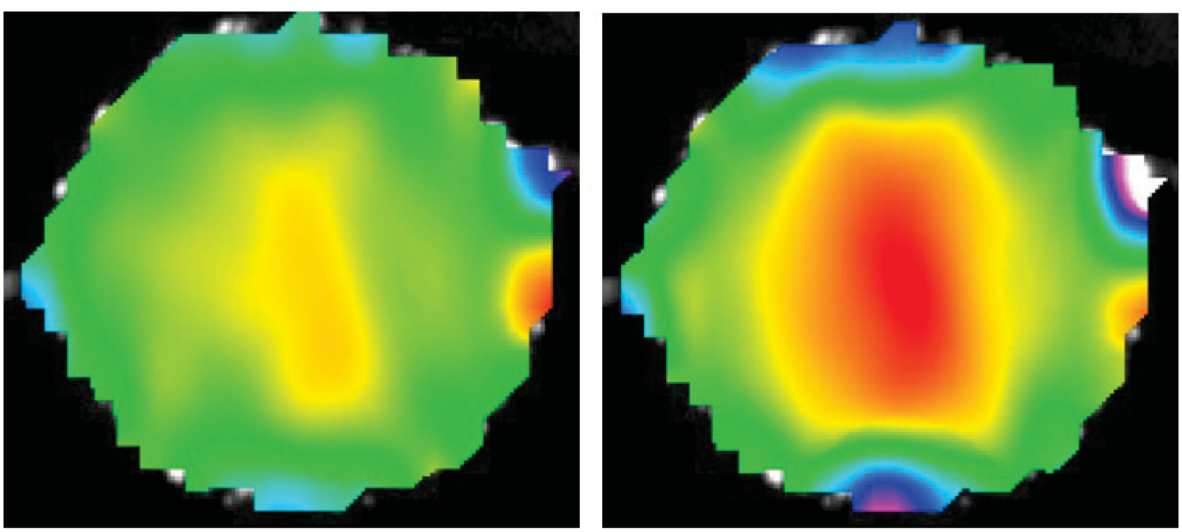


Figure 14. Compression fixture for stressing discs in transverse tension via the Brazilian disc splitting test reveals both the expected centerline crack and an anomalous contact surface compression crack.



(a) (b)

Figure 15. Frozen frames from dynamic strain map video shows the initial application of a tensile stress (a) and buildup to a maximum splitting stress (b) in a vertical plane transverse to the compressive load.

Figure 15 provides two frozen frames from a dynamic in-situ strain map during compressive loading of a disk. The transverse component of the strain is seen building up in Figure 15a to the elevated level shown in Figure 15b. The region outside of the concave contact surfaces exhibits the tensile splitting component that can be calculated from the load via the following relationship:[17]

$$\sigma_t = \frac{2P}{\pi LD}\left[1 - \frac{\pi^2}{144}\right]$$

where σ_t is the splitting tensile stress, P is the maximum applied load, and L and D are the thickness and diameter of the specimen, respectively.

