



Baseline Characterization Database Verification Report—NBG-17 Billet V104

June 2024

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and David Cottle

Idaho National Laboratory



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June 2024

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INL ART Program

**Baseline Characterization Database Verification
Report—NBG-17 Billet V104**

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Technical Reviewer: (Confirmation of mathematical accuracy, and correctness of data and appropriateness of assumptions.)



6/19/2024

Michael E. Davenport
ART Project Manager

Date

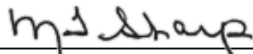
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SUMMARY

The purpose of this report is to present data collected in the Baseline Graphite Characterization Program, which is directly tasked with supporting the Idaho National Laboratory's research and development efforts on the Advanced Reactor Technologies Program. This program populates a comprehensive database that reflects the baseline properties of nuclear-grade graphite regarding individual grade, billet, and position within individual billets. The physical- and mechanical-property information being collected will be transferred to the Nuclear Data Management and Analysis System (NDMAS), and that database will help populate the handbook of property data available to member nations of the Generation-IV International Forum.

Transfer of these data from the applicable technical lead to the dissemination databases available to other end users requires a full review of the test procedures and data-collection efforts through an analysis of the multiple summary spreadsheets and values being collected. This report represents the analysis for NBG-17 Billet V104 and facilitates release of associated data to the NDMAS custodians.

Millions of raw data points have been collected during testing and quantification analyses for these billets. The summary scalar property values and supplementary traceability data are collected into comprehensive spreadsheets. Data sets are composed of single billets of graphite for any given grade, organized by mechanical test-specimen type, and further subdivided into individual spreadsheet tabs according to the specific test or evaluation being performed.

A direct analysis of properties was not conducted, and this report does not provide information on the validity or performance characteristics of the graphite itself. Rather, this report is intended as a verification of the completeness of actual data collected in accordance with PLN-3467, "Baseline Graphite Characterization Plan: Electromechanical Testing," [1] and PLN-3348 "Graphite Mechanical Testing," [2] and their representation of the measurement and test results with sole regard to the graphite billets under evaluation.

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ACRONYMS

AGC	Advanced Graphite Creep
ART	Advanced Reactor Technologies
CTE	Coefficient of thermal expansion
COV	Coefficient of variation
INL	Idaho National Laboratory
NDMAS	Nuclear Data Management and Analysis System

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Baseline Characterization Database Verification Report—NBG-17 Billet V104

1. INTRODUCTION

The Advanced Reactor Technologies (ART) Project Graphite Research and Development Program generates the extensive quantitative data necessary for predicting behavior and operating performance of available nuclear graphite grades. To determine in-service behavior of graphite for the latest proposed designs, two main programs are underway. The Advanced Graphite Creep (AGC) Program provides a set of tests that are designed to evaluate the irradiated properties and behavior of nuclear-grade graphite over a large spectrum of conditions, based on the operating environment of the high-temperature reactor core. [1] A limited amount of data can be generated on irradiated material because of the availability of space within the Advanced Test Reactor and the geometric constraints placed on the AGC specimens that will be inserted into the reactor. To supplement the AGC data set, the Baseline Graphite Characterization Program provides additional data that will characterize inherent property variability in nuclear-grade graphite without the testing constraints of the AGC Program.[2] This variability in properties is a natural artifact of graphite due to the geologic raw materials that are used in its production. This variability is quantified, not only within a single billet of as-produced graphite, but also from billets within a single lot, billets from different lots of the same grade, and across different billets of numerous grades of currently available nuclear graphite.

This report covers the release of physical and mechanical-property data from a billet of NBG-17 graphite. Billet V104 is from the second NBG-17 production lot of SGL Carbon's vibrationally molded graphite grade. Grade NBG-17 is fabricated from a pitch coke filler particle with a medium grain size of around 800- μm average filler particle diameter. Note that NBG-17 and SGL's grade NBG-18 have the same fabrication processes except the filler particle size for NBG-18 is twice the particle size of NBG-17 (around 1600 μm in diameter). Thus, by comparing the material properties of NBG-17 to the larger grain-sized NBG-18, the changes should be attributed to the grain size effects.

The baseline mechanical properties database for this billet, plots of which are included throughout this report, is composed solely of scalar results from each of the different evaluations (i.e., mechanical testing and physical properties) in summary form, and comprises tabbed spreadsheets occupied by more than 90,000 cells of individual characteristics or property values and associated tagging information.

2. DATABASES

The data sets being generated for the Baseline Graphite Characterization Program consist of properties collected on standard ASTM International-based mechanical test specimens, as shown in Figure 1. Details of specimen tracking, traceability, process flow, and the techniques being employed to facilitate those activities are provided in INL/EXT-10-19910. [3] For ease of reviewing the applicable data in this report, an example of a sectioning diagram for NBG-17 graphite, along with the applicable specimen-identification codes, is provided as Figure 2. This figure is representative of a quarter of a single sub-block of graphite from this billet. Detailed drawings of NBG-17 graphite billet sectioning can be found in Idaho National Laboratory (INL) Drawings 780553 and 780932. [4,5]

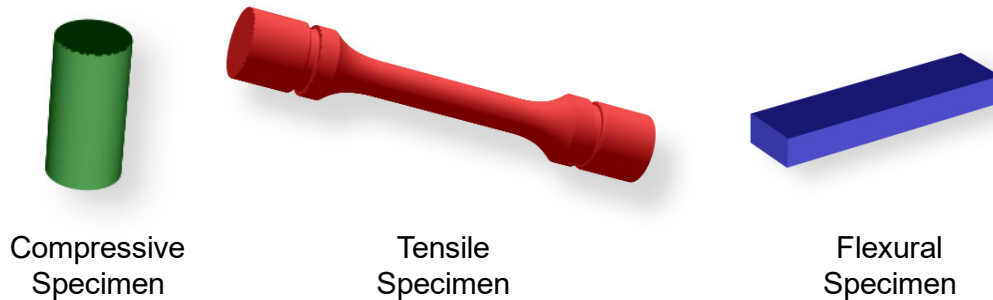


Figure 1. The three types of mechanical test specimens will be machined from stock graphite and provide the basis for material-property evaluations.

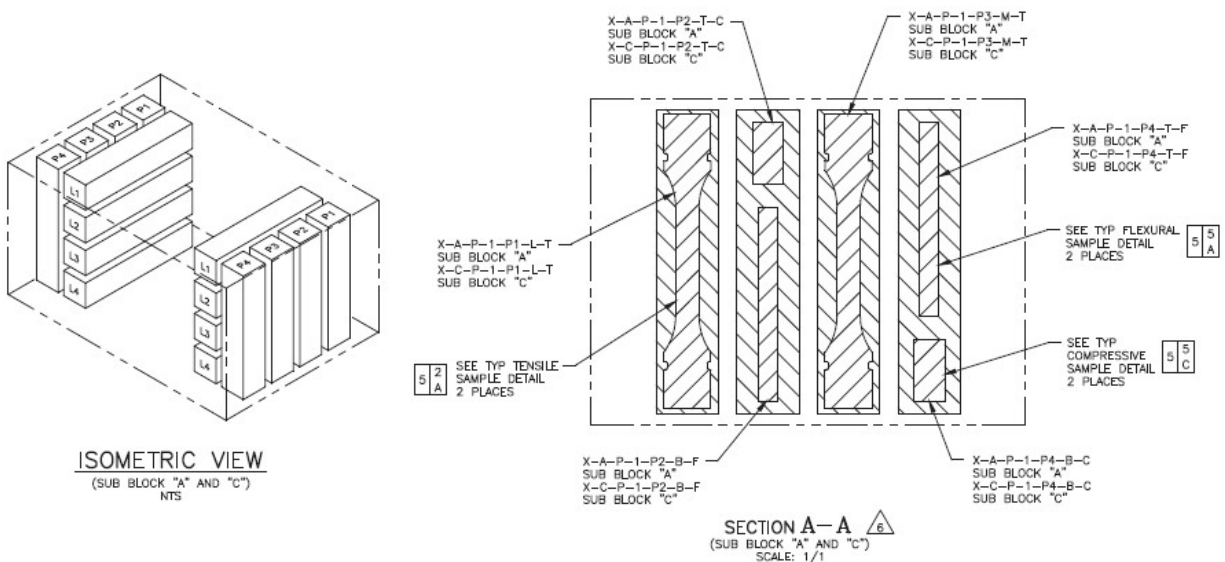


Figure 2. Individual specimen extraction and tracking identification from NBG-17 Billet V104.

Sections of this report covering each of the individual datasets for this billet are divided by mechanical test-specimen type—e.g., compressive, flexural, or tensile. They are organized so they present data in graphical form. Graphic representations are not sorted in any way aside from the actual order in which they were tested, which was randomized for the express purpose of minimizing test anomalies based on actual test timeframes. Some expectation of variation in the property values exists, but individual data points that fall within a reasonable property-value range are considered acceptable. Comparisons of extreme values with other associated properties—e.g., a comparison of maximum tensile load values with measured strain to determine whether they are related by the expected elastic modulus—are carried out where applicable. Each of these comparisons and analyses may not be explicitly included in this report. However, the process-control charts with standard-deviation values and/or property-trend charts for the various characteristics being measured are included both in this section as well as the appendices. The mean, standard deviation, and coefficient of variation (COV) are listed as well. (There, the ± 1 , 2, and 3 standard deviations are represented by the yellow, orange, and red dotted lines, and the mean is represented by the green line.)

A goal of the Baseline Graphite Characterization Program is to identify and quantify interbillet variation. However, the focus of this analysis is to compare values from complete data sets to quickly identify outlying points. One example would be a zero value for a specific property—quickly identifiable on a test-result trend graph—providing an indication that the specific spreadsheet cell is improperly

empty. Another example would be a large disparity between a limited number of points on that same test-result trend graph that results from missing values in other cells—i.e., dimensional measurements from which final properties are calculated. This verification will couple those observations with a comprehensive data scan of individual points to determine whether the data set can be considered complete and the scalar summary points provided to the NDMAS are appropriately representative of the billet under evaluation.

2.1 Compression-Specimen Database (NBG-17 V104)

2.1.1 Compression Testing

Compression testing was performed per ASTM C695-15 [6] and PLN-3467.[1] Figure 3 shows the maximum applied load for each of the 284 compression specimens from Billet V104. As was mentioned previously, some variation in graphite properties is expected, and this variation is reflected in the difference in test-frame loading. The recorded load values (Figure 3) correlate directly with the compressive stress values (Figure 4), confirming the stress calculations were performed correctly. Four of the specimens had slightly low load values, which led to low strength values. These low values are attributed to material variation and will be kept in the database. An additional check of critical-property values is the measured deflection (Figure 5) of the loading surface, or upper platen, as measured by a calibrated deflectometer. Within geometric variations, the deflection should reflect the calculated compressive strain, as shown in Figure 6. Figure 5 shows a displacement value that lies outside of the control limits. This value produced a low strain value in Figure 6 that is also outside the control limits. This discrepancy is also believed to be attributed to material variation and will also be added to the database. Other plots of supporting data from the compressive specimens are shown in Appendix A, “Additional Compression Specimen Database Plots (NBG-17 V104).”

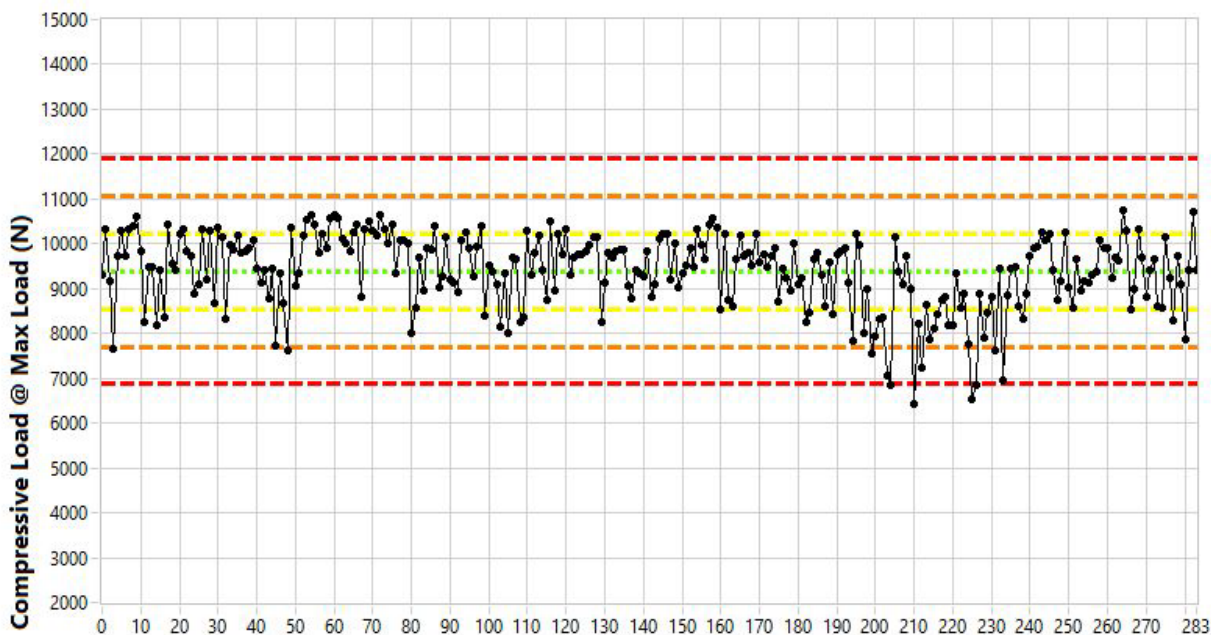


Figure 3. Compressive load at max load (N), mean = 9384, standard deviation = 838, COV = 8.9%.

