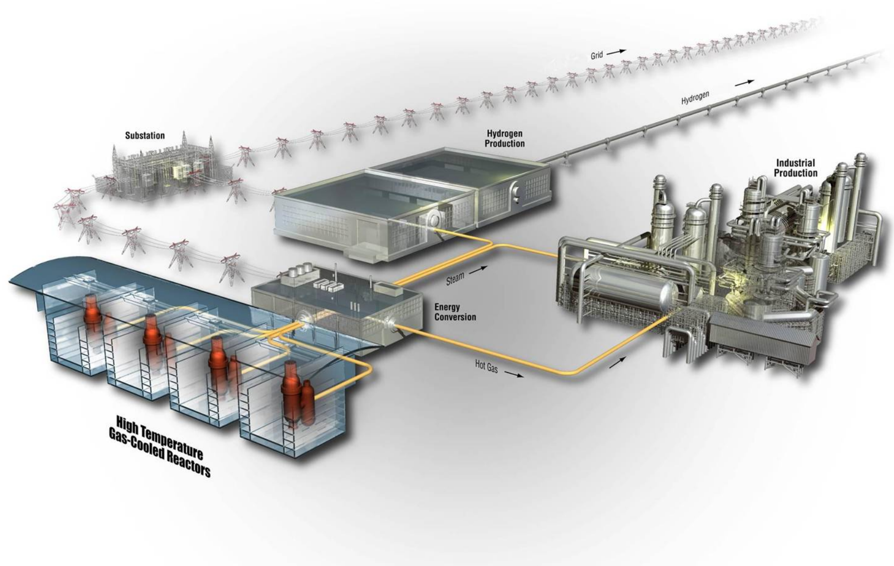
INL/EXT-14-33120

Initial Comparison of Baseline Physical and Mechanical Properties for the VHTR Candidate Graphite Grades

M.C. Carroll

September 2014



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http://www.inl.gov

Prepared for the
U.S. Department of Energy
Office of Nuclear Energy
Under DOE Idaho Operations Office
Contract DE-AC07-05ID14517

VHTR Program

Initial Comparison of Baseline Physical and Mechanical Properties for the VHTR Candidate Graphite Grades

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September 2014

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ABSTRACT

High-purity graphite is the core structural material of choice in the Very High Temperature Reactor (VHTR) design, a graphite-moderated, helium-cooled configuration capable of producing thermal energy for power generation as well as process heat for industrial applications that require temperatures higher than the outlet temperatures of present nuclear reactors.

The Baseline Graphite Characterization Program is establishing accurate as-manufactured mechanical and physical property distributions in nuclear-grade graphites by providing comprehensive data that captures the level of variation in measured values. In addition to providing a thorough comparison between these values in different graphite grades, the program is also carefully tracking individual specimen source, position, and orientation information in order to provide comparisons both in specific properties and in the associated variability between different lots, different billets, and different positions from within a single billet.

This report is a preliminary comparison between each of the grades of graphite that are considered "candidate" grades from four major international graphite producers. These particular grades (NBG-18, NBG-17, PCEA, IG-110, and 2114) are the major focus of the evaluations presently underway on irradiated graphite properties through the series of Advanced Graphite Creep (AGC) experiments. NBG-18, a medium-grain pitch coke graphite from SGL from which billets are formed via vibration molding, was the favored structural material in the pebble-bed configuration. NBG-17 graphite from SGL is essentially NBG-18 with the grain size reduced by a factor of two. PCEA, petroleum coke graphite from GrafTech with a similar grain size to NBG-17, is formed via an extrusion process and was initially considered the favored grade for the prismatic layout. IG-110 and 2114, from Toyo Tanso and Mersen (formerly Carbone Lorraine), respectively, are fine-grain grades produced via an isomolding process.

An analysis of the comparison between each of these grades will include not only the differences in fundamental and statistically-significant individual strength levels, but also the differences in the overall variability in properties within each of the grades that will ultimately provide the basis for predicting in-service performance. The comparative performance of the different types of nuclear-grade graphites will naturally continue to evolve as thousands more specimens are fully characterized with regard to strength, physical properties, and thermal performance from the numerous grades of graphite being evaluated.

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ACRONYMS

AGC	Advanced Graphite Creep
VHTR	Very High Temperature Reactor

Initial Comparison of Baseline Physical and Mechanical Properties for the VHTR Candidate Graphite Grades

1. INTRODUCTION

High-purity graphite is the core structural material of choice in the conceptual design for the Very High Temperature Reactor (VHTR), a graphite-moderated, helium-cooled configuration capable of producing thermal energy for power generation as well as process heat for industrial applications that require temperatures higher than the outlet temperatures of present nuclear reactors. While nuclear-grade graphite is an ideal material for this design based upon its extremely high temperature capabilities coupled with an optimum combination of thermal stability, machinability, and low cost, the quasi-brittle mechanical properties of graphite have been shown to exhibit a relatively large amount of scatter in measured strength levels[1]. The mechanical response of nuclear graphites are dependent upon the inherent defect structure, composed of boundaries between filler particles, pores, voids, inclusions, and cracks that are present at all practical length scales[2]. The measured mechanical properties in various graphite specimens will be strongly based upon the size distribution of these defects and their relative orientation with respect to the stress axis.

The Baseline Graphite Characterization Program is establishing an accurate representation of as-manufactured mechanical, physical, and thermal properties in nuclear-grade graphites by providing comprehensive data that captures the level of variation in measured values. In addition to providing a detailed comparison between these values in different graphite grades, the program is also carefully tracking individual specimen source, position, and orientation information in order to provide comparisons in properties between different lots, different billets, and different positions from within a single billet. This analysis includes not only mean values from extensive data sets, but also a full evaluation of the inherent variability in those properties in order to provide a full representation of the expected critical properties of interest for nuclear applications.