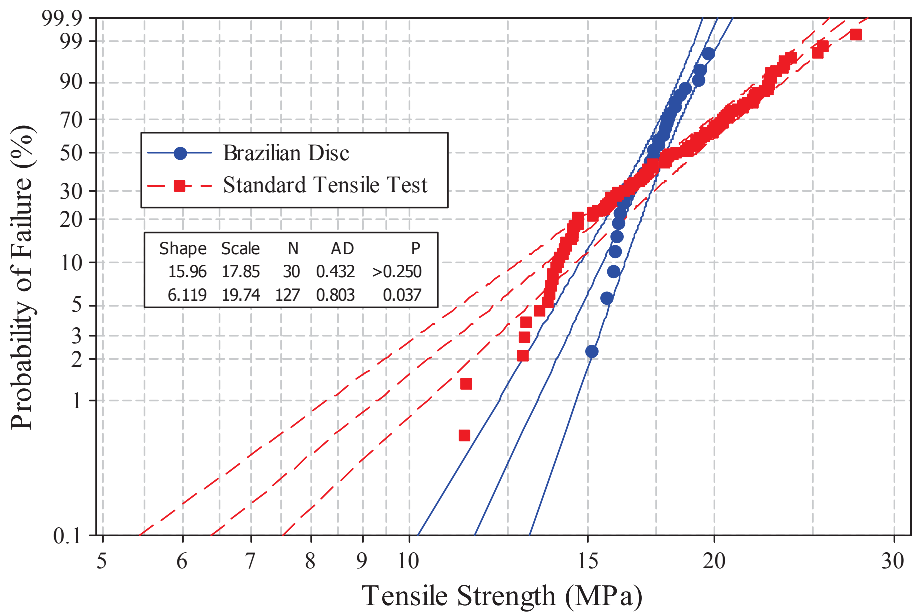


Figure 16. The distribution in tensile strength between standard tensile testing and Brazilian disc splitting shows reasonably good agreement, with the splitting test actually demonstrating less spread in the data despite drawing specimens from throughout the billet.

The first qualified data on the strict comparison between standard tensile testing and Brazilian disc splitting was collected on the single billet of PCEA being reported on in this study. Results2. The distributions of values obtained utilizing the Brazilian disc splitting (30 specimens) and the standard tensile test per ASTM C749-08 (127 specimens) are shown in Figure 16. The comparison is reasonably correlative; the shape parameter is considerably higher for the splitting test at 15.96 versus 6.12 for the tensile test while the measured tensile strength is only slightly lower using the Brazilian disc technique, at 17.85 MPa versus 19.74 MPa While continued testing will allow larger specimen populations to be leveraged to come up with appropriate expected deviations in values between the tests, the Brazilian disc splitting technique is showing immediate promise as a means to evaluate tensile strength in specimens that are extremely limited on geometry.

4. SUMMARY

Completion of all Baseline Graphite Characterization testing on the first billet of PCEA graphite from the newest batch obtained by the Advanced Reactor Technologies VHTR program allowed an initial assessment of the batch-to-batch variation in PCEA, with a specific focus on manufacturing/densification artifacts that may have led to the larger population of data available from the three billets testes thus far in the program from the first batch exhibiting low outlying property values. Direct comparisons of cumulative distributions between the new PCEA and pooled data from the original batch demonstrated measurably lower levels of variability from the new batch billet. While work will continue that includes multiple billets from the new batch, the early indication is that a much more consistent and predictable set of property values is possible from this candidate grade of extruded graphite.

Presenting the data as cumulative distributions is also germane to the evolution of graphite property analysis with respect to present efforts by the ASTM International subcommittee on Manufactured Carbon and Graphite Products.There is a movement within this subcommittee to reconsider the specifications on graphite in terms of minimum values and instead present required or desired graphite properties in terms of the parameters comprising the two-parameter Weibull distribution. Of the 1,290 property values collected from the billet of new PCEA, Table 1 provides those parameters for each of the main properties that are readily plotted in a Weibull cumulative distribution function.

Table 1. The listed Weibull parameter values for each of the properties plotted via two-parameter Weibull cumulative distribution functions. Units apply to the scale parameter (characteristic value).

Property	**Scale**	**Shape**
Compressive Strength	67.17 MPa	15.76
Flexural Strength	30.87 MPa	8.38
Tensile Strength	19.74 MPa	6.12
Three-point Flexural Strength	46.51 MPa	7.34
Brazilian Disc Splitting Tensile Strength	17.85 MPa	15.96
Tensile Test Young’s Modulus	9.69 GPa	14.57
Flexural Specimen (Resonant Frequency) Dynamic Young’s Modulus	11.04 (GPa)	18.02
Compressive Specimen (Resonant Freq.) Dynamic Young’s Modulus	11.11 GPa	18.02
Dynamic Young’s Modulus – Sonic Velocity	13.36 GPa	19.29
Flexural Specimen Shear Modulus (Resonant Freq. – Torsion)	4.65 GPa	31.92
Compressive Specimen Shear Modulus (Sonic Velocity – Shear Wave)	5.03 (GPa)	33.63
Electrical Resistivity	7.77 mW-m	32.22
Density	1.84 g/cm^3	253.3

The data distributions provided in this report are intended as an evaluation of the trends being seen in the pooled data sets from the two batches of PCEA graphite. Final release of the individual data and all associated metadata will result in raw data sets being accessible through the Nuclear Data Management and Analysis System (NDMAS).