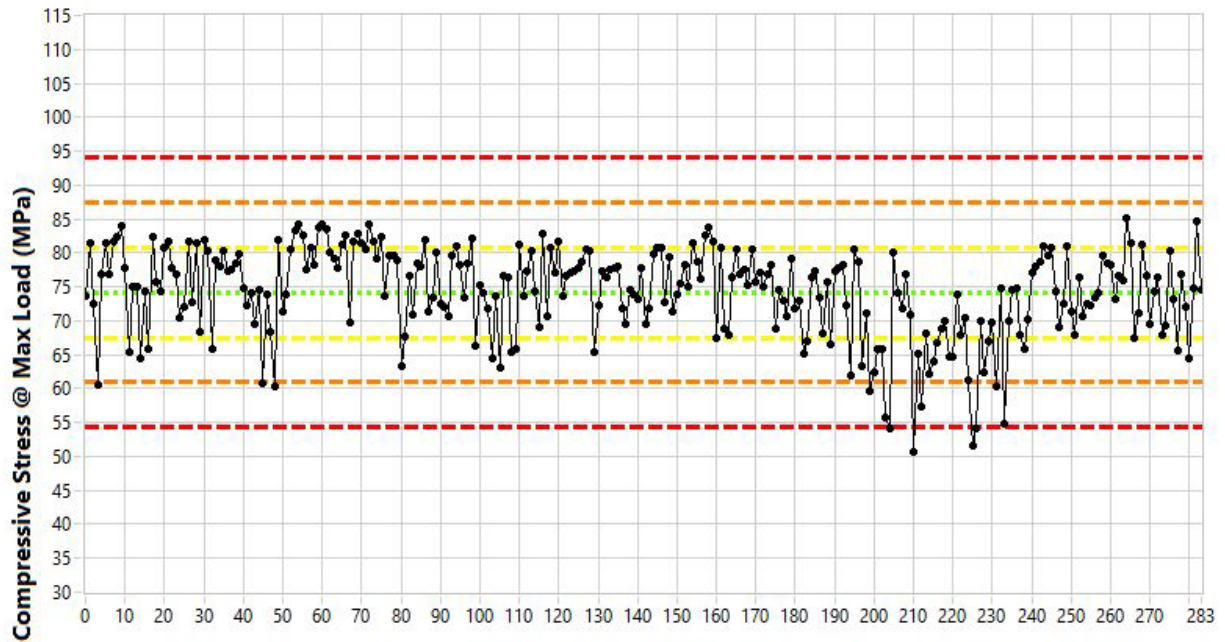


Figure 4. Compressive stress at max load (MPa), mean = 74.2, standard deviation = 6.6, COV = 8.9%.

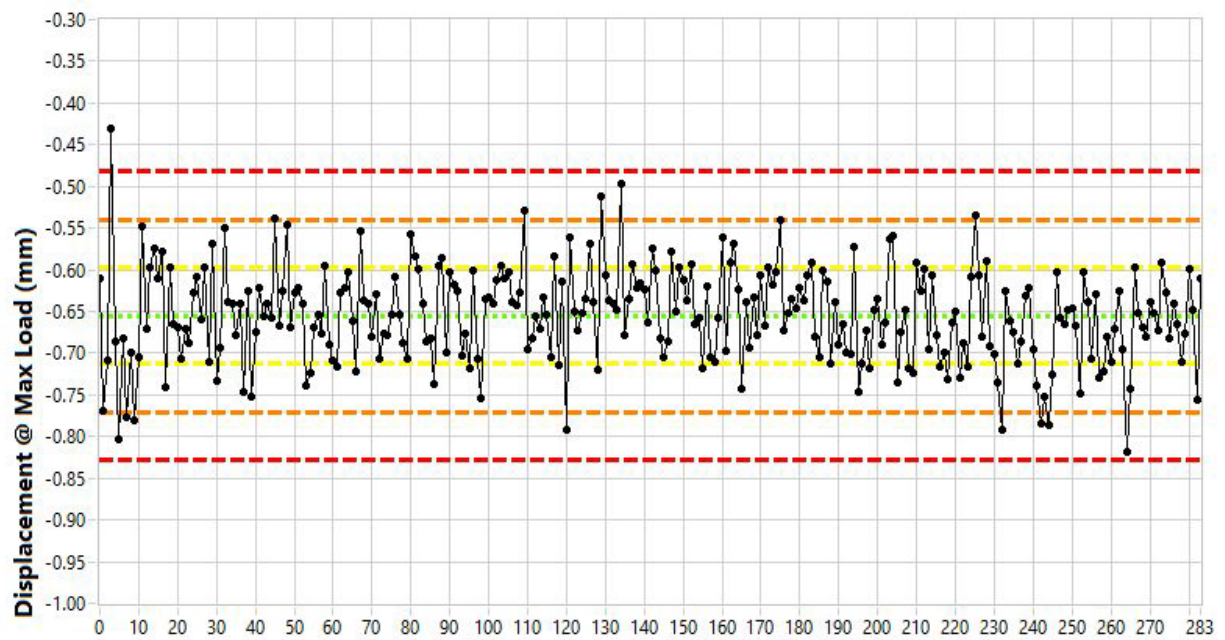


Figure 5. Displacement at max load (mm), mean = -0.66, standard deviation = 0.058, COV = 8.8%.

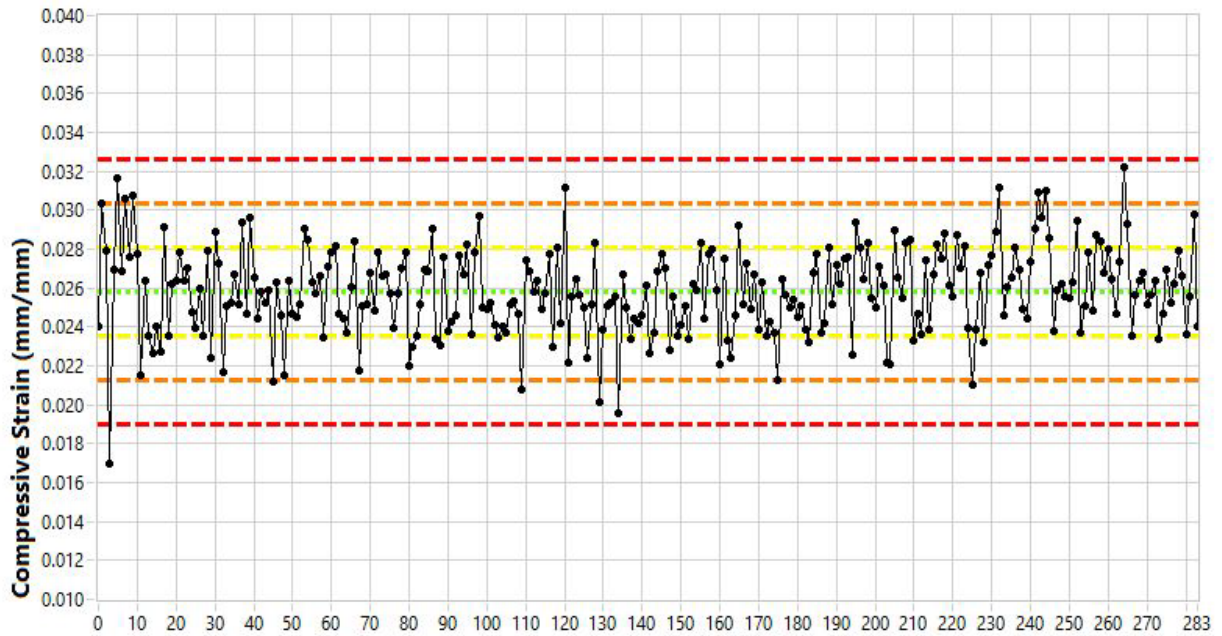


Figure 6. Compressive strain (mm/mm), mean = 0.0258, standard deviation = 0.0023, COV = 8.9%.

2.1.2 Fracture Surface Categorization

Fracture surfaces from compressive specimens offer an additional opportunity to collect scalar data that can be sorted with respect to graphite type and position. To allow for consistency in what is essentially a qualitative attribute, a description of each of the fracture types is provided to the user of the Graphite Mechanical Properties Data Acquisition Software. Figure 7 shows a screen shot of this categorization, along with the distribution of the recorded fracture categories for each of the 284 compressive specimens from NBG-17 Billet V104 (with no anomalous values indicative of an unallowable characterization).

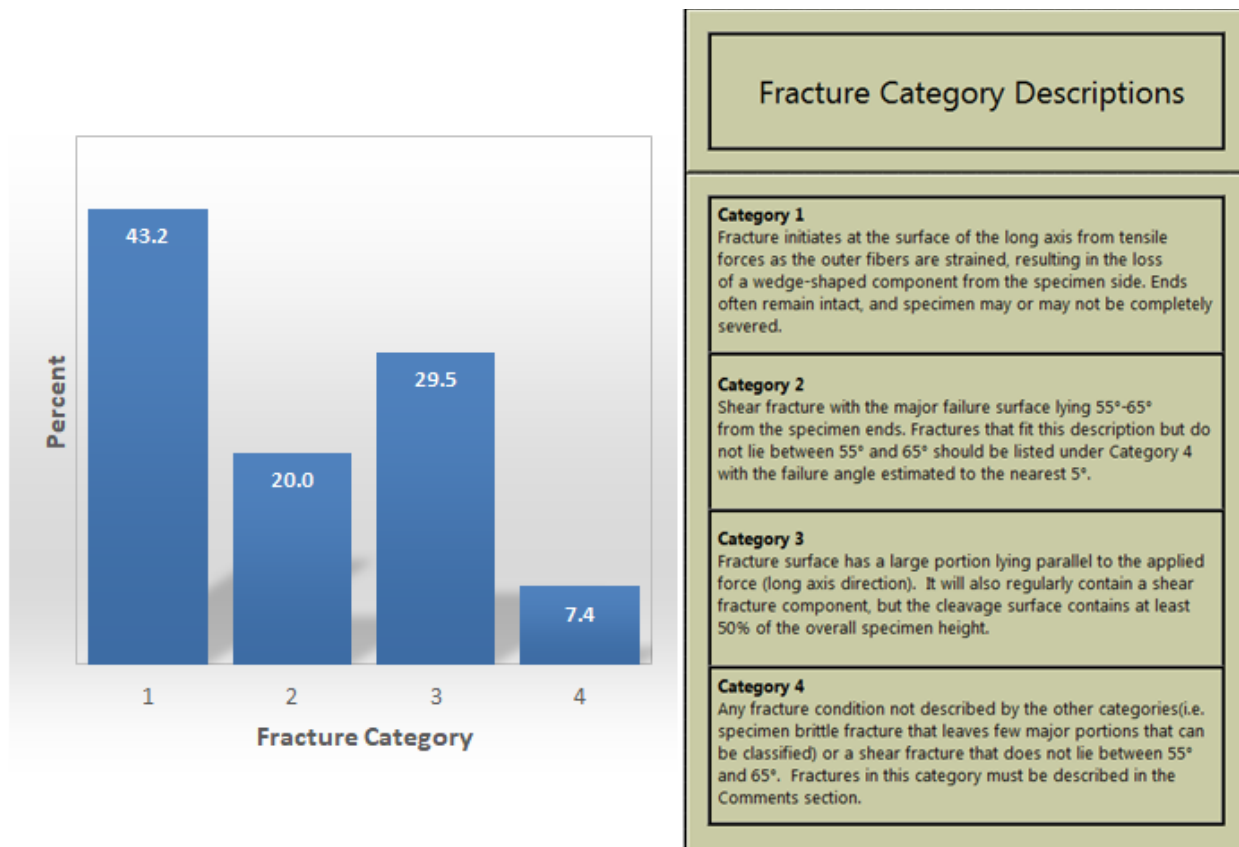


Figure 7. Fracture categorization results and description.

2.1.3 Electrical Resistivity, Modulus, and Coefficient of Thermal Expansion

Resistivity measurements were performed on 70 specimens before they were broken in compression. These values can be seen in Figure 8. There was one outlier in the resistivity measurements, but further investigation into this data point did not yield a reason to withhold it from the database.

Young's and shear modulus by sonic velocity (performed on 71 specimens) and Young's modulus by sonic resonance (performed on 200 specimens) tests were performed on a subset of compression specimens before they were broken. None of these data appear to vary significantly from known modulus values for NBG-17 graphite. These data are plotted in Figure 9 through Figure 11. Similar to that of the resistivity plot, there was a sonic resonance modulus value outside of the control limits in Figure 11. Examination of the specimen's other data revealed no reason to throw out this modulus data.

Figure 12 shows the coefficient of thermal expansion (CTE) from 70 compression specimens before they were broken. These data show a tight grouping over the 10 measurement temperatures. All the above tests were carried out via the appropriate ASTM standards.[7,8,9,10,11]

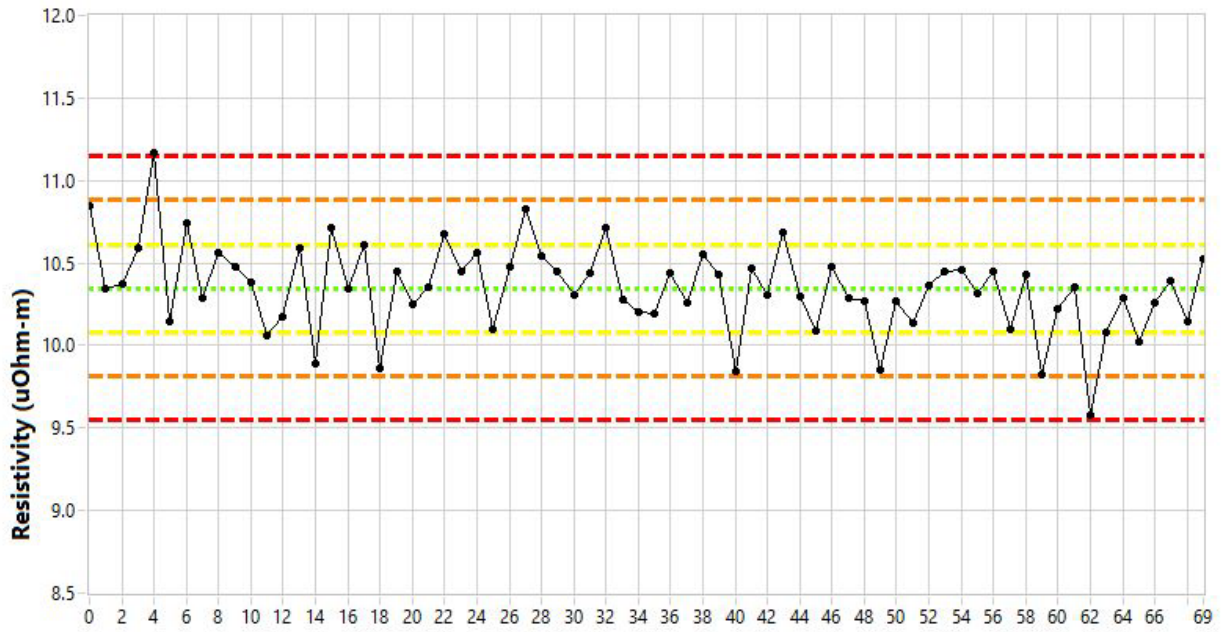


Figure 8. Resistivity ($\mu\text{Ohm-m}$), mean = 10.35, standard deviation = 0.27, COV = 2.6%.