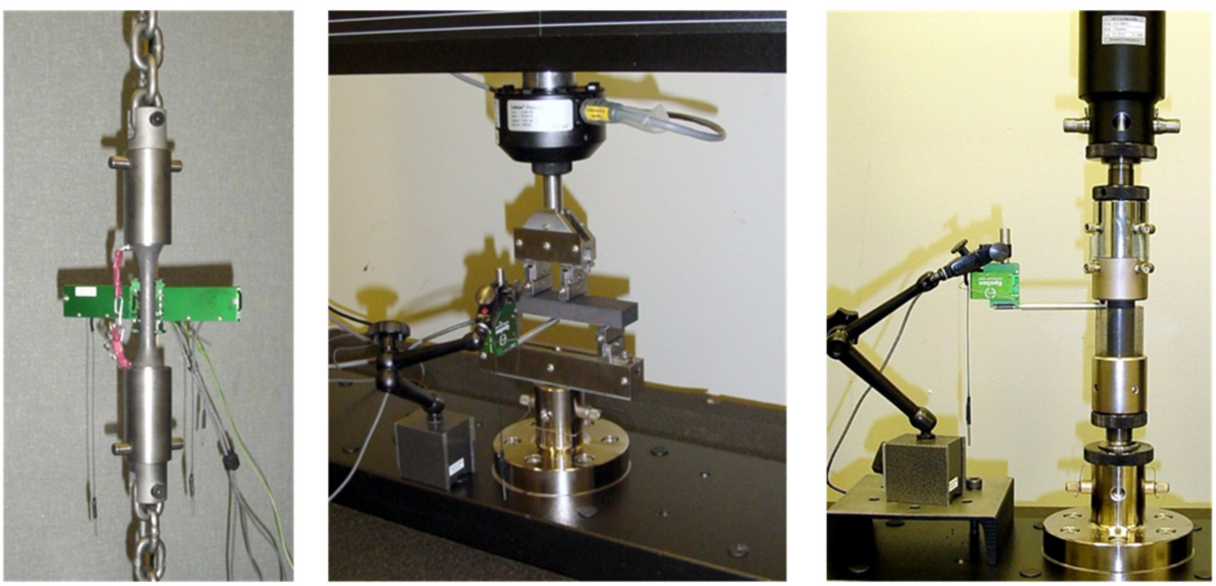
This report is a preliminary comparison between each of the five major grades of graphite considered “candidate” grades for the VHTR design and are the focus of the series of Advanced Graphite Creep irradiation experiments[3]. NBG-18, a medium-grain pitch coke graphite from SGL from which billets are formed via vibration molding, and PCEA, a smaller grain, petroleum coke graphite from GrafTech formed via an extrusion process, were initially the favored grades for pebble-bed and prismatic designs[4] and were the focus[5] of Report INL/EXT-13-30011 “Statistical Comparison of the Baseline Mechanical Properties of NBG-18 and PCEA Graphite.” This report expands the qualitative and statistical analysis to the other three major candidate grades – NBG-17, IG-110, and 2114. NBG-17 graphite from SGL is essentially NBG-18 with the grain size reduced by a factor of two, while IG-110 and 2114, from Toyo Tanso and Mersen (formerly Carbone Lorraine), respectively, are fine-grain grades produced via an isomolding process.

This report focuses on each of these candidate graphite grades with an emphasis on the measured differences in property values and their associated variability from a statistical analysis standpoint. As with INL/EXT-13-30011, the ongoing evaluation process will continue to analyze critical property variability from an inter- and intra-billet standpoint from within specific grades, but this report is structured to present larger-scale comparisons by individual graphite grade. Data sets are therefore grouped entirely by grade and include property measurements from three full-size billets of NBG-18, three full-size billets of PCEA, single full billets of NBG-17 and 2114, and two partial billets of IG-110. The comparisons will include both nondestructive analyses performed to ascertain specific physical properties and the mechanical strength property data obtained through the testing to failure of more than 5,000 graphite specimens.

As the program continues to collect critical physical and mechanical property data, numerous additional billets from differing batches will help solidify the predictive capability associated with nuclear-grade graphites based upon the position-, billet-, and grade-specific information being tagged on each individual data set. This report provides the basis for the continued refinement of grade-specific comparisons as thousands of additional specimens are fully characterized from the numerous grades of graphite being evaluated.

2. EXPERIMENTAL PROCEDURES

The physical and mechanical properties being reported are based upon a systematic evaluation of specimens machined to the specific guidelines of the published standards from ASTM International. Tensile testing (Figure 1a) is carried out via ASTM C749-08[6], flexural testing (Figure 1b) is carried out via ASTM C651-10[7], and compressive testing (Figure 1c) is carried out via ASTM C695-91[8]. As reported in INL/EXT-13-30011, the relatively simple shapes of flexural (rectangular bars) and compressive specimens (right circular cylinders) render 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[9]) and sonic velocity (ASTM C769-09[10]). 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[11]. These evaluations are performed on specimens prior to mechanical testing, allowing each specimen's individual position to be a spatial reference point describing multiple properties from within a single billet.



(a) (b) (c)

Figure 1. Examples of the ASTM-based configurations for (a) tensile, (b) flexural, and (c) compression testing of nuclear-grade graphite.

The goals of this program necessitate the accurate tracking of individual specimen source, position, and orientation information, each of which is recorded and embedded in the applicable test files. For the grade-specific focus of this report, little emphasis will be placed on the tracking of individual specimen positions or the specific effect of those positions within a particular billet. Specific tracking methodology is detailed in INL/EXT-13-30011 and INL/EXT-10-19910 "Baseline Graphite Characterization: First Billet[12]." The data sets are set up to track that data, however, so that ongoing comprehensive evaluations can be broken down into trends that are both qualitatively and quantitatively indicative of the variability within individual billets. The sectioning plans are subject to some level of variability in number of individual tensile, flexural, or compressive specimens produced based on relative geometries and a random sampling of sub-blocks selected for final machining, but the NBG-18 billets generally yield 580-770 total specimens, PCEA yields 450-600 specimens, NBG-17 yields 600-760 specimens, and 2114 yields 760-840 total specimens per full-sized billet. The first billet of IG-110 under evaluation was a partial billet and only142 specimens were machined and tested, but the full billets of IG-110, one of which has been machined and is in the testing phase, will general yield 700-800 total specimens for mechanical testing and evaluation.

