INL/MIS-17-42396-Revision-0

Baseline Characterization Database Verification Report IG-110 Billet 08-9-052-7

David Rohrbaugh

July 2017

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Prepared for the
U.S. Department of Energy

Under DOE Idaho Operations Office
Contract DE-AC07-05ID14517

Title: Baseline Characterization Database Verification Report – IG-110 Billet 08-09-0527

ECAR No.: 3621 Rev. No.: 0 Project No.: 32138 Date: 07/11/2017

Engineering Calculations and Analysis

ECAR Title: Baseline Characterization Database Verification Report – IG-110 Billet 08-9-052-7

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1. Confirmation of completeness, mathematical accuracy, and correctness of data and appropriateness of assumptions.
2. Concurrence of method or approach. See definition, LWP-10106.
3. Concurrence of procedure compliance. Concurrence with method/approach and conclusion.
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ECAR No.: 3621 Rev. No.: 0 Project No.: 32138 Date: 07/11/2017

REVISION LOG

Rev.	Date	Affected Pages	Revision Description
0	07/11/2017	All	Newly issued document.

Title: Baseline Characterization Database Verification Report – IG-110 Billet 08-9-052-7

ECAR No.: 3621 Rev. No.: 0 Project No.: 32138 Date: 07/11/2017

1. Quality Level (QL) No.	NA	**Professional Engineer's Stamp** **NA**
2. QL Determination No.	NA	
3. Engineering Job (EJ) No.	NA	
4. SSC ID	NA	
5. Building	IRC	
6. Site Area	REC	

7. Objective/Purpose:

The purpose of this Engineering Calculations and Analysis Report is to present the data being collected in the Baseline Graphite Characterization program, which is directly tasked with supporting the Idaho National Laboratory's (INL's) research and development efforts on the Advanced Reactor Technologies (ART) program. This program is populating a comprehensive database that will reflect the baseline properties of nuclear-grade graphite with regard to 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.

The transfer of this 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 that analysis for IG-110 billet 08-9-052-7 and facilitates the release of the associated data to the NDMAS custodians.

8. If revision, please state the reason and list sections and/or pages being affected:

NA

9. Conclusions/Recommendations:

Based on a review of the data spreadsheets compiled from physical and mechanical property measurements on nuclear-grade graphite billet IG-110 08-9-052-7, no notable errors or omissions were found that will preclude the transfer of these data to the NDMAS site for storage.

In addition to a full visual review of the data files to determine whether or not obvious errors, such as missing information, were made with the data collected, graphical representations were made of individual evaluations to provide a means to spot anomalies. The techniques employed are an adequate means of ensuring that the comprehensive amount of data collected reflect the intended values of interest. 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.

CONTENTS

FIGURES

PROJECT ROLES AND RESPONSIBILITIES

Project Role	Name (Typed)	Organization	Pages covered (if applicable)
Performer	David Rohrbaugh	D520	All
Checker[a]	W. David Swank	B120	All
Independent Reviewer[b]	N/A		
CUI Reviewer[c]	N/A		All
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Responsibilities:

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NOTE: *Delete or mark "N/A" for project roles not engaged. Include ALL personnel and their roles listed above in the eCR system. The list of the roles above is not all inclusive. If needed, the list can be extended or reduced.*

SCOPE AND BRIEF DESCRIPTION

This engineering calculations and analysis report is a validity evaluation of the physical and mechanical property databases collected on a billet of nuclear grade graphite (i.e., IG-110 Billet 08-9-052-7) in support of the ART Baseline Graphite Characterization Program.[1,2] Millions of raw data points that 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 comprised 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.

This report is not a direct analysis of properties and will not provide information on the validity or performance characteristics of the graphite itself. Rather, it is intended as a verification of the completeness of actual data collected in accordance with PLN-3467,[3] "Baseline Graphite Characterization Plan: Electromechanical Testing," and its representation of the measurement and test results with sole regard to the graphite billets under evaluation.

DESIGN OR TECHNICAL PARAMETER INPUT AND SOURCES

Mechanical and physical property testing is carried out in accordance with PLN-3348, "Graphite Mechanical Testing," PLN-3467,[3] "Baseline Graphite Characterization Plan: Electromechanical Testing," and PLN-3267,[4] "AGC-2 Characterization Plan."

ASSUMPTIONS

None

COMPUTER CODE VALIDATION

Data collection and storage is organized as reported in PLN-3467 and Idaho National Laboratory (INL)/EXT-10-19910,[5] "Baseline Graphite Characterization: First Billet". Individual computers being used run Windows XP operating systems and store data on Microsoft Office Excel 2007 spreadsheets.

Control of individual test equipment is carried out by proprietary Netzsch software (IRC C-20) or Instron's Bluehill (Version 2) software (load frames in IRC B-11). Both software suites are commercially available packages. Updates and data transfers/integration are handled outside of INL's network system on a dedicated local area network.

The comprehensive interface between data collection, evaluation, and storage computers is handled through the customized LabVIEW-based Graphite Mechanical Properties Data Acquisition Software (Version 4.0). The Baseline Graphite Characterization Program's version control and operability checks are documented and validated in a registered laboratory notebook, LAB 2143, "Baseline Graphite Characterization." All pertinent lifecycle documentation is recorded in accordance with LWP-20000-01, "Conduct of Research Plan." Validation of commercial packages is handled via integrated system checks specific to each new element or upgrade as appropriate.

DISCUSSION/ANALYSIS

Introduction

The ART Project Graphite Research and Development Program is generating the extensive amount of quantitative data necessary for predicting the behavior and operating performance of the available nuclear graphite grades. To determine the in-service behavior of graphite for the latest proposed designs, two main programs are underway: The Advanced Graphite Creep (AGC) Program. The 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 very high-temperature reactor core.[1] A limited amount of data can be generated on irradiated material because of the limited availability of space within the Advanced Test Reactor and the geometric constraints placed on the AGC specimens that will be inserted. 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 being 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 nuclear graphite that are presently available.