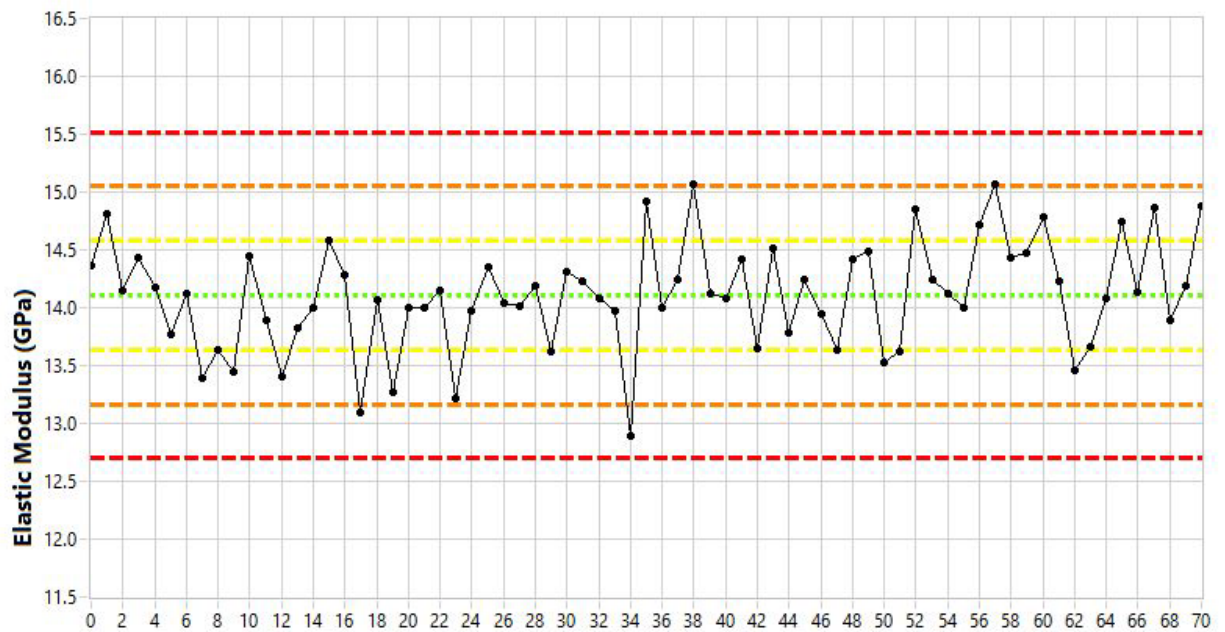


Figure 9. Elastic modulus by sonic velocity method (GPa), mean = 14.1, standard deviation = 0.47, COV = 3.3%.

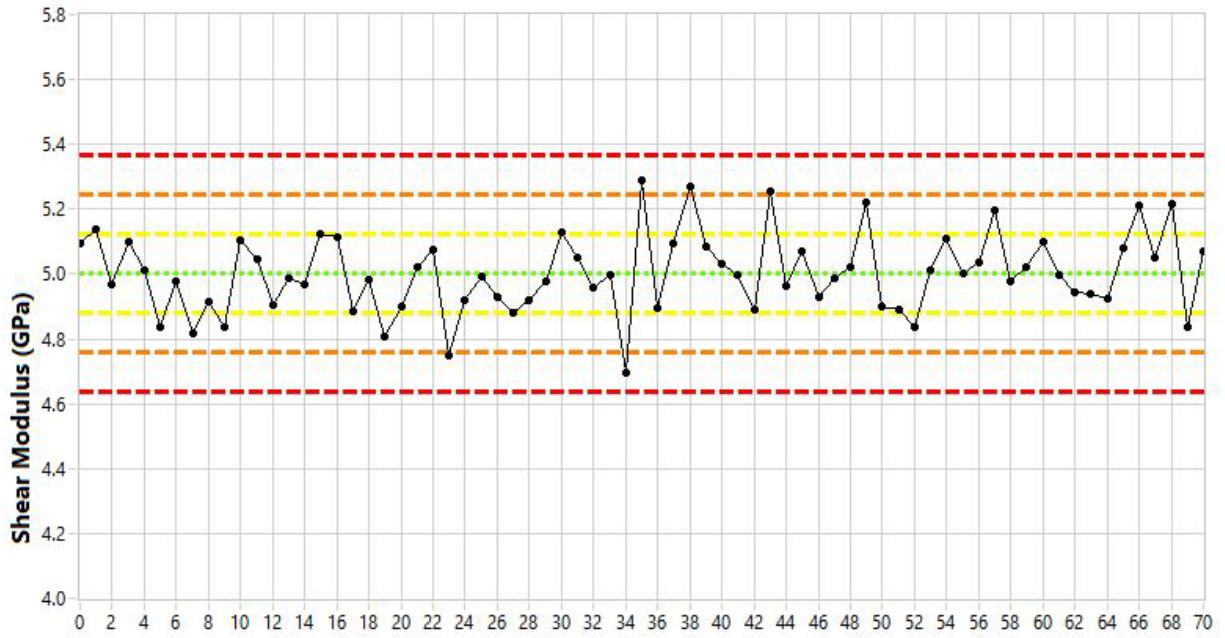


Figure 10. Shear modulus by sonic velocity method (GPa), mean = 5.0, standard deviation = 0.12, COV = 2.4%.

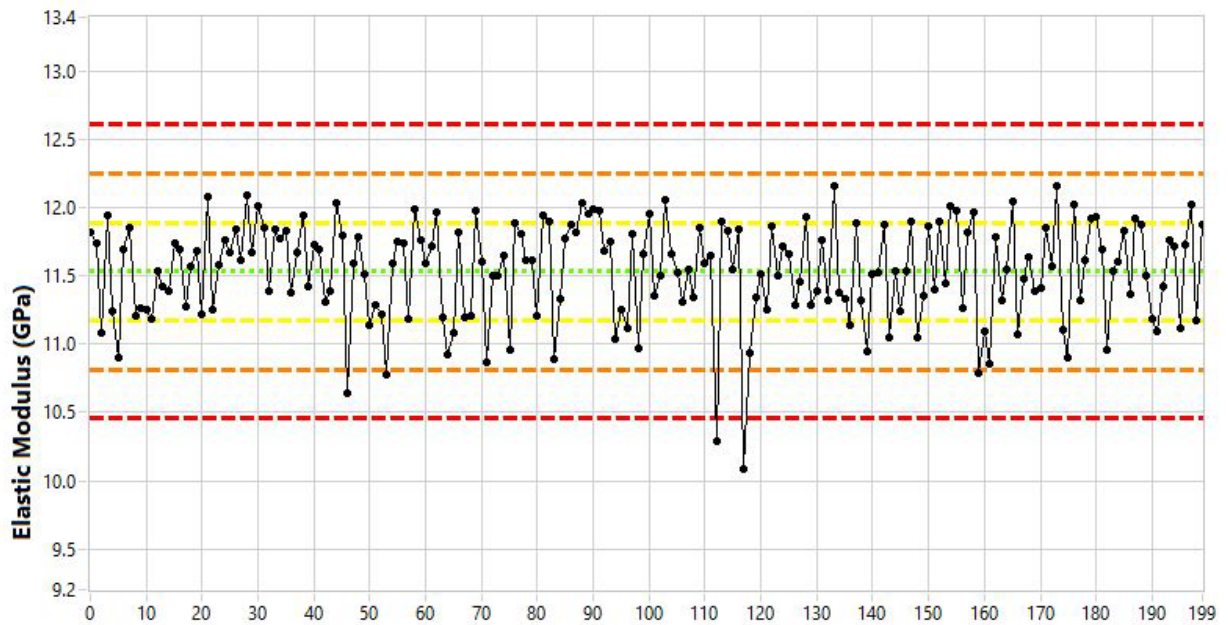


Figure 11. Elastic modulus by sonic resonance method (GPa), mean = 11.54, standard deviation = 0.36, COV = 3.1%.

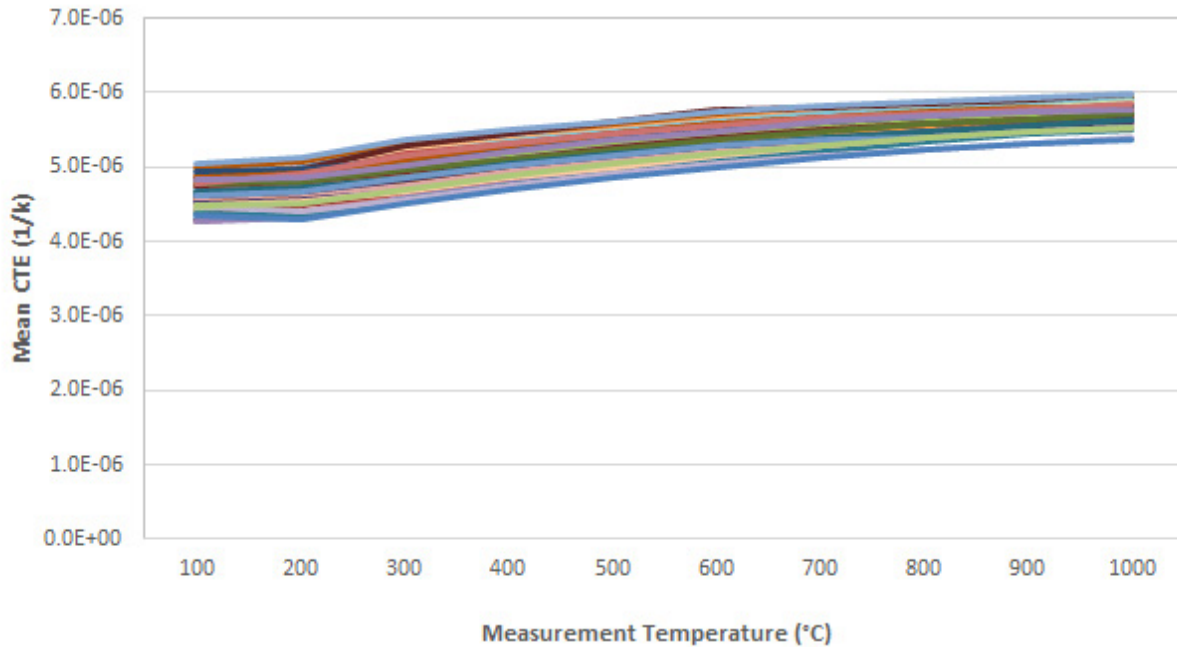


Figure 12. Mean CTE (1/K).

2.1.4 Density Values

The relatively simple geometric shape of the compressive specimens provides an opportunity to collect density data (per ASTM C559-90 [12]) for a large portion of the specimens extracted from each billet. While not true performance properties, density measurements are relatively straightforward to collect and are often reflective of bulk mechanical properties. The density values recorded for the compression specimens are shown in Figure 13. The two density outliers in Figure 13 were investigated and deemed acceptable since the mechanical test data for those specimens were within their control limits. But further analysis would need to be performed before its used in any kind of qualification.

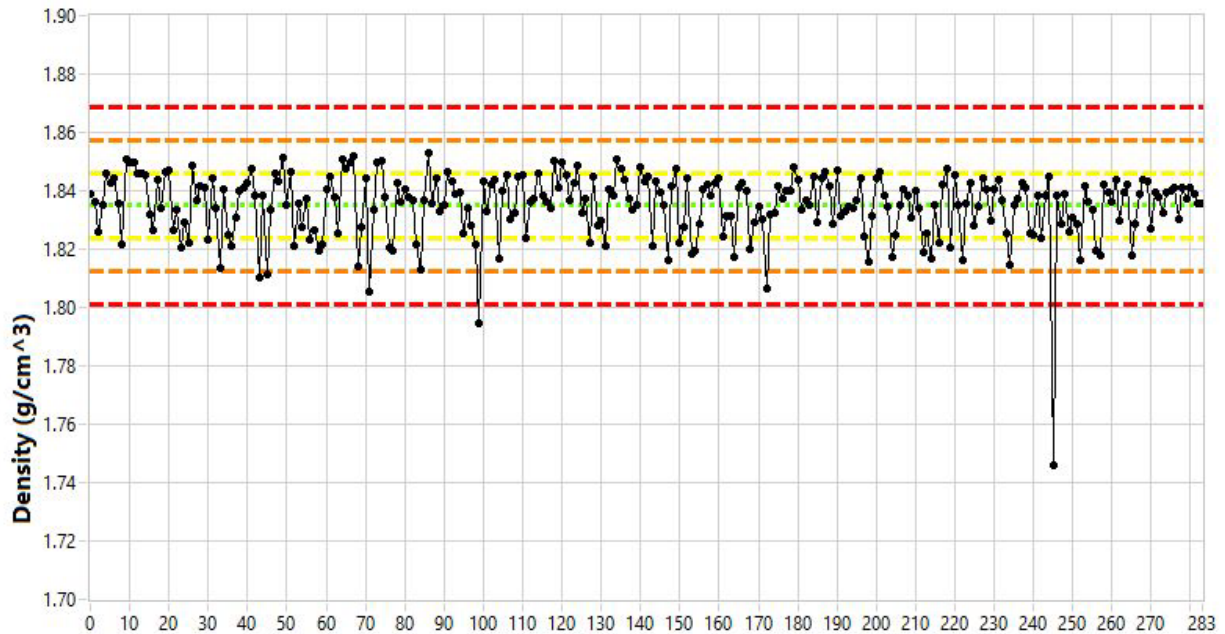


Figure 13. Density (g/cm^3), mean = 1.8349, standard deviation = 0.0113, COV = 0.62%.