The distribution of data in this report will be largely presented as cumulative probability density functions *F(x)* according to Weibull distributions of the following form:

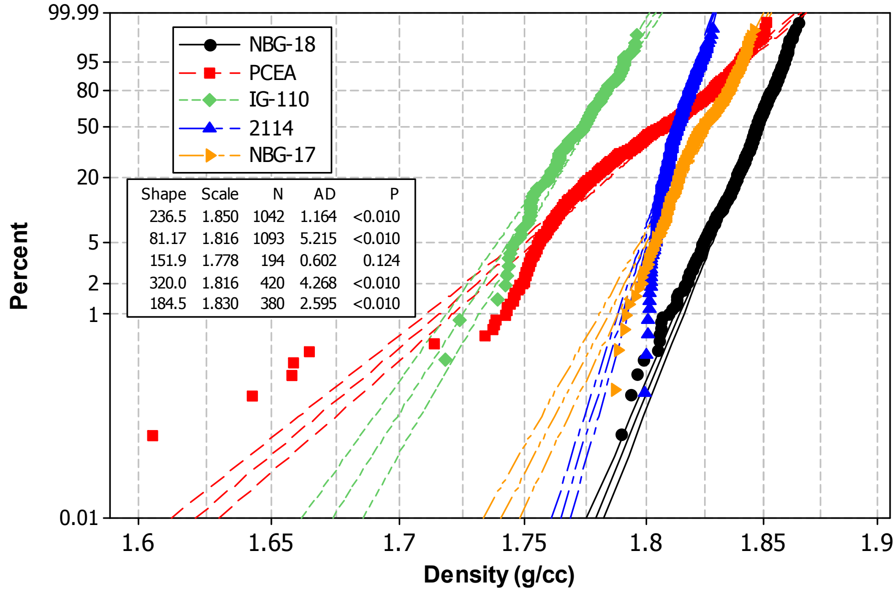
$$F(x) = 1 - e^{-(\frac{x}{\alpha})^{\gamma}}$$

In this two-parameter relationship, x is the individual measured property, α is the associated scale factor below which 63.2% of the values from measured data set fall, and γ is the shape parameter describing the slope of the cumulative function. The two-parameter Weibull distribution is presently the only statistical analysis technique specifically adopted as an ASTM standard (ASTM D7846-12[13]) for the evaluation of graphite mechanical properties. While our studies will continue to evaluate the merits of other distribution types with the goal of providing the most accurate model for property distribution probabilities, the Weibull distribution will remain the primary tool for presenting data sets for comparison. The approach for providing better fits for data distributions will be discussed in the following sections.

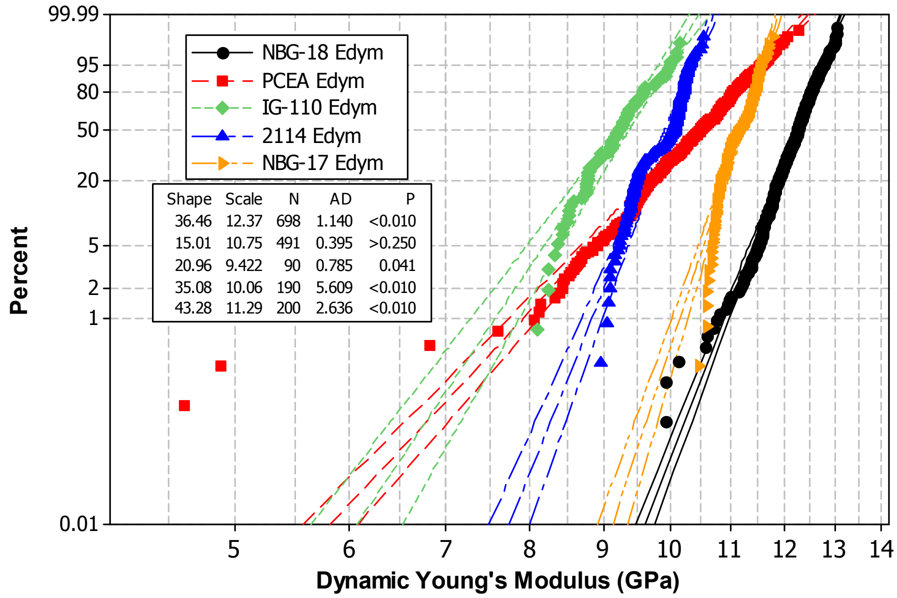
3. PHYSICAL PROPERTIES

As outlined in Section 2, density measurements per ASTM C559-90 are a relatively straightforward endeavor on specimens with simple shapes that can be accurately measured for overall volume and weighed. Per ASTM D7219-08[14], graphite has a minimum allowable density of 1.7 g/cc to be considered nuclear-grade, a value that has an integral effect on the core neutronics and predictability of the moderated neutron flux in an operating reactor. The vast majority of the measured values fall within this range. Outside of the relationship to core power analyses and ultimate operational predictability, density is not considered a performance property. It is, however, a reasonably accurate predictor of the variation in other physical and mechanical properties, so trends in density distributions and comparisons with other properties are an effective preliminary evaluation of the quality of individual test specimens. Additionally, the dimensions and mass of specimens are required inputs for various other physical and mechanical property calculations. A comprehensive data set on density values is therefore a natural component of the overall database.