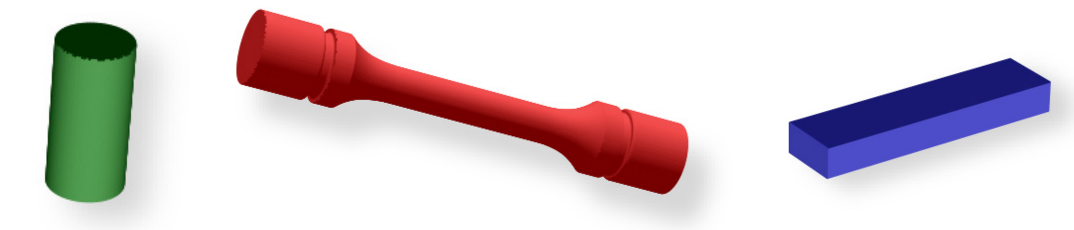
This particular report covers the release of physical and mechanical property data from a partial billet of IG-110 graphite. The graphite billet (IG-110-08-9-052-7) is a block of iso-molded graphite with a fine grain structure. The main baseline mechanical properties database for this billet, plots of which are included throughout this report, are comprised solely of scalar results from each of the different evaluations (mechanical testing and physical properties) in summary form, and consists of tabbed spreadsheets being occupied by over 12,000 cells of individual characteristic or property values and associated tagging information.

This report is intended as a validation review of the billet listed above. It is not an analysis of property characteristics or trends beyond the evaluation necessary to determine whether or not the data that has been collected is reflective of the properties of this particular graphite billet. It is an acceptance of the test methods used, the data calculations and conversions being carried out, and a review of the values from the standpoint of determining whether or not they reflect anomalous behavior that must be further investigated.

Ultimately, this report is the justification for the transfer of this data set into a storage and analysis system that is available for internal and external analysts to be utilized in evaluating the relevant characteristics and performance of nuclear-grade graphite.

Database Analysis

The multitude of data sets being generated for the Baseline Graphite Characterization Program consist of properties collected on standard American Society of Testing and Materials (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 is provided in detail in INL/EXT-10-19910.[5] For ease of reviewing the applicable data in this report, an example of a sectioning diagram for IG-110 graphite, along with the applicable specimen identification codes, is provided in Figure 2. This figure is representative of a single sub-block of graphite from this billet.



Compressive Specimen

Tensile Specimen

Flexural Specimen

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

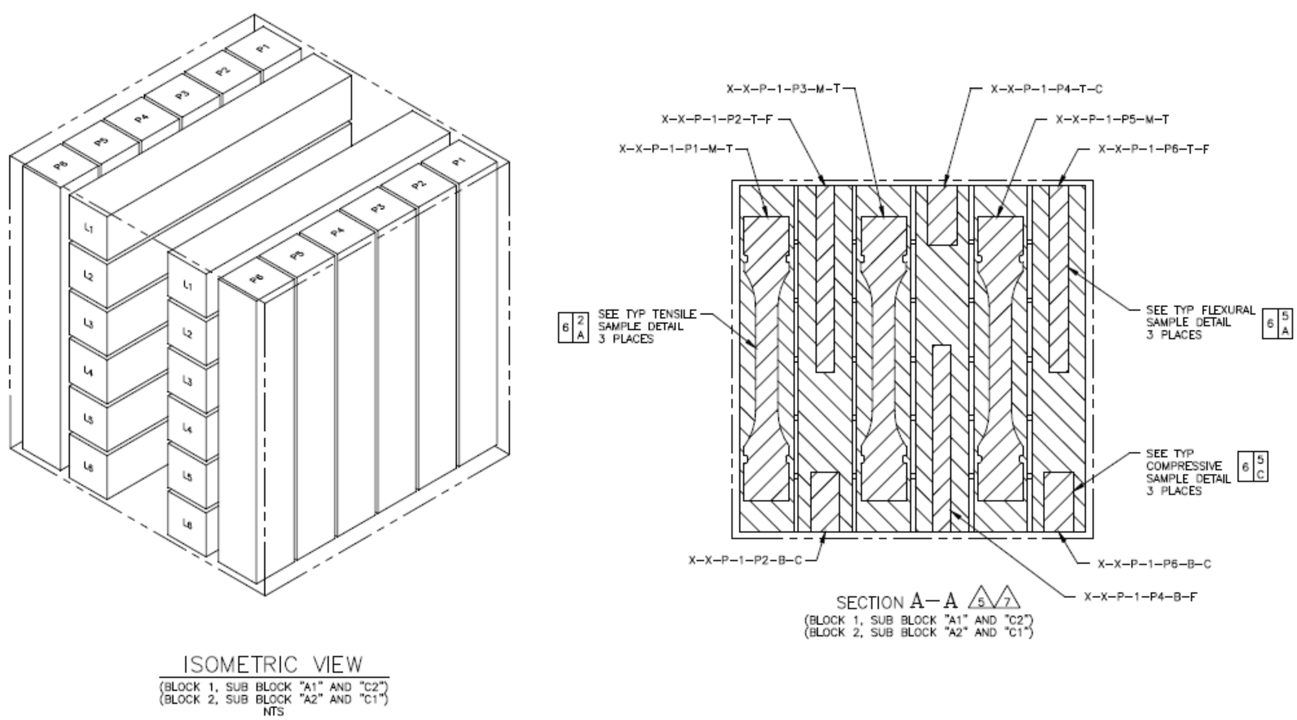


Figure 2. Individual specimen extraction and tracking identification from IG-110-08-9-052-7.

Sections of this report cover each of the individual databases for this billet, are divided by mechanical test specimen type (e.g., compressive, flexural, and tensile), and are organized so they present data in graphical form. The 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 (i.e., 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 the text of this report, but the process control charts with standard deviation values and/or property trend charts for the various characteristics being measured are included (±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).