2.2 Flexural Specimen Database (NBG-17 V104)

2.2.1 Flexural Testing

Flexural testing was performed per ASTM C651-91, [13] with clarifications to ambiguities in the standard identified in PLN-3467 and PLN-3348. [1,2] As with the presentation of compression-specimen results, test validation lies not only in the documented adherence to applicable test plans and standards, but also in the noted correlations between recorded test properties and analyses for extreme or anomalous values. Additional verification of test conditions can be accomplished through an analysis of the physical characteristics of the specimens.

Figure 14, Figure 15, and Figure 16 show the measured width, thickness, and length for all flexural specimens tested. Figure 14 shows that there is a step change in the width measurements of the last 63 specimens measured. This is because those specimens were machined about six months later by the machine shop. These specimen dimensions still meet the testing requirements of ASTM C651-91.

Figure 17 and Figure 18 show the relationship between flexural load and recorded flexural stress for the 284 specimens tested in flexure from NBG-17 Billet V104. Further comparisons and verification can be made with measured deflection (Figure 19), which will reflect an additional correlation with stress values through material elastic constants. Note the large measured mid-span deflection at max load value for specimen #12 in Figure 19 which is clearly outside of the outside of the control limits. This result is included in this report, but the usage of these measured values must be evaluated for future analysis.

Other plots of supporting data from the flexural specimens are shown in Appendix B, “Additional Flexural Specimen Database Plots (NBG-17 V104).” Note that 285 specimens had all the dimensional and property measurements performed on them. However, the load-frame software did not save one specimen’s data set. Therefore, there are only 284 data points on the mechanical test plots.

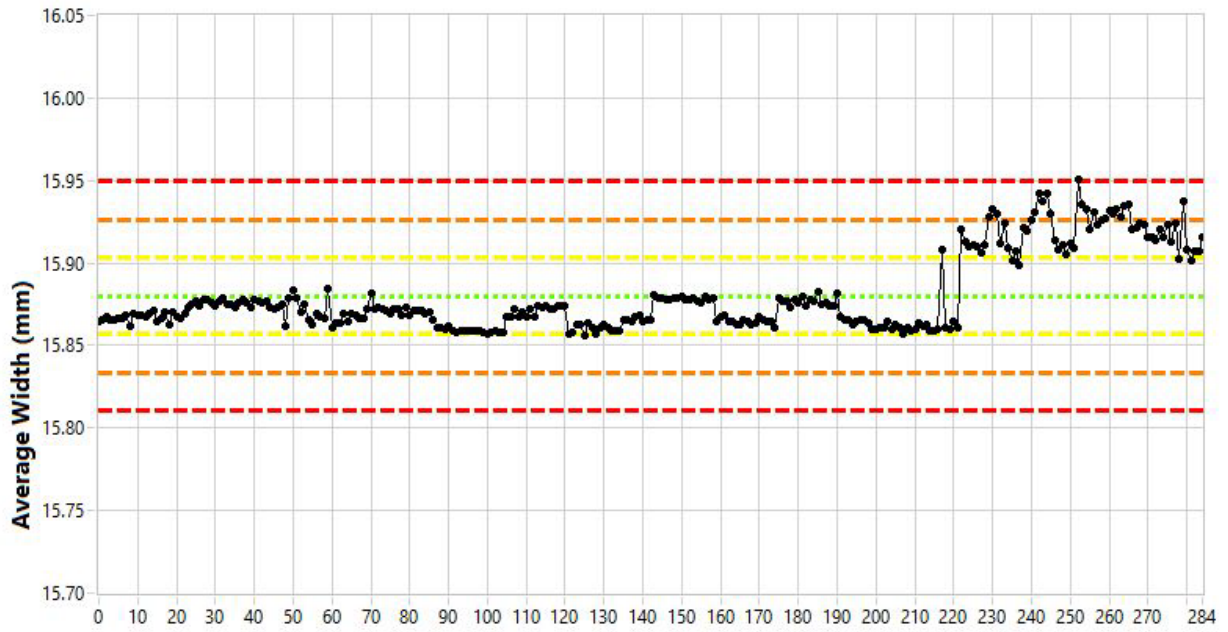


Figure 14. Average width (mm), mean = 15.88, standard deviation = 0.0231, COV = 0.15%.



Figure 15. Average thickness (mm), mean = 8.1296, standard deviation = 0.0113, COV = 0.14%.

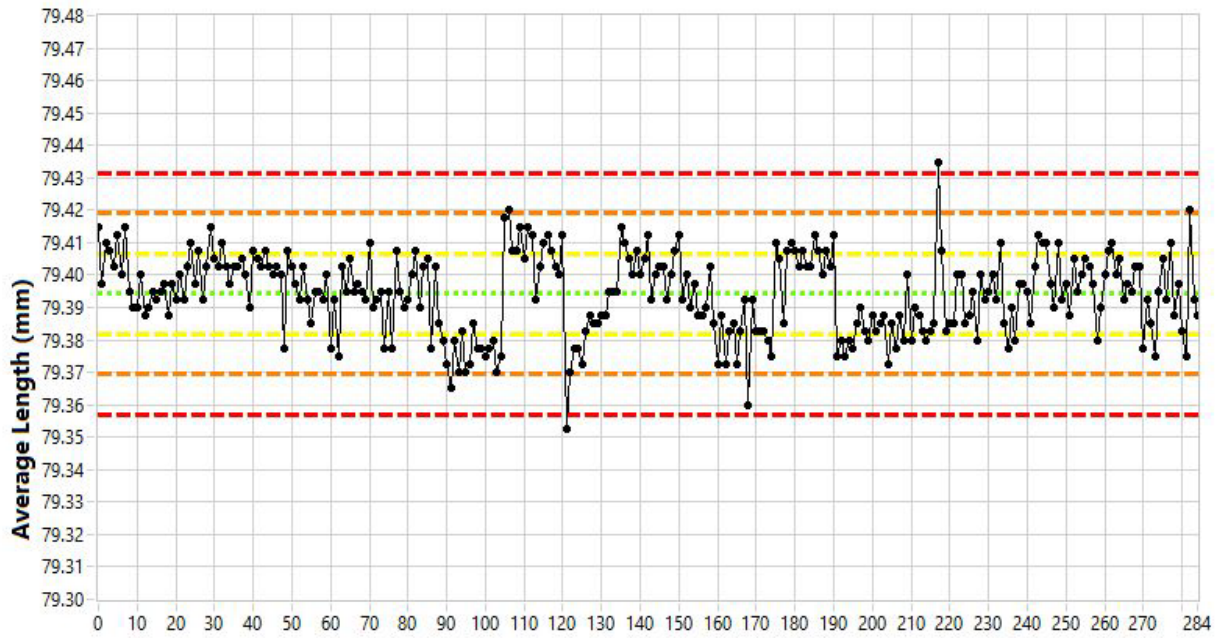


Figure 16. Average length (mm), mean = 79.394, standard deviation = 0.0124, COV = 0.016%.

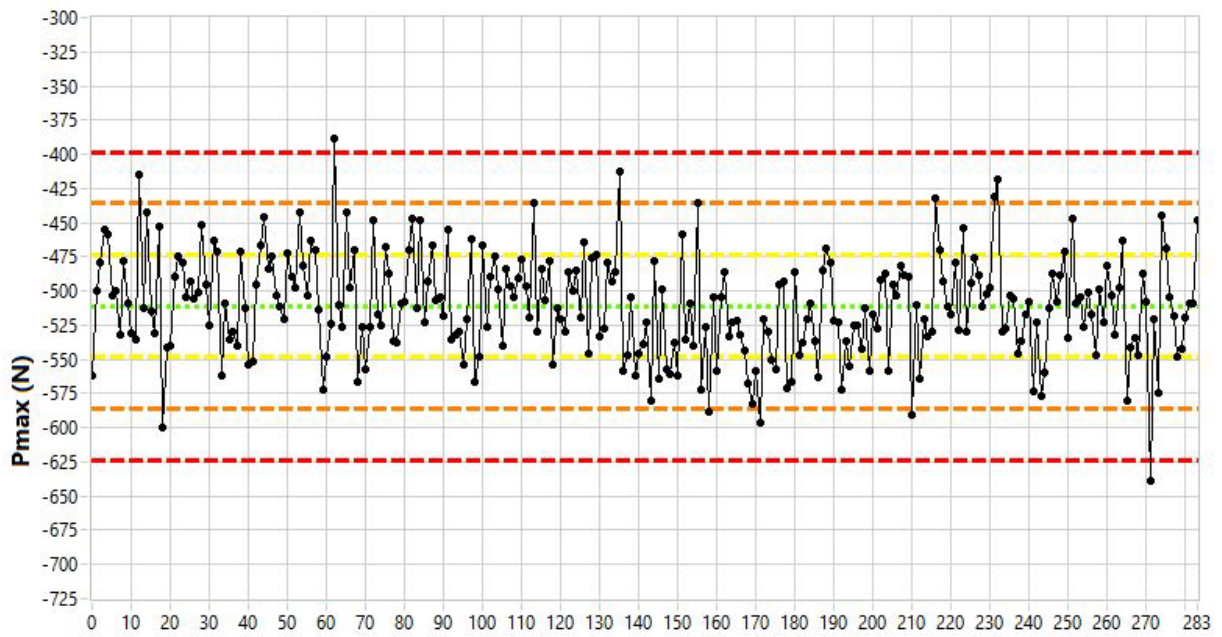


Figure 17. Max load (N), mean = -511, standard deviation = 37.5, COV = 7.3%.

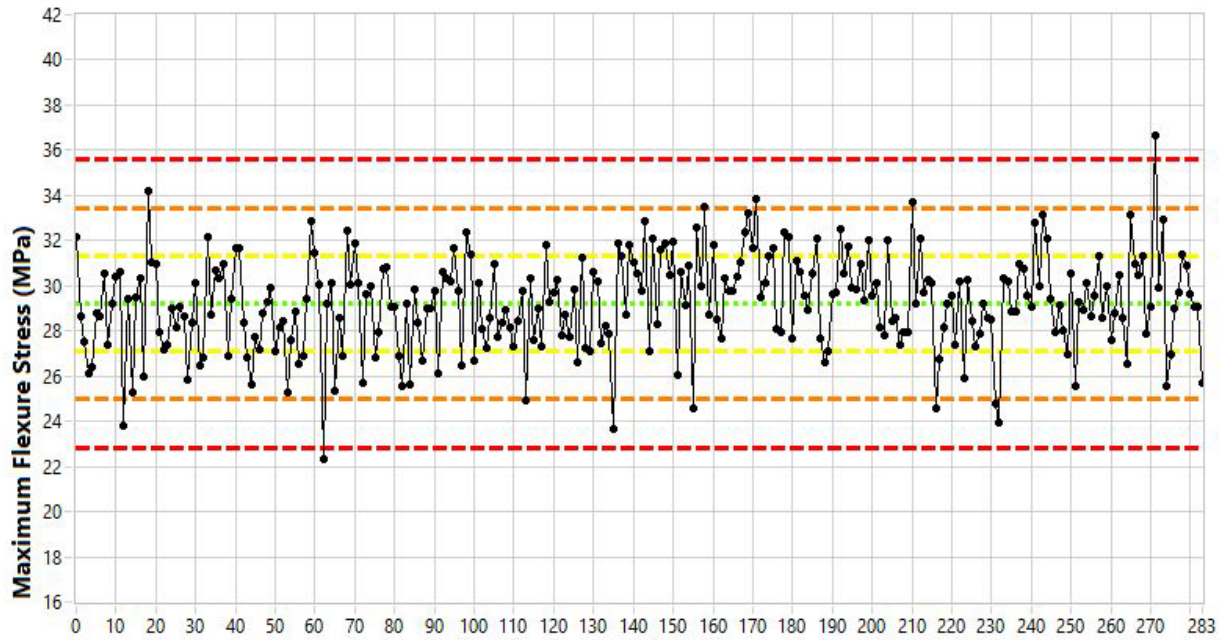


Figure 18. Maximum flexure stress (MPa), mean = 29.2, standard deviation = 2.1, COV = 7.2%.

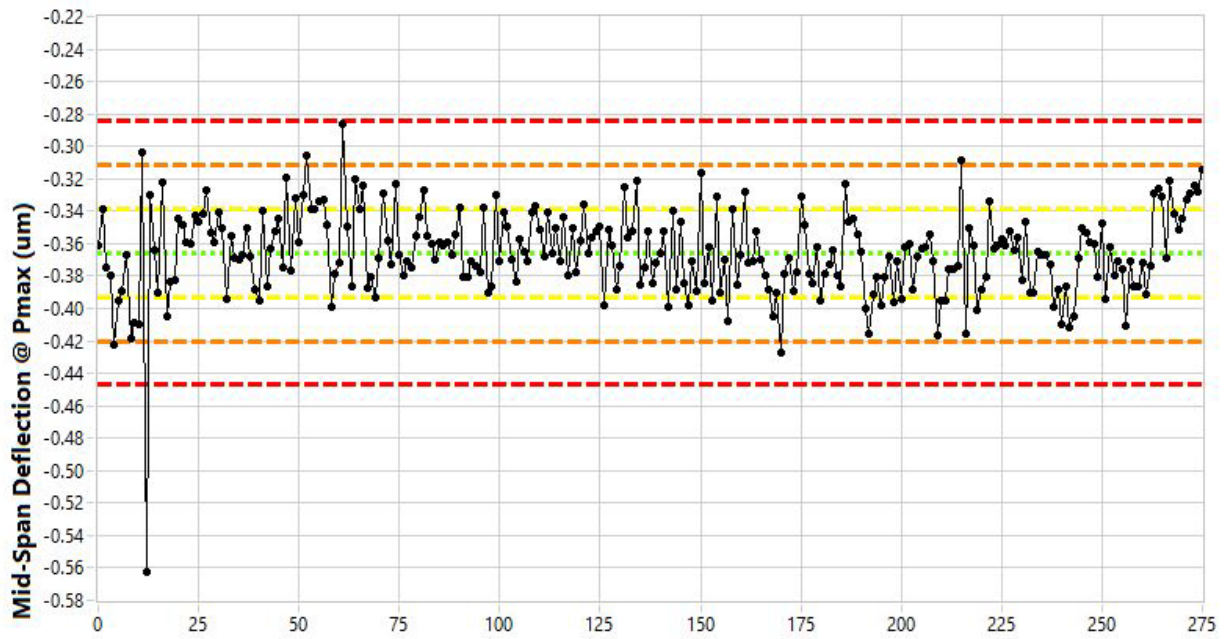


Figure 19. Midspan deflection at max load (μm), mean = -0.366, standard deviation = 0.0271, COV = 7.4%. Note, eight of the 284 specimens' deflectometer readings did not zero before the flexural test. Thus, those data points were not included in the control chart.