Flexure bars machined from graphite into parallelepipeds to strict tolerances are ideal for capturing elastic constants via resonant frequency measurements. In the longitudinal (flexure) mode, the resonant frequency of vibration is indicative of the elastic modulus or dynamic Young's modulus. Figure 2 shows both the density of the candidate grades tested thus far (a) and the measured dynamic Young's modulus via resonant frequency from impulse excitation (b). As the previous paragraph describes, an indication of the general relationship between grades exhibited by the modulus measurements is revealed by the density distributions. NBG-18, for example, has both the highest density and highest overall modulus values, while IG-110 has the lowest values in both measured properties. This is not entirely surprising, as the calculation for modulus from resonant frequency measurements takes specimen dimensions and mass into account so the resulting values are directly related. As revealed in a comparison between the two distributions, however, the relative scale of the distributions from the two different measurement data sets do not line up. This shift in values is therefore a product of the different resonant frequencies being measured, along with the resulting different elastic moduli.



(a)



(b)

Figure 2. Density (a) and dynamic Young's modulus (b) comparisons between the candidate graphite grades reveal similar trends in the overall property distributions.

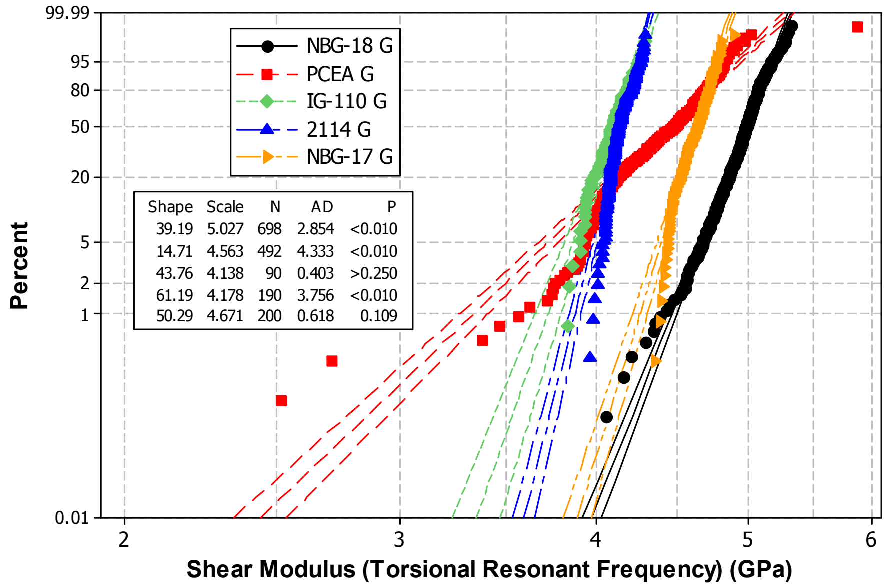


Figure 3. Shear modulus values for the five grades of graphite based upon the resonant frequency of vibration in torsion.

Measured fundamental frequencies through impulses that result in torsional vibrations provide dynamic shear modulus values (Figure 3). Just as with the density and dynamic Young's modulus property distributions, the extruded PCEA graphite has the highest level of variability (lowest shape parameter), while NBG-18 and IG-110 show the highest and lowest values, respectively. A calculation utilizing the elastic (*E*) and shear (*G*) moduli through the following relationship

$$\mu = (E / 2G) - 1$$

provides an approximation of the individual Poisson's ratio for the individual grades being evaluated. Using the Weibull scale parameter as the characteristic modulus value based upon the associated distributions, Table 1 provides scalar Poisson's ratio (μ) values for each of the candidate grades of graphite.

Table 1. Poisson's ratio values (μ) calculated from the Weibull scale parameters from the modulus measurements in flexural and torsional modes.

Poisson's Ratio by Grade

NBG-18	0.230
PCEA	0.178
IG-110	0.138
2114	0.204
IG-110	0.209

While the use of Weibull-based characteristic values to calculate a scalar Poisson's ratio will be a sufficient estimate for most analyses, the tracking of each data point by billet position allows for a more complete evaluation of the range of Poisson's ratio values based upon frequency measurements from individual specimens. Since each flexure bar is measured for both elastic and shear modulus values, each bar provides an individual Poisson's ratio value. The distribution of values based upon measurements at spatial points from each grade is provided in Figure 4.

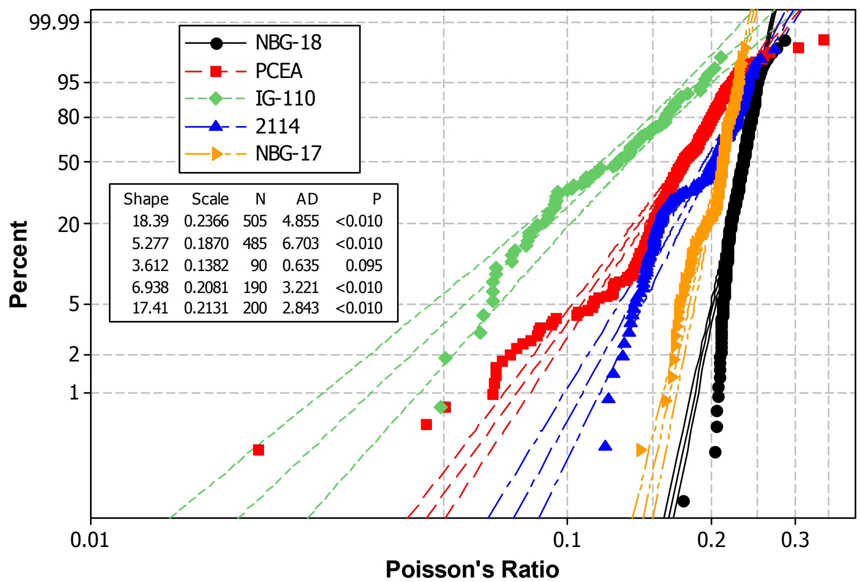


Figure 4. The distribution of Poisson's ratio values based upon the ratio of elastic to shear moduli measured in individual specimens.