One of the clear goals of the Baseline Graphite Characterization Program is to identify and quantify inter-billet 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 result 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.

Compression Specimen Spreadsheet Database (IG-110-08-9-052-7C)

Compression Testing

Compression testing was performed per ASTM C695-91[6] and INL PLN-3467. Figure 3 shows the maximum applied load for each of the 48 compression specimens from Billet 08-9-052-7. As was mentioned previously, some variation in properties is expected in graphite, and this variation is reflected in the difference in test frame loading. The compressive strength values (Figure 4) correlate directly with the recorded load values, confirming the stress calculations were performed correctly. 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.

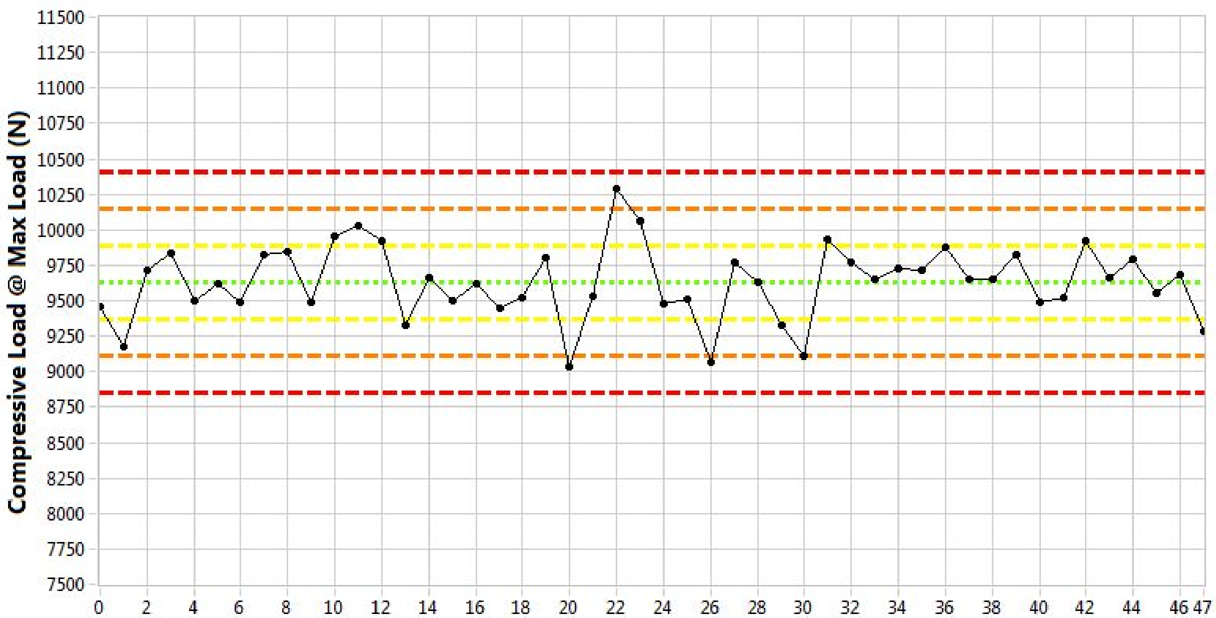


Figure 3. Compressive load at max load (N), mean = 9630.1, standard deviation = 260.76.

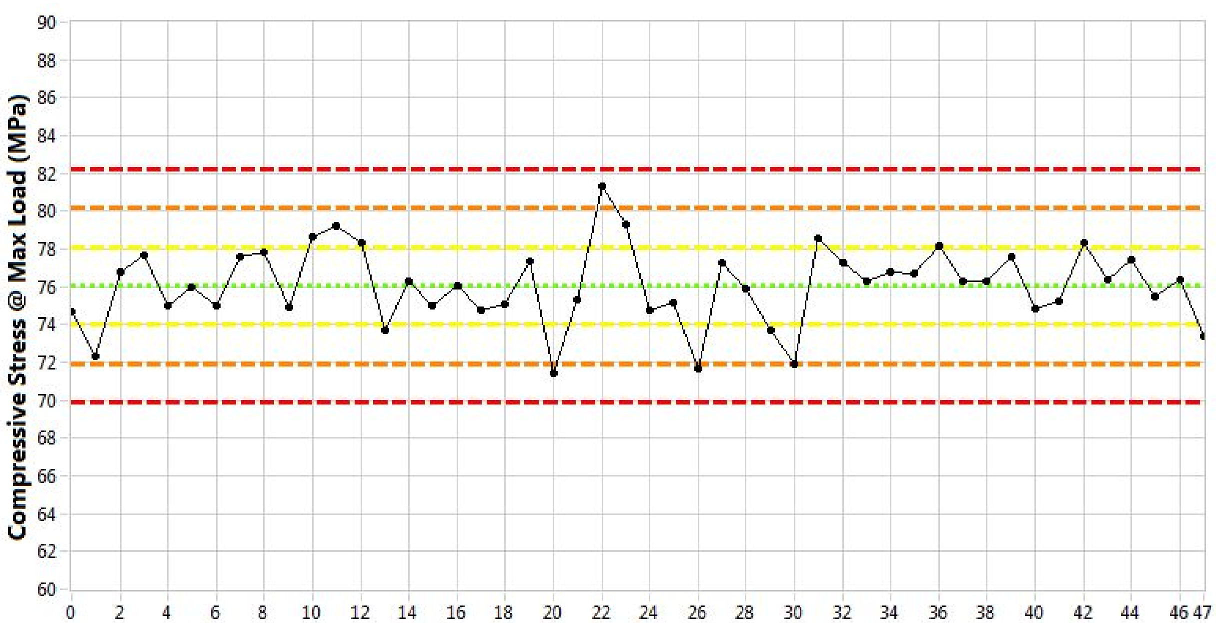


Figure 4. Compressive stress at max load (MPa), mean = 76.1, standard deviation = 2.06.