2.2.2 Density Values

Similar to the compression specimens, the flexural specimens' geometry facilitated an opportunity to measure density. Figure 20 shows density from the flexural specimens. All flexural specimens' data and associated deviations compare well with the compression specimens' density data.

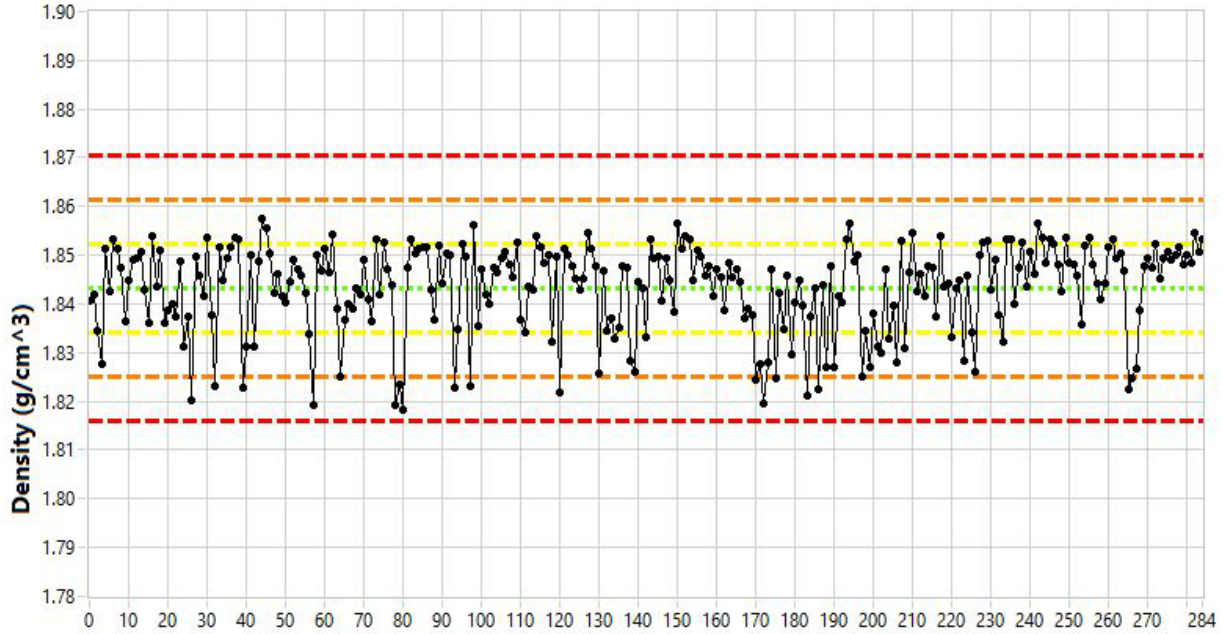


Figure 20. Density (g/cm^3), mean = 1.8432, standard deviation = 0.0091, COV = 0.49%.

2.2.3 Fundamental Frequency

The precise parallelepiped geometry of flexural specimens renders them particularly valuable for accurate measurements of fundamental frequency to collect elastic constants for both dynamic Young's modulus and shear modulus (ASTM C747-93 [8]). Values for fundamental-frequency-based moduli, both in flexural and torsional modes (shown in

Figure 21 and Figure 22, are calculated from the equations provided in ASTM C1259-08. [9]

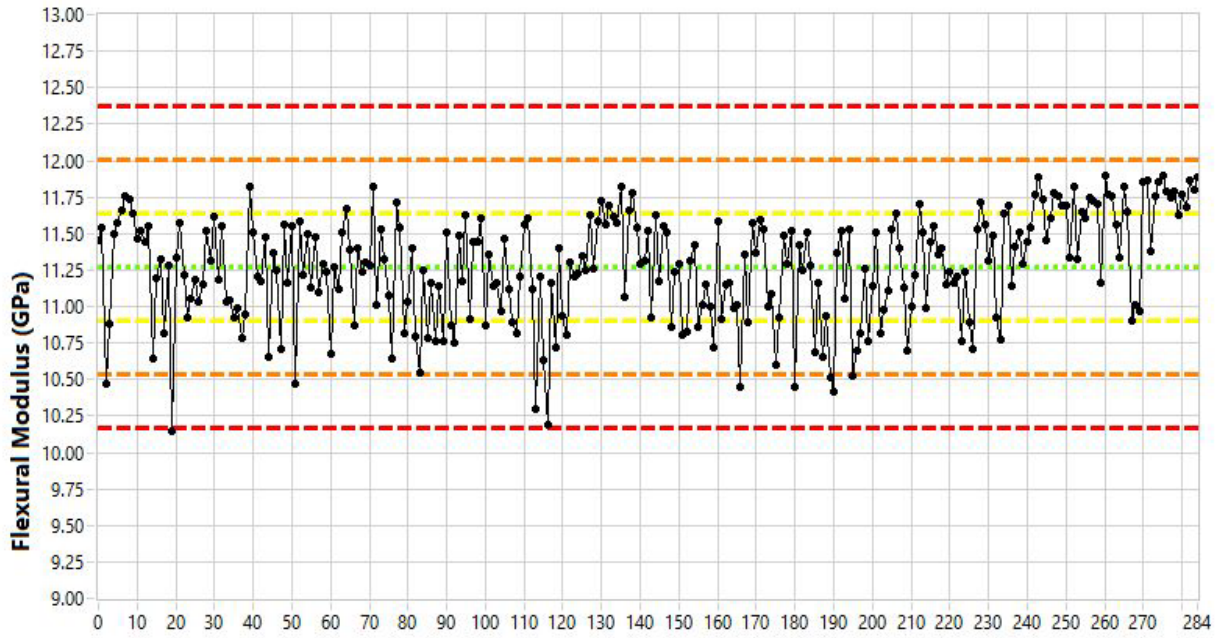


Figure 21. Flexural vibration mode modulus (GPa), mean = 11.27 standard deviation = 0.37, COV = 3.3%.

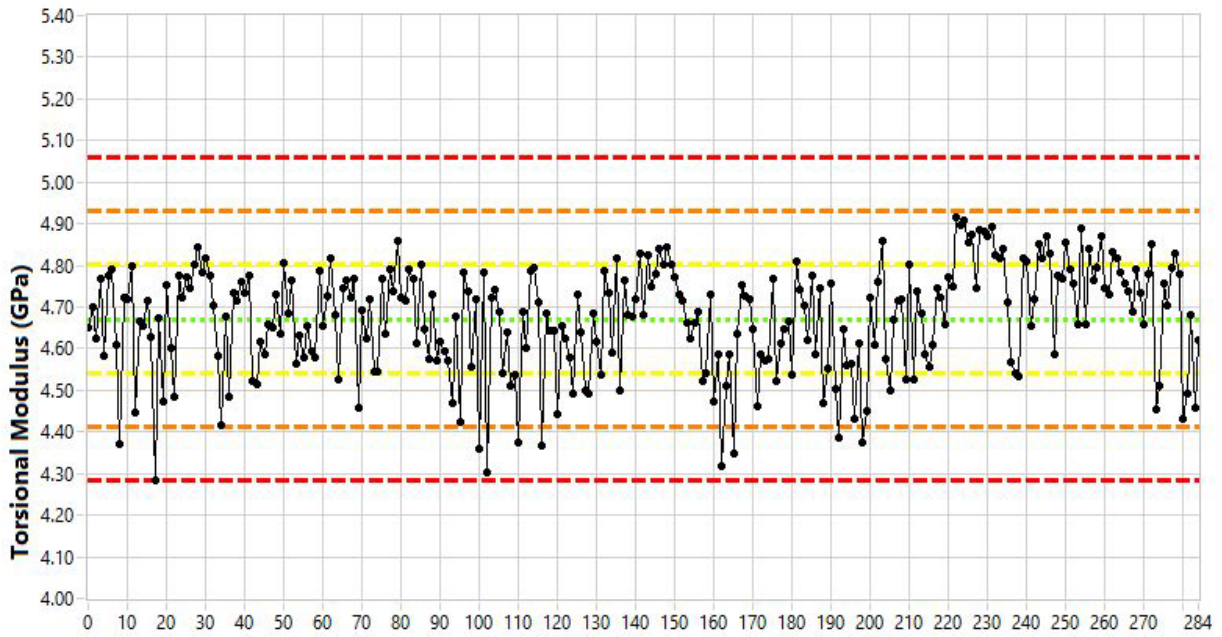


Figure 22. Torsional vibration mode modulus (GPa), mean = 4.67, standard deviation = 0.13, COV = 2.8%.

2.3 Tensile Specimen Database (NBG-17 V104)

2.3.1 Tensile Testing

Tensile testing was performed per ASTM C749-08.[14] Data verification follows the principles discussed in previous sections. As with other specimen types, data verification lies not only in documented adherence to applicable test plans and standards, but in noted correlations between recorded test properties and analyses for outlying values. Additional verification of test conditions can be carried out through an analysis of ancillary physical characteristics.

A total of 279 specimens were tested in tension. Figure 23 shows the gauge diameters of the tensile specimens. Four of the specimens' gauge diameters were low and outside of the control limits. However, their corresponding strengths were within the control limits, so these data were included in the database.

Figure 24 and Figure 25 show the relationship between tensile load and recorded tensile strength for all the specimens tested in uniaxial tension from the NBG-17 Billet V104. Figure 26 shows the recorded tensile stress for the tested specimens. Further comparisons and verification can be made with extensometer-based measured strain (shown in Figure 27), which will reflect an additional correlation with stress values through material elastic constants. Comparing the extreme values again shows this relationship to be valid. Two of the strain values in Figure 27 were low, and outside of the control limits, but their corresponding stress values were acceptable, so these data were kept and included in the database. Other plots of supporting data from the flexural specimens are shown in Appendix C, "Additional Tensile Specimen Database Plots (NBG-17 V104)."



Figure 23. Minimum gauge diameter (mm), mean = 8.753, standard deviation = 0.01, COV = 0.11%.

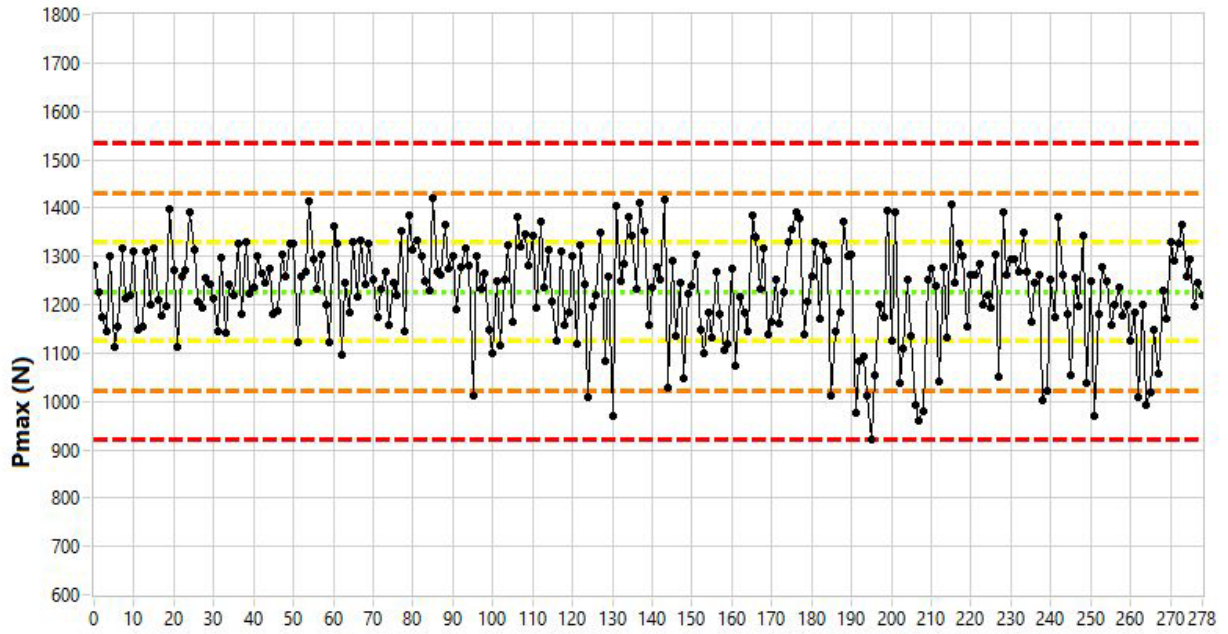


Figure 24. Max load (N), mean = 1226.5, standard deviation = 102.2, COV = 8.3%.