4. MECHANICAL PROPERTY COMPARISONS

4.1 Compressive Testing

A compressive test property comparison between the five candidate billets of graphite is shown in Figure 5. A considerably wider distribution in measured compressive strength is seen in the extruded graphite, while the highest overall compressive strength is exhibited by the fine-grained isomolded grade 2114. In the case of measured strength, that quality is not due entirely to the grain size or formation technique, as the similarly fine-grained and isomolded IG-110 graphite has a lower range of compressive strength values than the SGL graphites NBG-18 and NBG-17. IG-110 also exhibits an inflection in the distribution at the lower compressive strength values that results in a section of the distribution with a much higher slope. This is indicative of a higher level of strength "consistency" in that particular grade, with strength values that do not drift into lower outlier values at the low end of the distribution. Contrast this quality to that of NBG-17, which, for the specimens tested thus far, exhibits an opposite inflection in distribution slope at the lower end. This change in the shape of the NBG-17 distribution is indicative of a number of specimens failing at much lower values than would be predicted by the Weibull distribution, likely due to some distribution of flaws that are disparate in nature that result in failure at lower stresses but are not inherent to the entire specimen population.

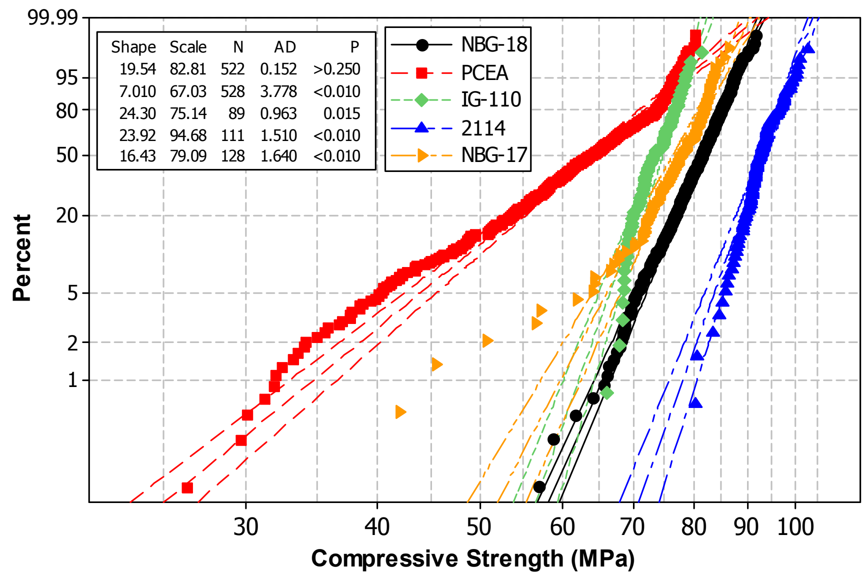


Figure 5. Compressive strength distributions for the candidate grades. The extruded PCEA graphite exhibits the highest level of variation as evidenced by the shallow slope in the Weibull cumulative distribution. The fine-grained isomolded grades yield high values (2114) and relatively low values (IG-110).

With regard to compressive deformation, Figure 6 shows the amount of compressive strain prior to failure in each of the grades. As the modulus values from the previous section showed, the IG-110 requires the lowest levels of applied stress for a given amount of elastic or pseudo-plastic deformation. By maintaining its integrity through a higher level of deformation, the stress applied to failure results in a much higher level of overall strain than is seen in the other graphite grades.

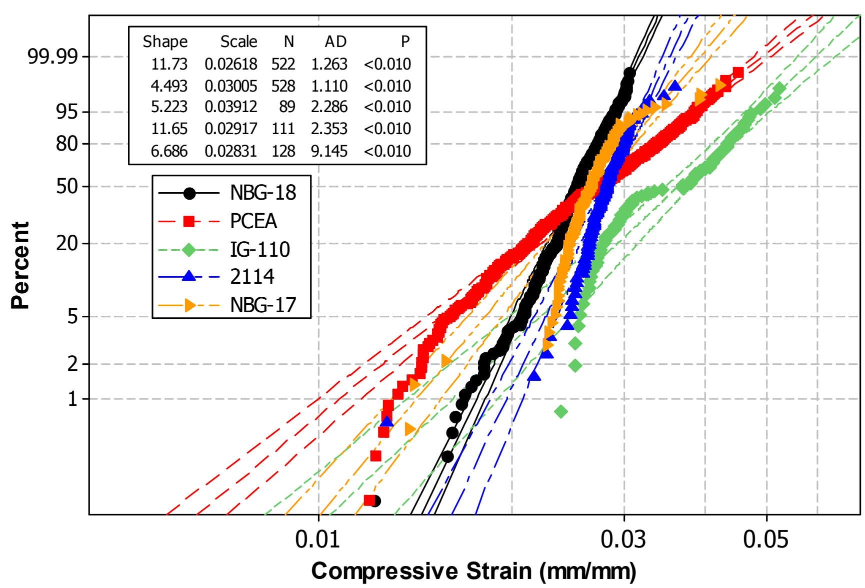


Figure 6. The property values for compressive strain at failure for each of the grades. The IG-110 exhibits the highest level of deformation prior to fracture in compression, which occurs at a relatively low stress as would be expected based upon the modulus values.

4.2 Flexural Testing

The distributions of measured flexural strength for the candidate grades are shown in Figure 7. As with other properties already presented, the extruded medium-grain PCEA has the highest level of variability in the measured data. The finer-grained grades of graphite (2114 and IG-110) demonstrate the highest level of strength in flexure, while the medium-grained NBG-17 exhibits slightly higher flexural strength than its larger-grained counterpart from SGL, NBG-18. 2114 and IG-110 show flexure strength characteristic values of over 41.3 and 36.6 MPa, respectively; NBG-18's characteristic value is the lowest at just over 28.4 MPa. ASTM D7219 provides minimum values for flexure strength that vary considerably based upon the formation technique and relative purity of the graphite. For the isomolded graphite, this value is 35 MPa, while extruded and molded (including vibration-molded) grades require strength levels greater than 21 MPa. The characteristic values are all above the required values for each of the candidate grades.