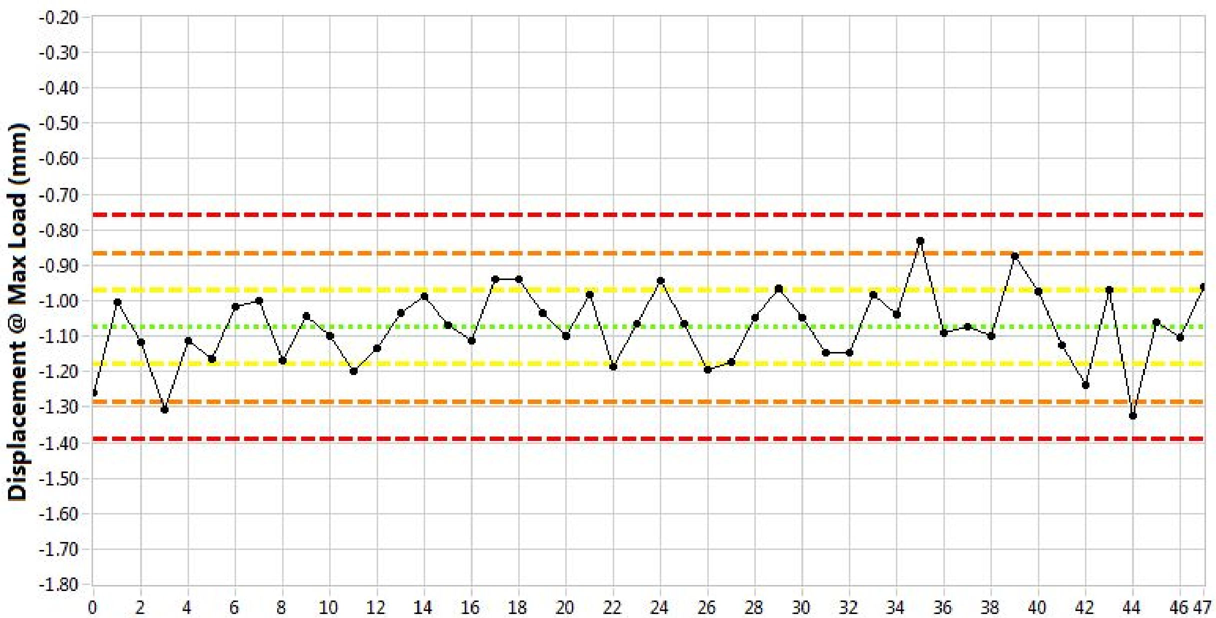


Figure 5. Displacement at max load (mm), mean = -1.0741, standard deviation = 0.1047.

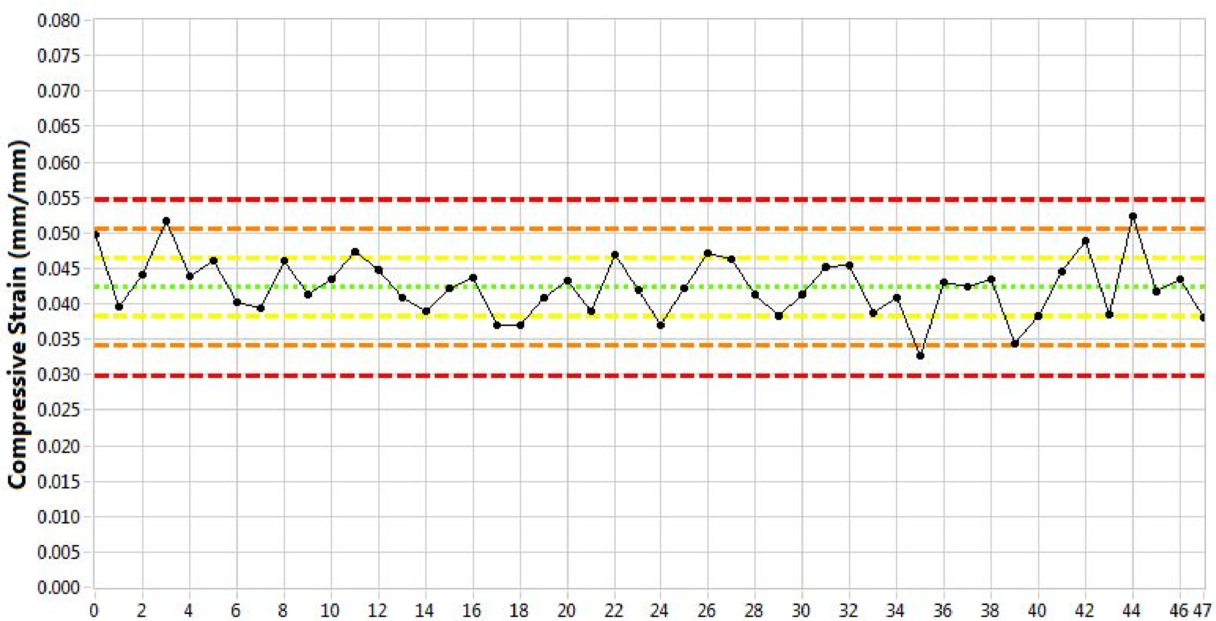
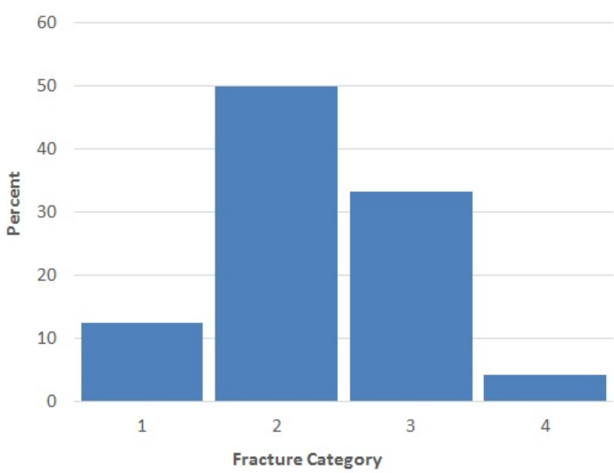


Figure 6. Compressive strain (mm/mm), mean = 0.0424, standard deviation = 0.0041.