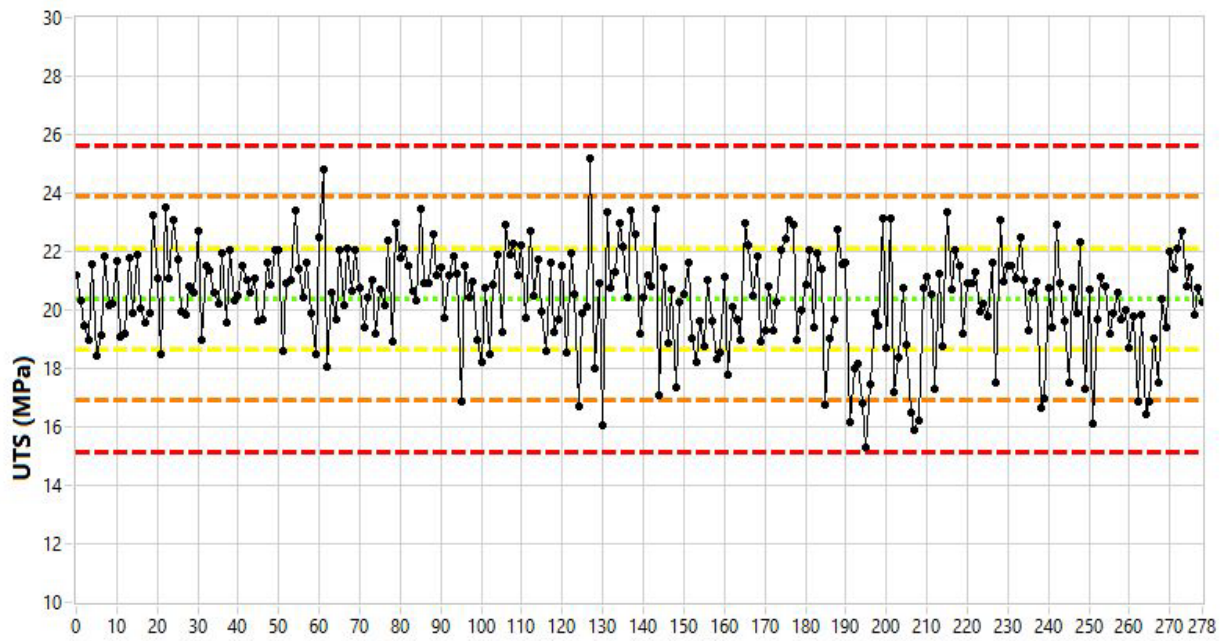


Figure 25. Ultimate tensile strength (MPa), mean = 20.39, standard deviation = 1.75, COV = 8.6%.

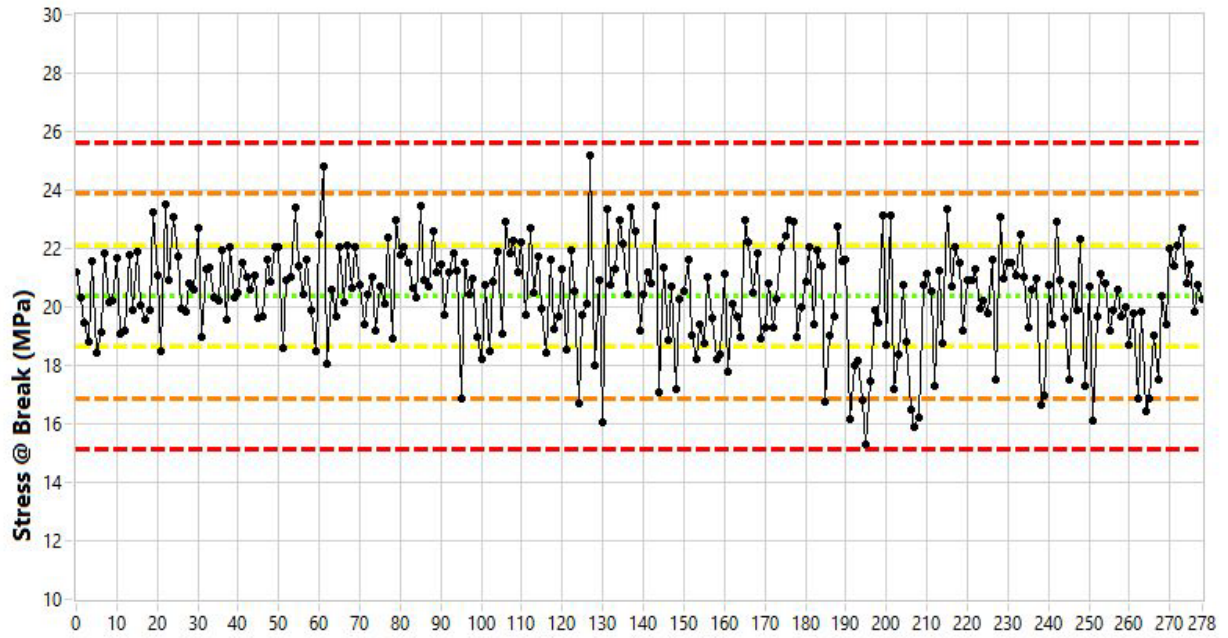


Figure 26. Stress at break (MPa), mean = 20.38, standard deviation = 1.75, COV = 8.6%.

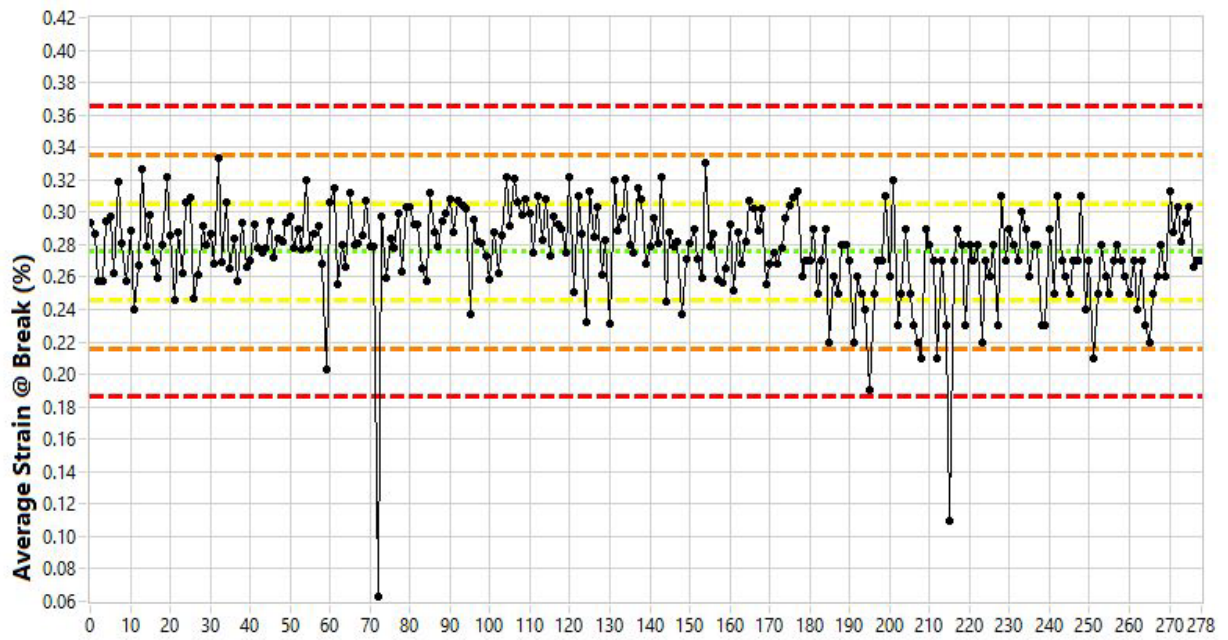


Figure 27. Average strain at break (%), mean = 0.276, standard deviation = 0.03, COV = 10.9%.

2.4 Re-Machined Specimen Properties

Two of the key components to direct comparisons between baseline and AGC data are (1) the analyses of specimens with similar geometries and (2) employment of similar test techniques for comprehensive validation. The geometry of the tensile specimens provides the opportunity to “re-machine” the unstressed sections of the specimen ends (shown in Figure 28) to the same dimensions as AGC piggyback specimens. A random cross-section of tensile specimens was re-machined to repeat tests on AGC-sized specimens (i.e., diffusivity and split-disc testing). Using actual test specimens for re-machining enables continued employment of the specimen identification and tracking-code system, because specimens are machined from tracked locations and can reuse the identification code.

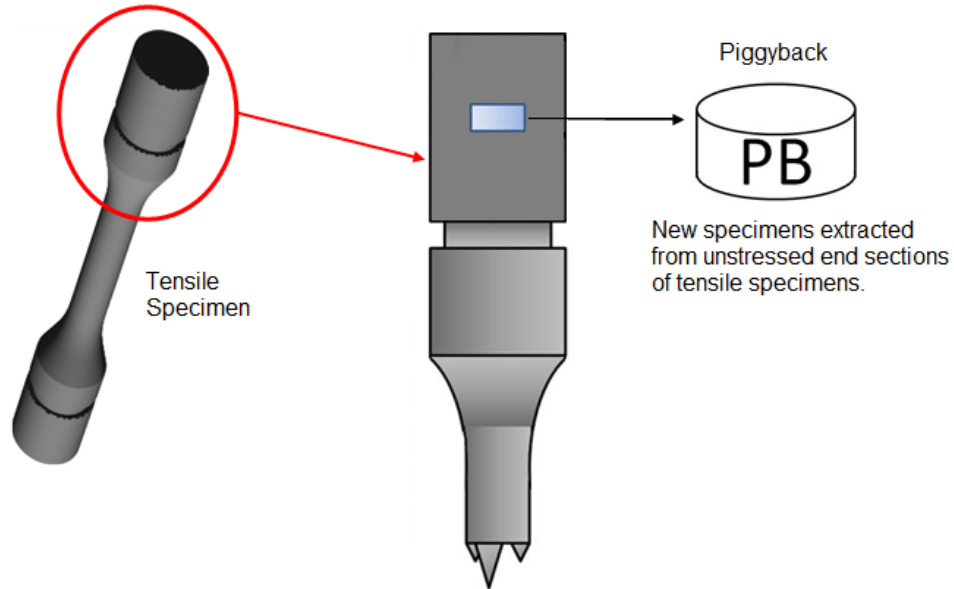


Figure 28. Unstressed specimen remnants from tensile specimens are re-machined into AGC-sized piggyback specimens.

2.4.1 Re-machined Split Disc Testing

Disc-splitting tensile-strength testing was performed in accordance with ASTM Standard D8289-19 [16]. This allows for a direct comparison of tensile data to data that were acquired through strict application of ASTM C749-08. Figure 29 and Figure 30 show strength and load data from the split-disc testing. The mean strength calculated from the split-disc testing was roughly 4.9% lower than the traditional tensile testing (Figure 25) but the standard deviation was about 20% lower.

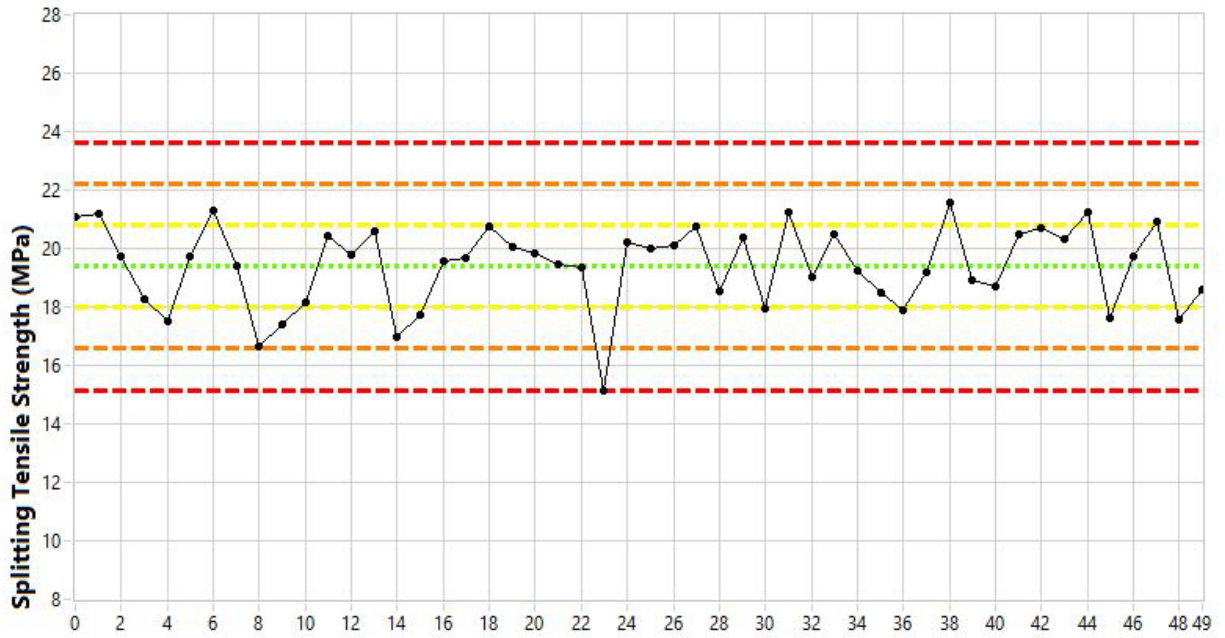


Figure 29. Disc splitting tensile strength (MPa), mean = 19.4, standard deviation = 1.4, COV = 7.2%.

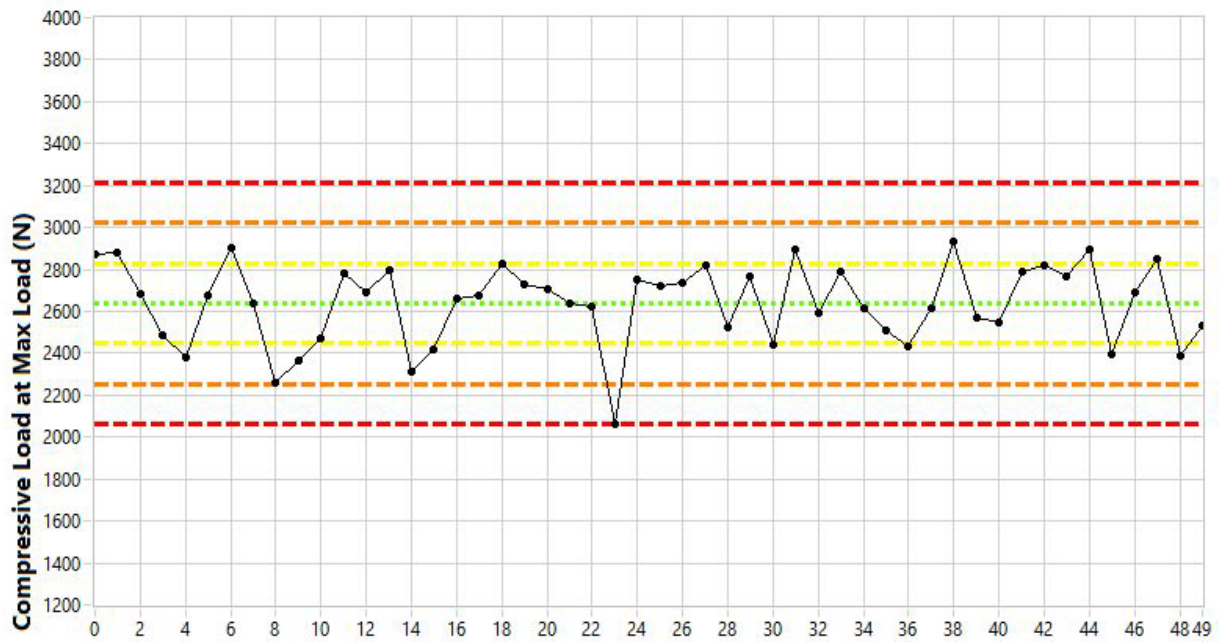


Figure 30. Disc splitting compressive load at max load (N), mean = 2638.3, standard deviation = 191.8, COV = 7.3%.