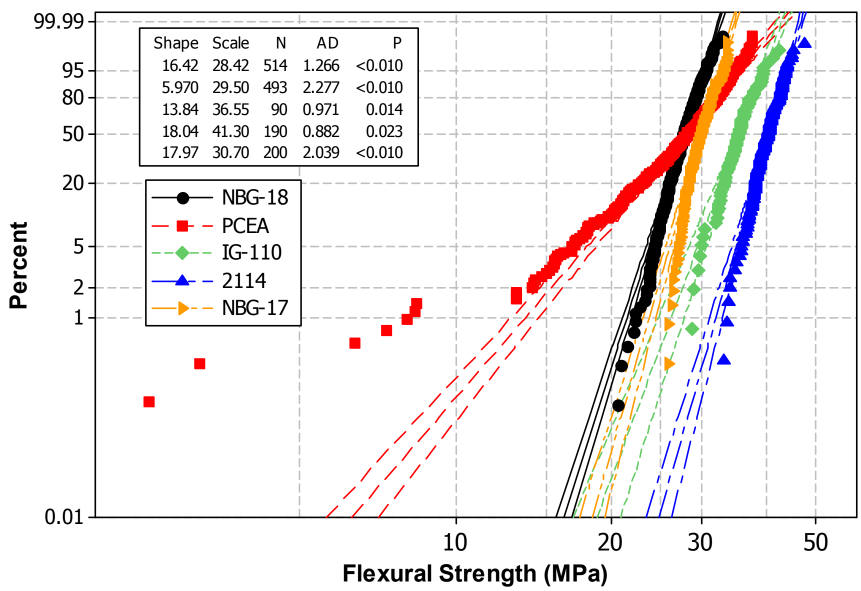


Figure 7. Flexural strength results for the specimens from each of the grades tested. A higher degree of variability and distribution of comparatively low outlier values exist for the extruded graphite, despite the range of flexural strengths that at the upper end are higher than the vibration-molded grades.

In the case of flexural testing, the flaw distribution leading to fracture in nuclear-grade graphite is given a limited amount of volume in which to be situated in order to be subjected to the highest stress levels. Testing in flexure subjects the flexure bars to a highest applied stress state between the two load rollers at the outer (lower) surface of the specimen under load. The finest-grained specimens are likely populated by flaw distributions that are homogeneous within the stressed volume, resulting in a high Weibull modulus value and elevated strength levels. By contrast, the less homogeneous extruded graphite has a widespread distribution of flaws, evident from the lower measured strength levels and lower Weibull modulus value. The lower modulus value is indicative of a larger number of specimens with critical flaws are captured in the high stress regions, resulting in lower relative strength levels. Those specimens that do not have critical (or otherwise limiting) flaws in the volume under the highest stress exhibit strength values that are higher in the upper range of the data distribution. These strength results indicate that the extruded graphite grade has a higher distribution of critical flaws (lower Weibull modulus values and lower measured strength values) than either the vibration-molded or isomolded grades.

Regarding the “fit” of the data sets to the Weibull distribution, it is notable that the non-extruded grades exhibit a relatively tight range of values similar in shape parameter that do not have an associated set of low “outlier” values at the lowest section of the distribution. This quality is indicative of the distinct potential for a better fit through the addition of a location, or threshold, parameter (μ) to the Weibull probability density function:

$$F(x) = 1 - e^{-(\frac{x-\mu}{\alpha})^{\gamma}}$$

Re-plotting the non-extruded grades in a three-parameter distribution (Figure 8) reveals a much better fit to the modeled values for shape, scale and threshold. The extruded grade, with its distribution of low outliers, would not benefit from a location parameter. In this case, a curve fit that attempts to represent potential values at very low probabilities would result in a non-descriptive (near-zero or negative) location parameter even though a “goodness-of-fit” test might reveal a better data fit than a standard two-parameter distribution. The addition of a location parameter to appropriate data sets, however, is potentially a crucial factor in conservative value estimates for nuclear-grade materials. The ideally-fitted location parameter essentially provides a “lowest probable value” below which no property values are expected, and the lowest conservative estimate is therefore equivalent to the location parameter value.

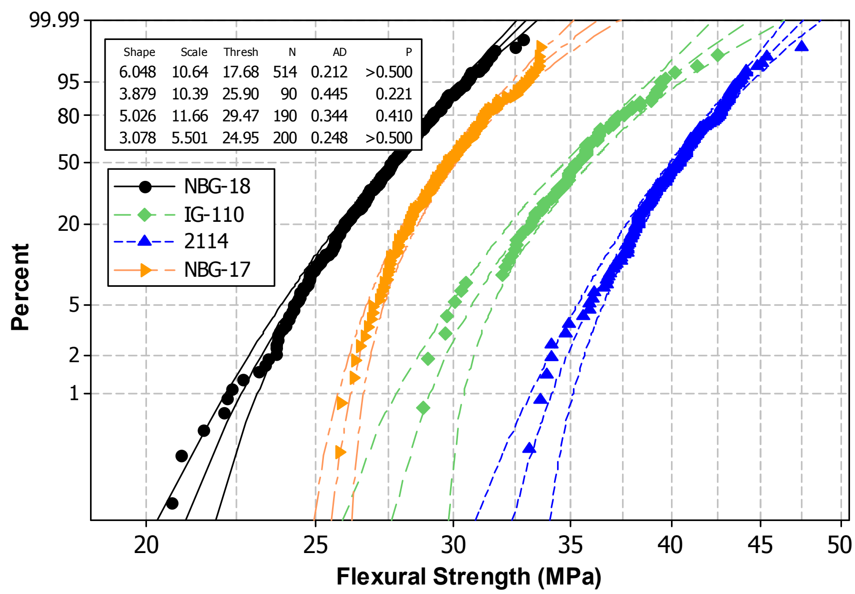


Figure 8. Adding the third parameter to the Weibull distribution function for location reveals a much better overall fit to the predicted distribution for the non-extruded grades.