Fracture Surface Categorization

The resulting 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. A screen shot of this categorization, along with the distribution of recorded fracture categories for each of the 48 compressive specimens from IG-110 08-9-052-7 (with no anomalous values indicative of an unallowable characterization) is provided in Figure 7.



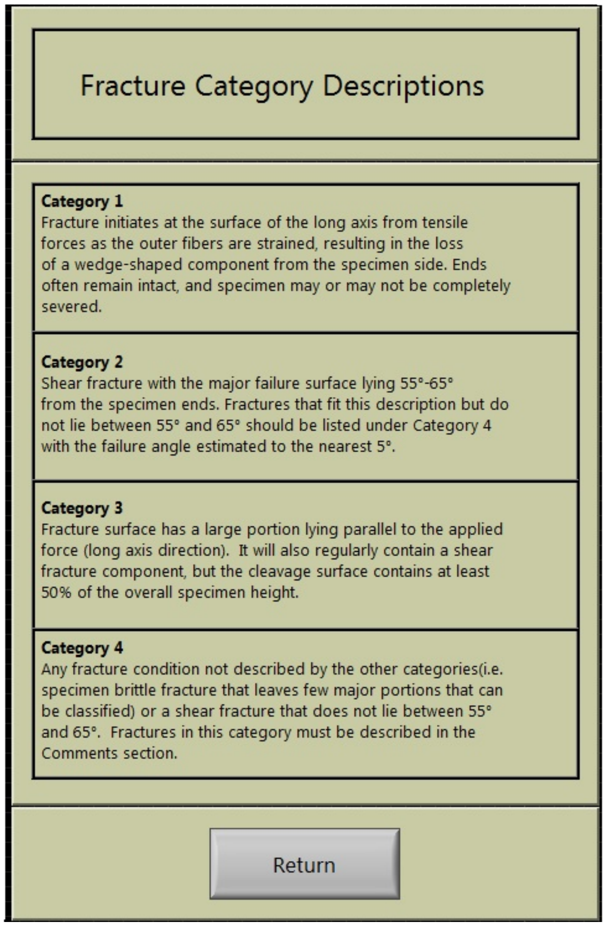


Figure 7. Fracture categorization results and description.

Density Values

The relatively simple geometric shape of the compressive specimens provides an opportunity to collect density data (per ASTM C559-90[7]) for a large portion of the specimens extracted from each billet. While not a true performance property, density measurements are relatively straightforward to collect and are often reflective of bulk mechanical properties. The density values recorded for the compression specimens (Figure 8) show only one anomaly. Further investigation revealed that this specimen's calculated volume, Figure A-4 (average length and average diameter too, Figure A-1 and Figure A-2) was comparable with the other specimens; however, it's mass (Figure A-3) was 2.1% lower than the group mean. This resulted in a low-density value. Despite this low-density value, the specimen's data was within three standard deviations with respect to the compression test measurements, such as compressive stress, load, displacement, etc.

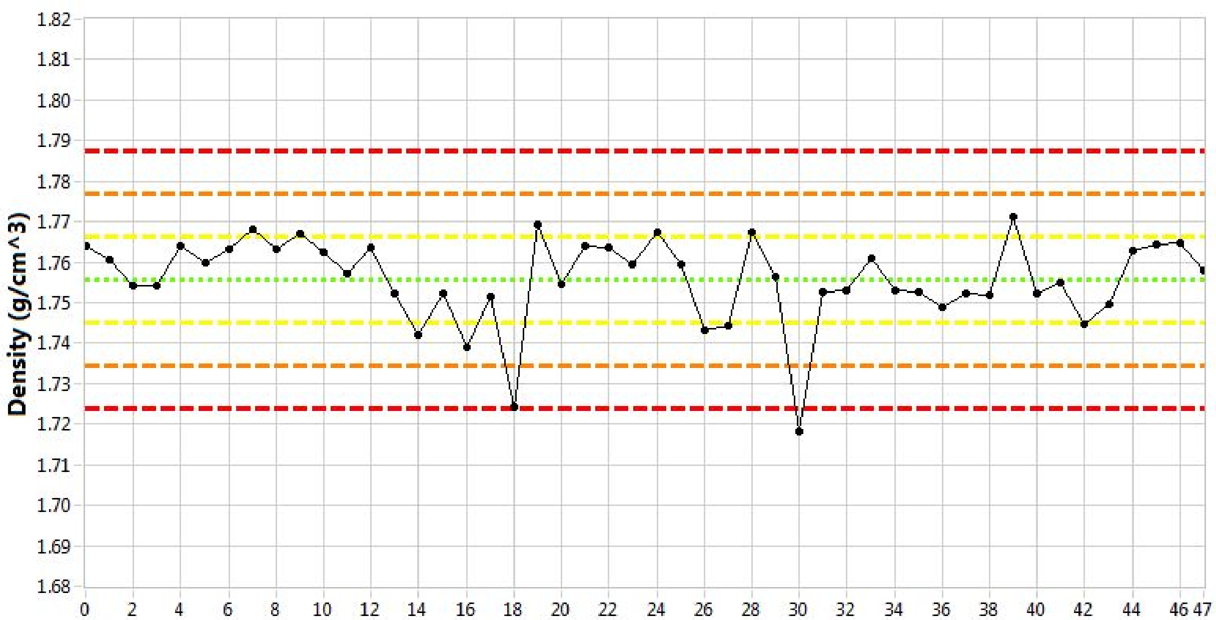


Figure 8. Max load (N), mean = -573.8, standard deviation = 42.73.