5. REFERENCE

1. Carroll, M. C., *Initial Comparison of Baseline Physical and Mechanical Properties for the VHTR Candidate Graphite Grades*" INL/EXT-14-33120, Idaho National Laboratory, September 2014.
2. Carroll, M. C., "Baseline Graphite Characterization Plan: Electromechanical Testing," PLN-3467, R.2 (Idaho National Laboratory), June 2015.
3. Carroll, M. C., W.E. Windes, D. T. Rohrbaugh, J. P Strizak, and T. D. Burchell, "Leveraging comprehensive baseline datasets to quantify property variability in nuclear-grade graphites," *Nucl. Eng. and Design*, Vol. 307, 2016, pp. 77–85.
4. Carroll, M. C.,. and D.T. Rohrbaugh, *Statistical Comparison of the Baseline Mechanical Properties of NBG-18 and PCEA Graphite*, INL/EXT-13-30011, Idaho National Laboratory, August 2013.
5. ASTM Standard C749-08, "Standard Test Method for Tensile Stress-Strain of Carbon and Graphite,"" ASTM International, West Conshohocken, PennsylvaniaPA, 2010.
6. ASTM Standard C651-11, "Standard Test Method for Flexural Strength of Manufactured Carbon and Graphite Articles Using Four-Point Loading at Room Temperature," ASTM International, West Conshohocken, Pennsylvania, 2011.
7. ASTM Standard C695-91, "Standard Test Method for Compressive Strength of Carbon and Graphite," ASTM International, West Conshohocken, Pennsylvania, 2010.
8. ASTM Standard C747-93, "Standard Test Method for Moduli of Elasticity and Fundamental Frequencies of Carbon and Graphite Materials by Sonic Resonance," ASTM International, West Conshohocken, Pennsylvania, 2010.
9. ASTM Standard C769-09, "Standard Test Method for Sonic Velocity in Manufactured Carbon and Graphite Materials for Use in Obtaining Young's Modulus," ASTM International, West Conshohocken, Pennsylvania, 2009.
10. ASTM Standard C559-90, "Standard Test Method for Bulk Density by Physical Measurements of Manufactured Carbon and Graphite Articles," ASTM International, West Conshohocken, Pennsylvania, 2010.
11. Carroll, M. C., *Preliminary Investigation of the Effect of AGC-2 Irradiation on the Strength of Different Grades of Nuclear Graphites*, INL/EXT-15-36044, R.0, Idaho National Laboratory, July 2015.
12. Carroll, M. C., D. T. Rohrbaugh, P. Chakraborty, and W. D. Swank, Evaluating alternate test techniques to characterize mechanical properties in nuclear-grade graphites," *HTR2016* (in submission).
13. Weibull, W., "A statistical distribution of wide applicability," *J. Appl. Mech.*, Vol. 18, 1951, pp. 293–297.
14. ASTM Standard D7846-12, "Standard Practice for Reporting Uniaxial Strength Data and Estimating Weibull Distribution Parameters for Advanced Graphites" ASTM International, West Conshohocken, Pennysylvania, 2012.
15. ASTM International, Discussion and action at ASTM International subcommittee meeting D02.F0 "Manufactured Carbon and Graphite Products," Bellevue, Washington, June 28–29, 2016.
16. ASTM Standard D7972-14, "Standard Test Method for Flexural Strength of Manufactured Carbon and Graphite Articles Using Three-Point Loading at Room Temperature," ASTM International, West Conshohocken, Pennsylvania, 2010.

17. Tsang, D., *International Nuclear Graphite Specialists Meeting,* Hangzou, China, September 15–18, 2014.