2.4.2 Re-machined Specimen Diffusivity

Thermal diffusivity values are collected from the re-machined tensile specimens per ASTM E1461-07. [15] Diffusion of heat through the specimen following application of thermal energy via a laser source demonstrates heat-transfer characteristics and can be used to calculate thermal conductivity for design purposes. The resulting group of diffusivity values, revealing a tight grouping of thermal-transfer characteristics, is shown in Figure 31.

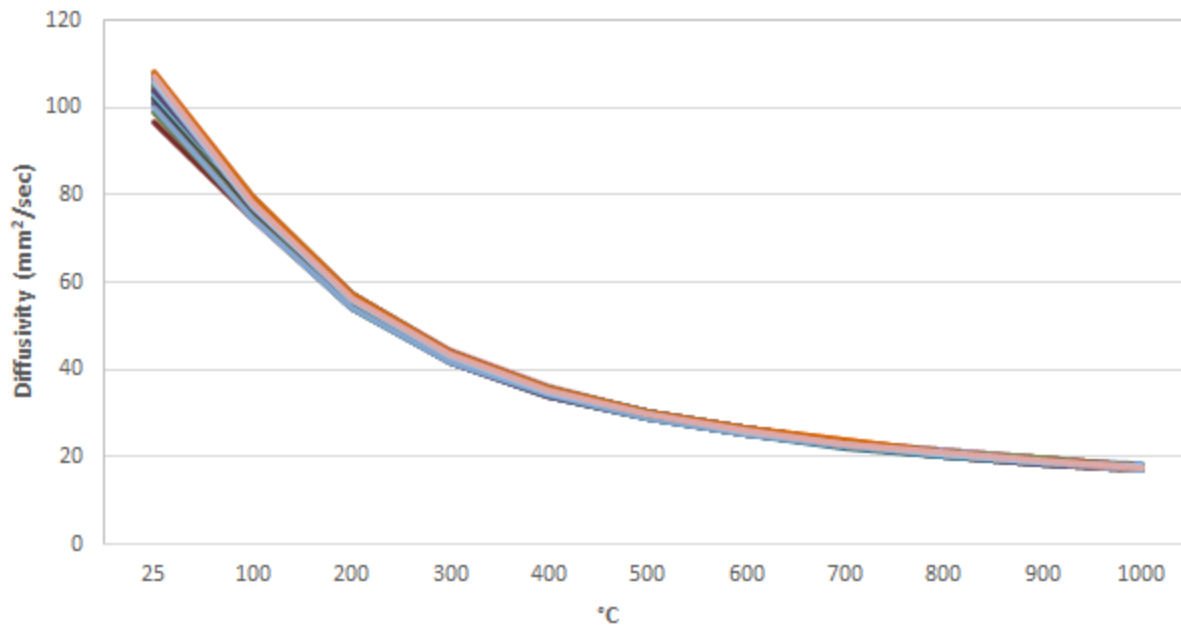


Figure 31. Re-machined specimen diffusivity (mm²/sec).

3. SUMMARY

Comprehensive data sets for NBG-17 Billet V104 have been compiled into summary files of property scalar values. The data spreadsheet files are divided by mechanical test-specimen type into three main sets: (1) compressive, (2) flexural, and (3) tensile. The multitude of tests and evaluations performed on each specimen type are individually tabbed in the main data-set files.

In addition to a full visual review of the data files to determine whether obvious errors were made with the data collected (e.g., missing information or otherwise blank cells), graphical representations were made of individual evaluations to provide a means to spot anomalies. A review of the data indicates that the files, as submitted, are fully representative of the measured properties of the graphite billets being tested, as outlined in the applicable test procedures and program plans.

4. APPENDICES

Appendix A, Additional Compression Specimen Database Plots (NBG-17 V104)

Appendix B, Additional Flexural Specimen Database Plots (NBG-17 V104)

Appendix C, Additional Tensile Specimen Database Plots (NBG-17 V104)

5. REFERENCES

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14. ASTM C749-08. 2008. "Standard Test Method for Tensile Stress-Strain of Carbon and Graphite." ASTM International.
15. ASTM E1461-07. 2007. "Standard Test Method for Thermal Diffusivity by the Flash Method." ASTM International.
16. ASTM Standard D8289-20. 2020. "Standard Test Method for Tensile Strength Estimate by Disc Compression of Manufactured Graphite." ASTM International.

Appendix A

Additional Compression Specimen Database Plots (NBG-17 V104)

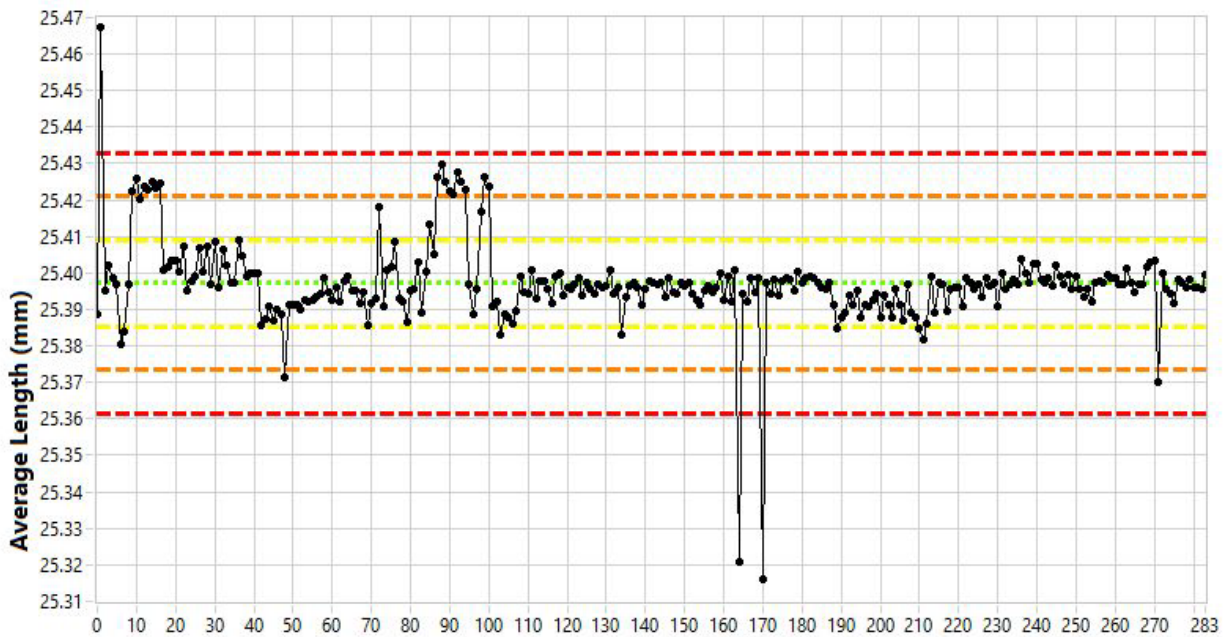


Figure A-1. Average length (mm), mean = 25.3972, standard deviation = 0.0119.

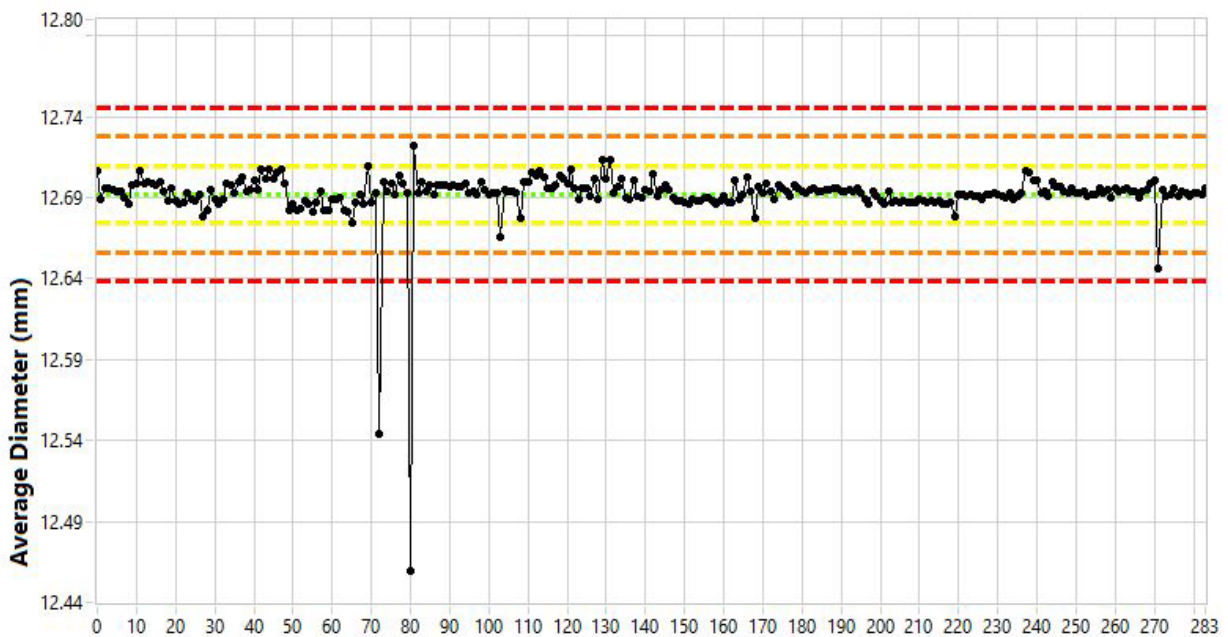


Figure A-2. Average diameter (mm), mean = 12.6920, standard deviation = 0.0179.

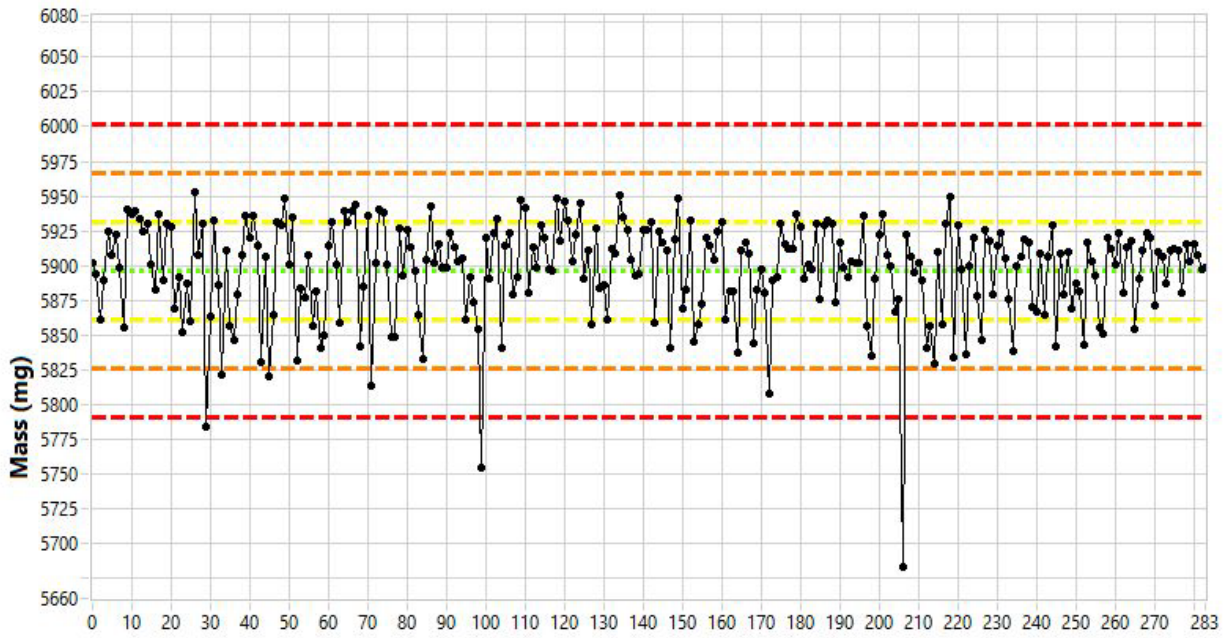


Figure A-3. Mass (mg), mean = 5896.6, standard deviation = 35.2.

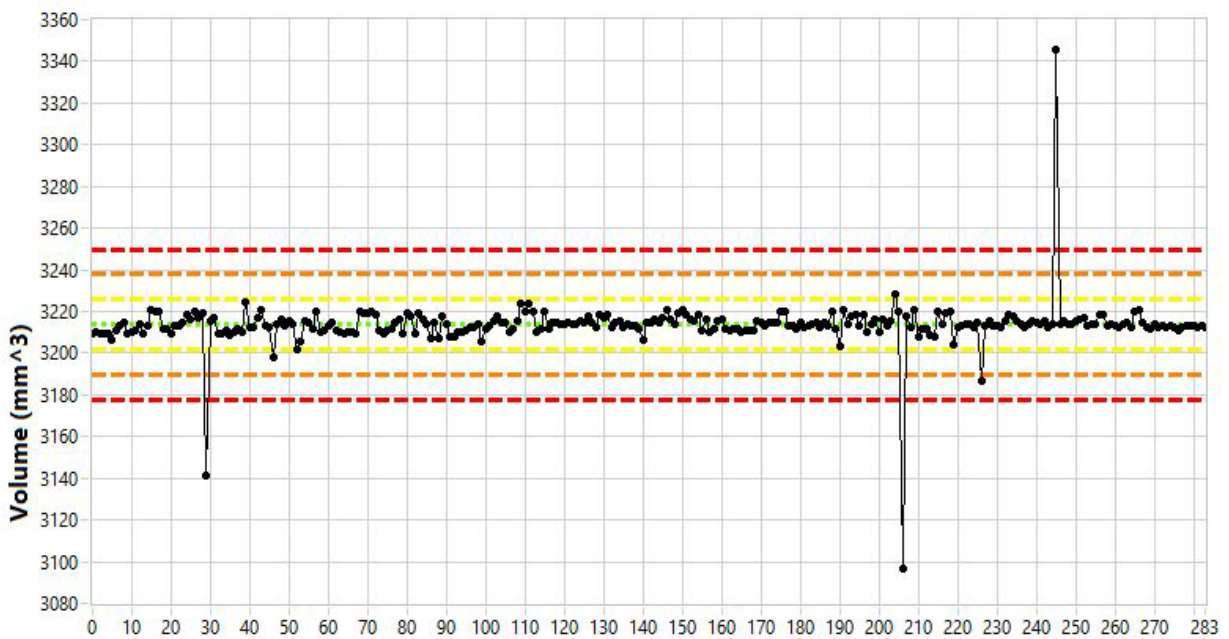


Figure A-4. Volume (mm³), mean = 3213.7, standard deviation = 12.07.