Flexural Specimen Database (IG-110-08-9-052-7F)

Flexural Testing

Flexural Testing was performed per ASTM C651-91[8] with clarifications to ambiguities in the standard identified in PLN-3467.[3] Similar to the presentation of the 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. Figure 9 and Figure 10 show the relationship between flexural load and recorded flexural stress for the 46 specimens tested in flexure from IG-110 08-9-052-7. Further comparisons and verification can be made with measured deflection as shown in Figure 11, which will reflect an additional correlation with stress values through material elastic constants.

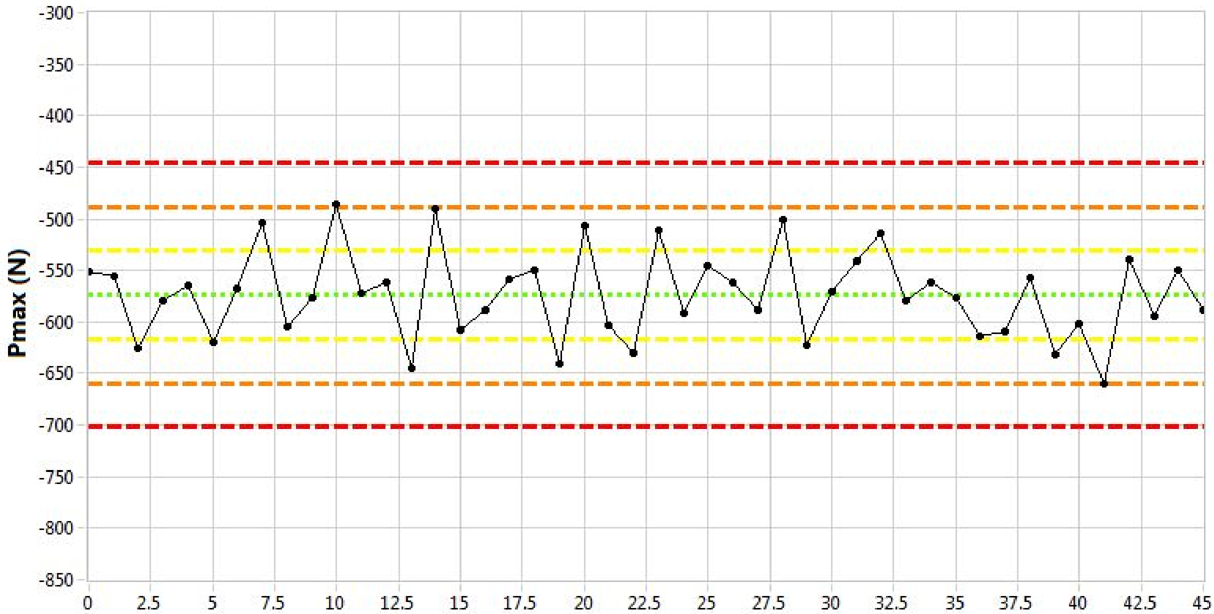


Figure 9. Max load (N), mean = -573.8, standard deviation = 42.73.

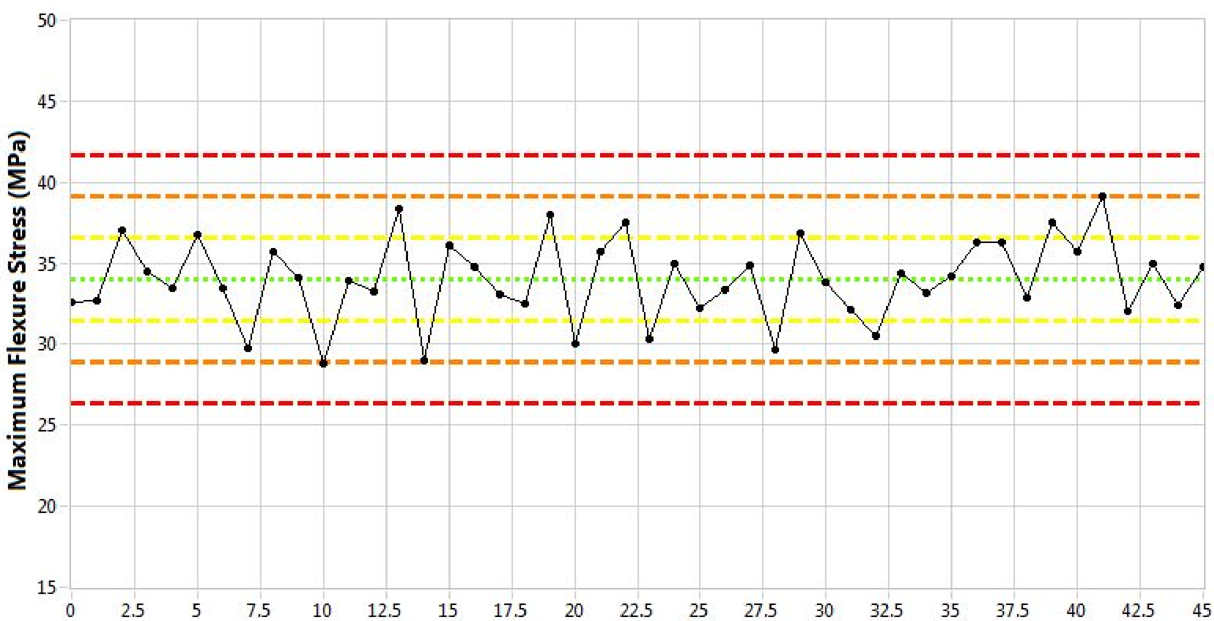


Figure 10. Maximum flexure stress (MPa), mean = 34.0, standard deviation = 2.55.