4.3 Tensile Testing

The quantitative distribution comparison of tensile strength between the candidate graphites is shown in Figure 9. As with the other mechanical test values, the trend in properties indicates a wider distribution in measured values for the extruded grades and the tightest distribution for the fine-grained grades. The highest strength levels, similar to flexural strength results, are also exhibited by the fine-grained grades. With characteristic strength values ranging between 18.5 and 20.4 MPa for the extruded/molded grades and 25.5 (IG-110) and 30.6 (2114) MPa for the isomolded grades, all exceed the required strength levels required by ASTM Standard D7219, at 15 and 22 MPa, respectively.

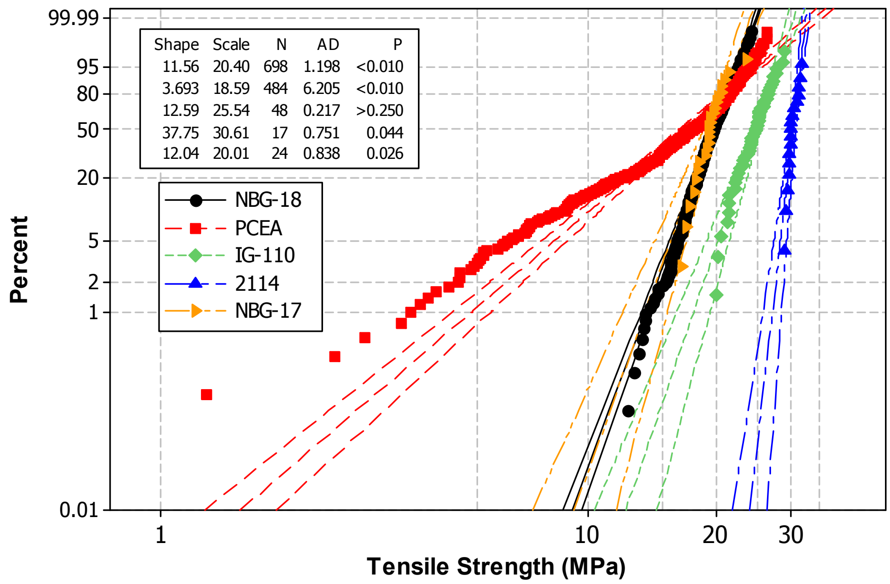


Figure 9. Tensile strength values for the billets tested show greater variability and a lower overall strength distribution in the extruded grade, and tightly distributed data and higher strengths for the isomolded grades with fine grain sizes.

Similar to the flexural test results, the tensile strength distributions appear to be much more accurately represented through the addition of a location parameter to the Weibull distribution. Figure 10 shows the re-plotted distributions and the associated fits to the three-parameter model.

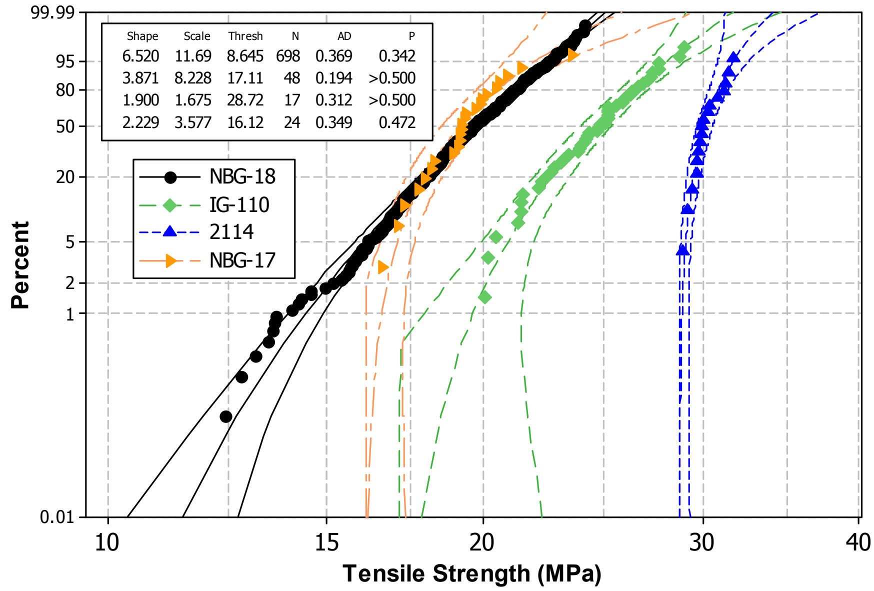


Figure 10. The addition of a threshold or location parameter to the data for the non-extruded grades results in a much better overall fit.

5. TESTING CROSS-CORRELATIONS

Based upon the flaw size, orientation, and distribution in nuclear-grade graphites, unsurprising testing anomalies have been noted in which the testing technique employed to ascertain a specific material property may not yield the same result as another test technique utilized to measure the same property. In determining elastic constants, the resonant frequency technique described in an earlier section is an effective means for measuring the elastic response due to impulse excitation. A more direct measurement of the relationship between stress and strain is recorded with each tensile test, in which the Young's modulus is the ratio between the stress applied by the load frame and the strain measured with an attached extensometer. The direct stress-strain relationship captured during the execution of individual tensile tests utilizes the two axes being plotted and is determined via the relationship $E=\sigma/\varepsilon$. Figure 11 shows the shift in values between modulus measurements using the two different techniques. In all cases, the measured dynamic Young's modulus is higher than the modulus measured during tensile loading. The fact that the distribution shift is not a straightforward linear correction provides an opportunity to determine whether qualities of individual nuclear graphite grades differ with regard to the interactive mechanisms, whereby the bonds in a graphite volume stretch elastically under load. Resonant frequency testing is based on the constructive interference between planar shock waves that propagate in a manner consistent with the elasticity in the atomic bonds of the graphite being tested, while the modulus measured during tensile testing is based upon bonds stretching throughout the entire volume of the gage section. Experience with numerous NBG-18 and PCEA specimens has shown that modulus testing based upon the *minimum* amount of interactive bond stretching at any given moment of the measurement – that acquired through the measure of sonic velocity, which is based upon a single planar sound wave – exhibits the highest overall distribution of measured modulus values. As shown in Figure 11, the shifts in shape and scale values between each of the candidate grades is not consistent, providing valuable information for the development of mechanistic models based upon the interaction of the actual graphite material and the inherent voids, cracks, and internal flaws that are interacting with the surrounding graphite during the deformation process.