Appendix B

Additional Flexural Specimen Database Plots (NBG-17 V104)

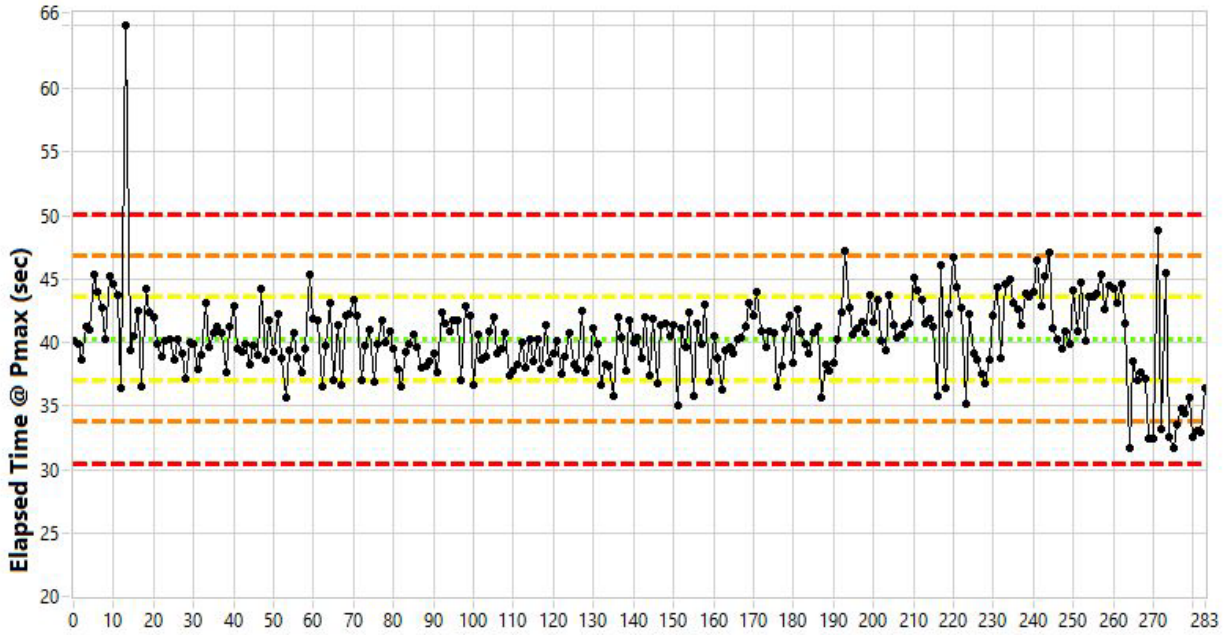


Figure B-1. Elapsed time at max load (sec), mean = 40.3, standard deviation = 3.27.

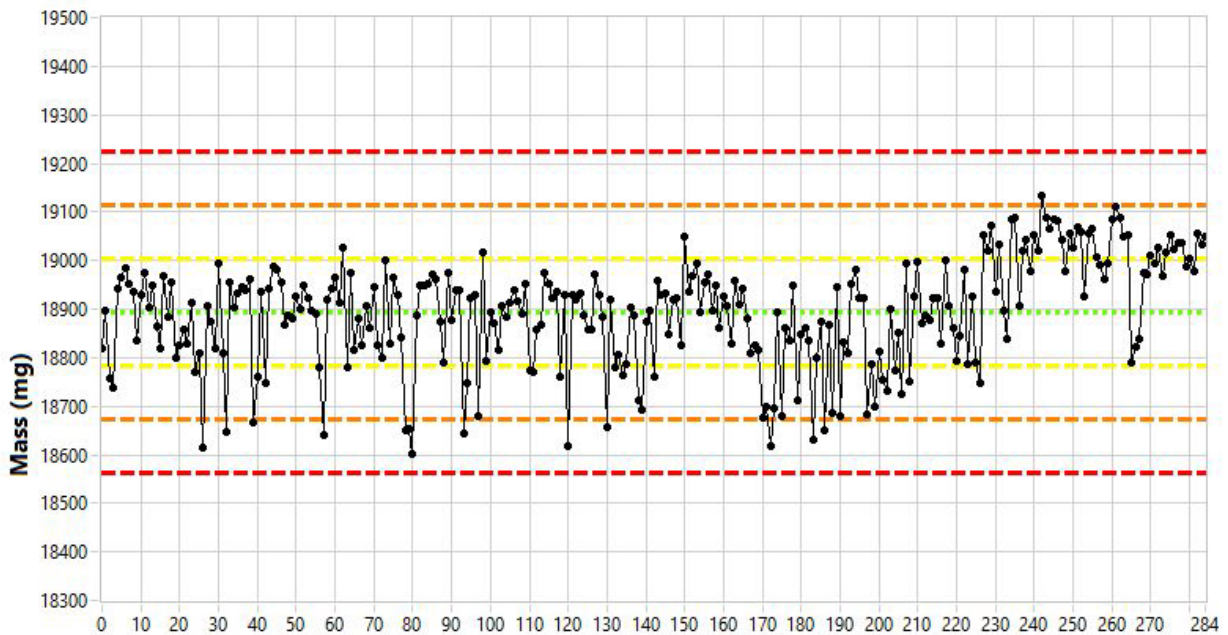


Figure B-2. Mass (mg), mean = 18893.8, standard deviation = 110.5.

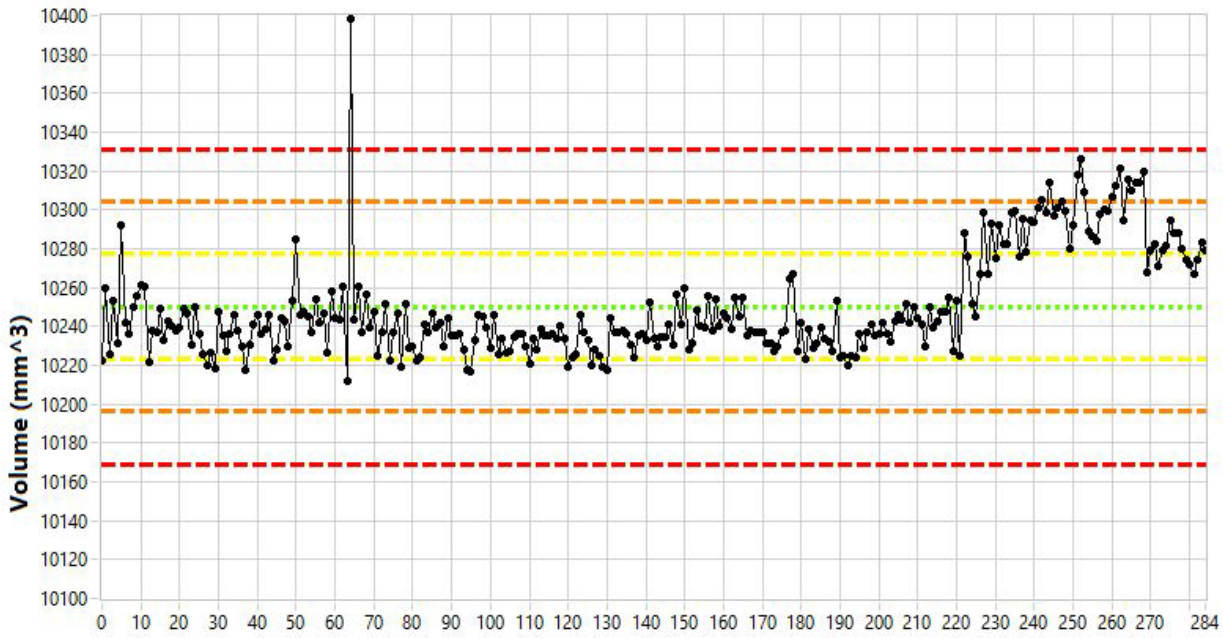


Figure B-3. Volume (mm^3), mean = 10250.2, standard deviation = 27.

Appendix C

Additional Tensile Specimen Database Plots (NBG-17 V104)

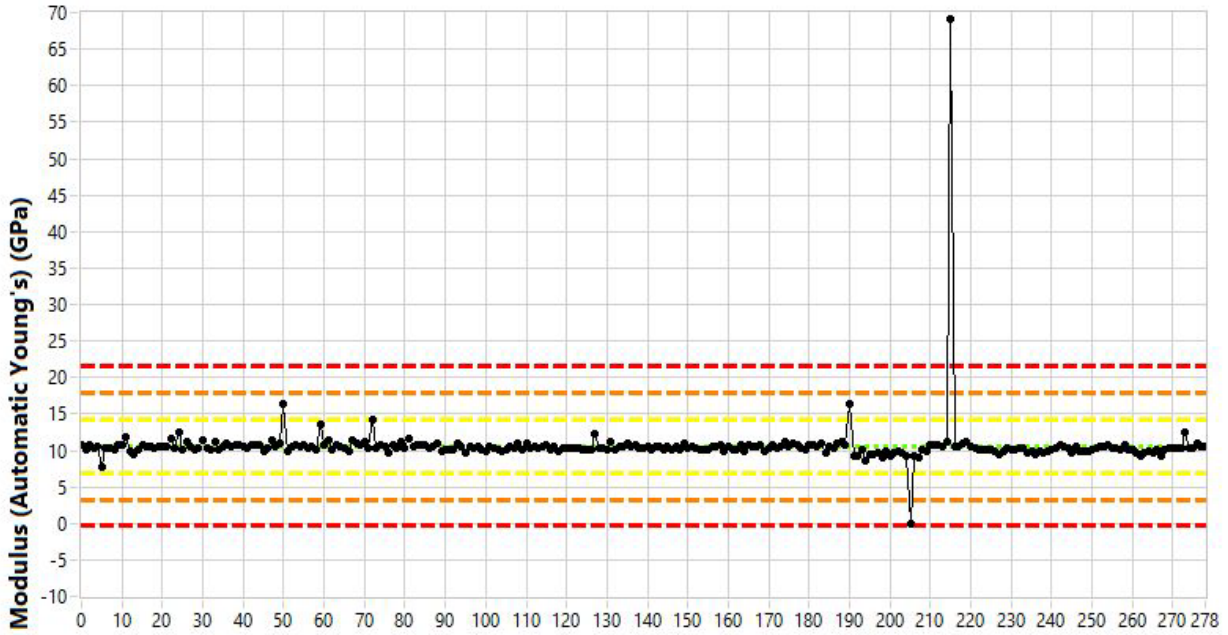


Figure C-1. Modulus (automatic Young's) (GPa), mean = 10.6, standard deviation = 3.7.

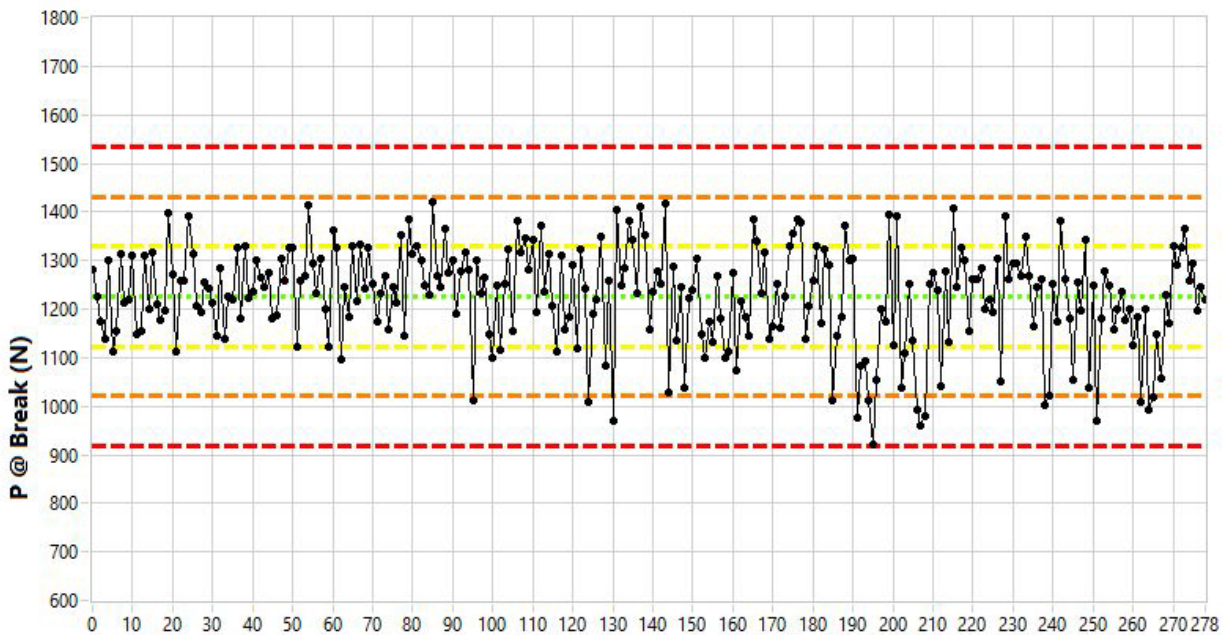


Figure C-2. Load at break (N), mean = 1225.9, standard deviation = 102.3.