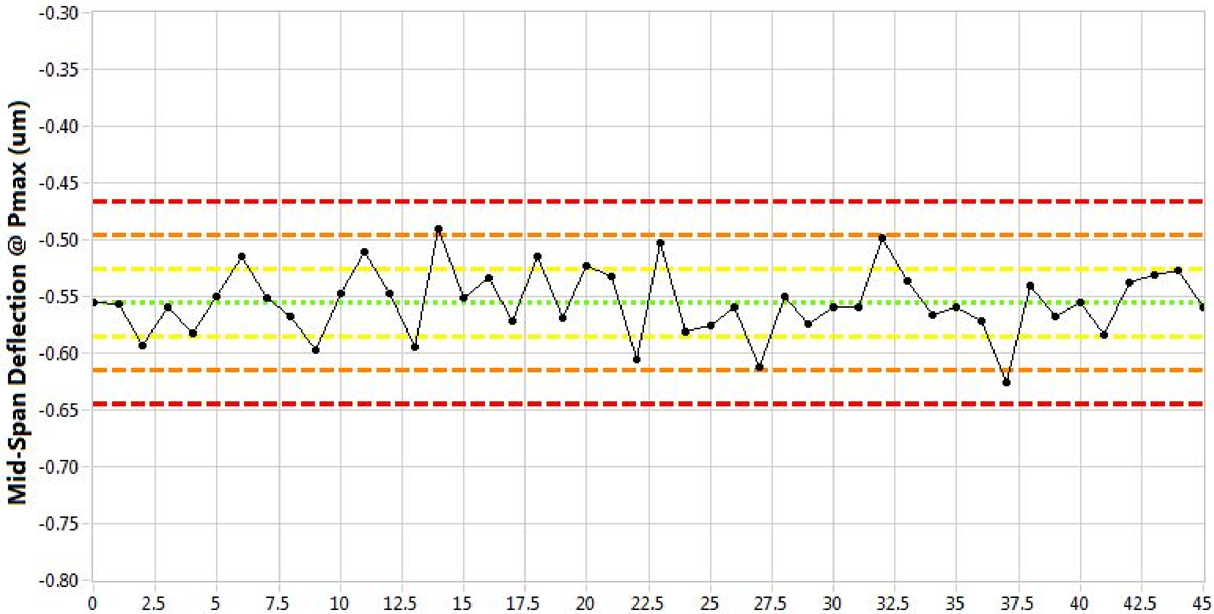


Figure 11. Mid-span deflection at max load (um), mean = -0.5556, standard deviation = 0.0296.

Density Values

Similar to the compression specimens, the flexural specimens' geometry facilitated an opportunity to make density measurements. Figure 12 shows density from the flexural specimens. All flexural specimens' data and associated deviations compare well with the compression specimens' density data.

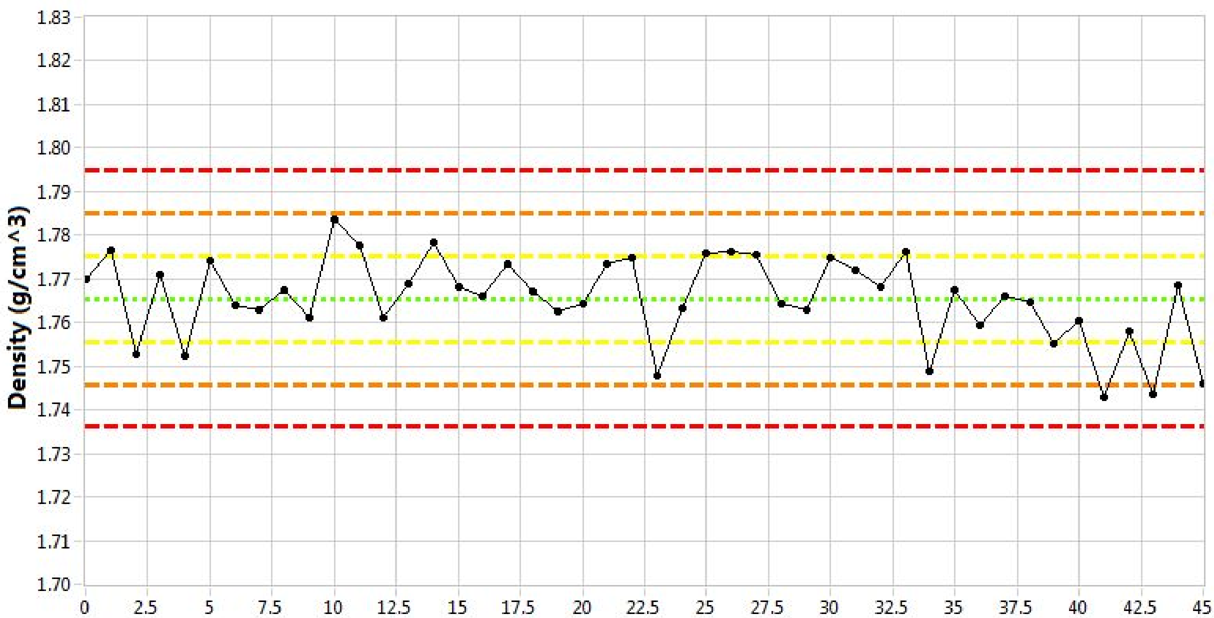


Figure 12. Density (g/cm^3), mean = 1.7654, standard deviation = 0.00980.

Fundamental Frequency

The precise parallelepiped geometry of flexural specimens renders them particularly valuable for accurate measurements of fundamental frequency to collect elastic constants, both for dynamic Young's modulus and shear modulus (ASTM C747-93[9]). Values for fundamental frequency-based moduli, both in flexural and in torsional modes (shown in Figure 13 and Figure 14), are calculated from the equations provided in ASTM C1259-08.[10] These data all fell within +/- 2 standard deviations from their respective means.