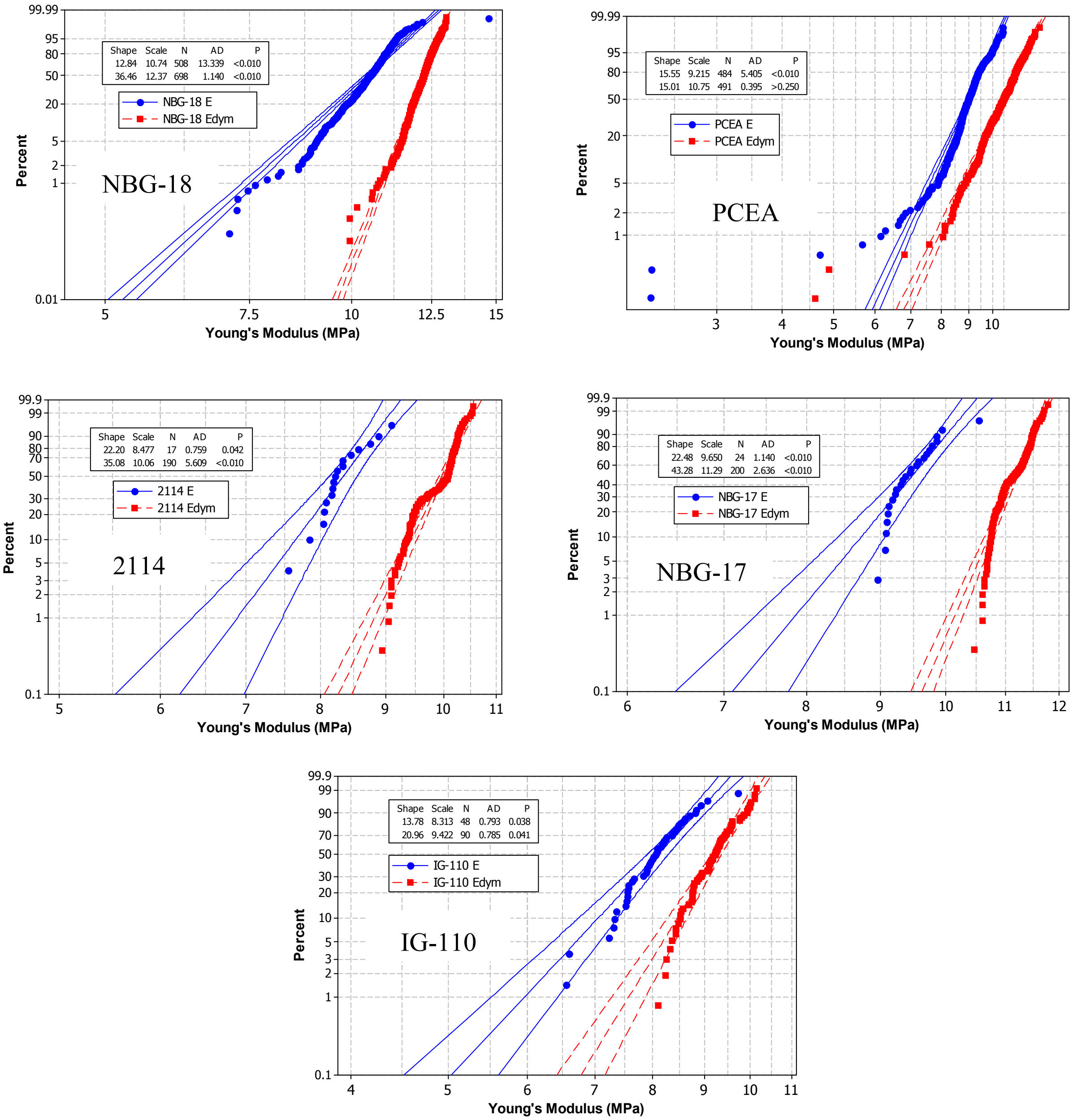


Figure 11. Plots by grade of the elastic modulus values gained directly from tensile test stress-strain curves (E) and through fundamental frequency measurements (E_{dym}). The techniques reveal differences in measured values likely based upon interactions with voids, cracks, pores and other types of flaws inherent to graphite, and the manner in which those interactions occur based upon the volume of graphite being sampled during the performance of the test.

6. SUMMARY

The comparative strength and elastic properties of each candidate grade of graphite in the USA VHTR program – NBG-18, NBG-17, PCEA, IG-110, and 2114 – have been compiled based upon the data collected to date in the Baseline Graphite Characterization program. Continued evaluations beyond the 5,000 specimens that have already been tested to failure at this point in the program will provide additional clarity to the trends that are being uncovered through the analysis of data performed on the completed specimens.

Weibull distributions of the graphite properties are the main means of determining the characteristic values that facilitate a thorough analysis of the combined data sets. Mean strength values are, as expected, higher for the finer-grained isomolded grades (IG-110 and 2114) than for the extruded or vibration-molded grades. The extruded grade almost universally exhibits a larger variability in measured values, underscoring the necessity for additional billets to be tested from separate lots. A high propensity for large voids was noted in the first lot of PCEA graphite to be obtained for the program, and the subsequent lot that was ordered provided the opportunity for the manufacturer to address the issue if it was related to the production process.

More important to the overall goals of the Baseline Graphite Characterization than measured values, however, are the associated distributions of the ever-growing data sets and their eventual applicability to the Advanced Graphite Creep distributions on the same or similar values measured pre-and post-irradiation. Accurate distribution models can continue to be refined as more data is collected across multiple grades of graphite, although it is expected that the relative relationships between the grades being compared have been represented by a sufficient data population to draw general conclusions about overall property/distribution differences.

7. REFERENCES

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