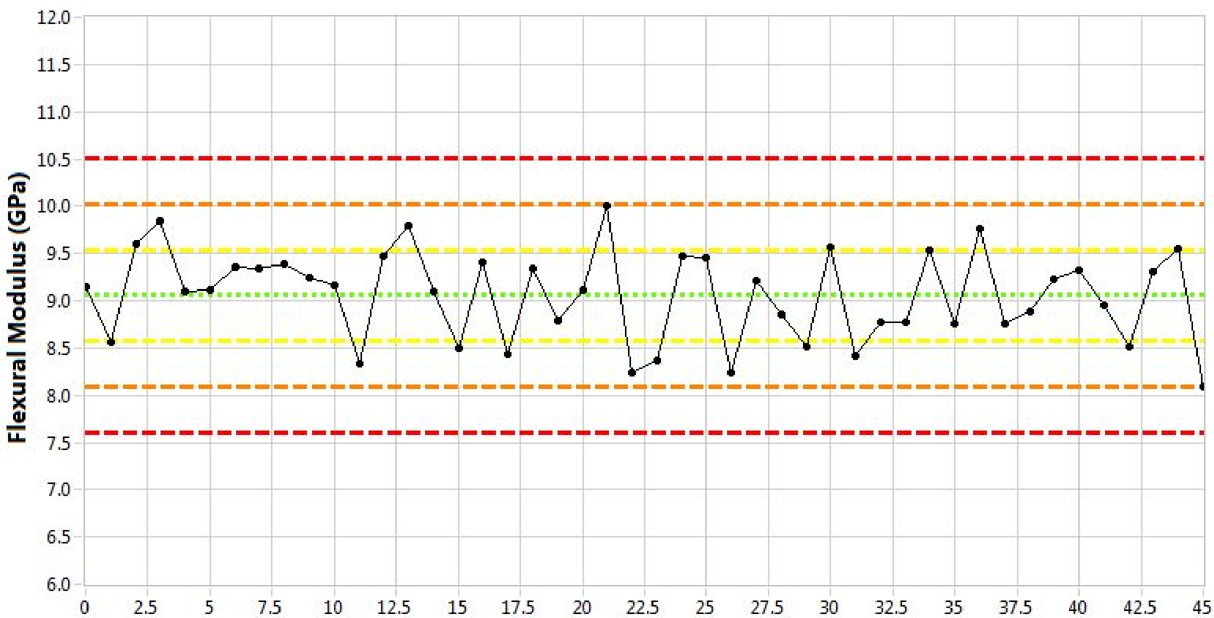


Figure 13. Flexural vibration mode modulus (GPa), mean = 9.1, standard deviation = 0.48.

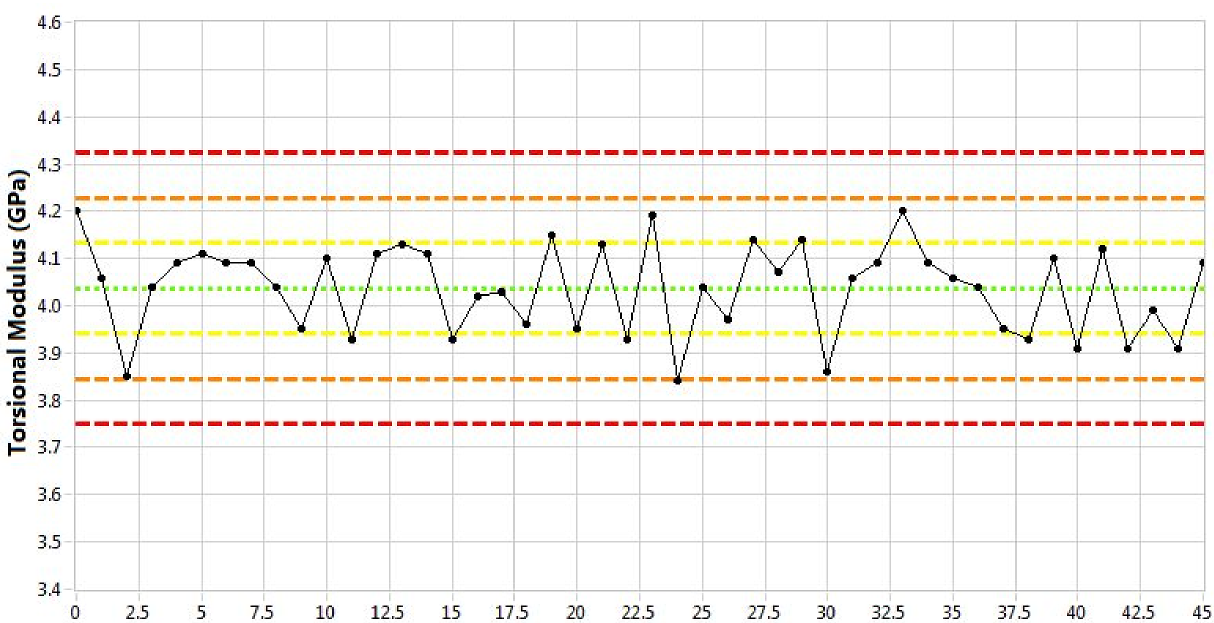


Figure 14. Torsional vibration mode modulus (GPa), mean = 4.0, standard deviation = 0.10.

Tensile Specimen Database (IG-110-08-9-052-7T)

Tensile Testing

Tensile testing was performed per ASTM C749-08.[11] Data verification follows the principles discussed in previous sections. As with the other specimen types, data verification lies not only in the documented adherence to applicable test plans and standards, but in the 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. The custom measurement software used to capture the tensile gauge diameters is programed to flag any measurement that deviates from the ASTM standard. However, as shown in Figure 15, the gauge diameters are consistent and compare well with each other. Figure 16 and Figure 17 show the relationship between tensile load and recorded tensile stress for the 48 specimens tested in uniaxial tension from IG-110 08-9-052-7.

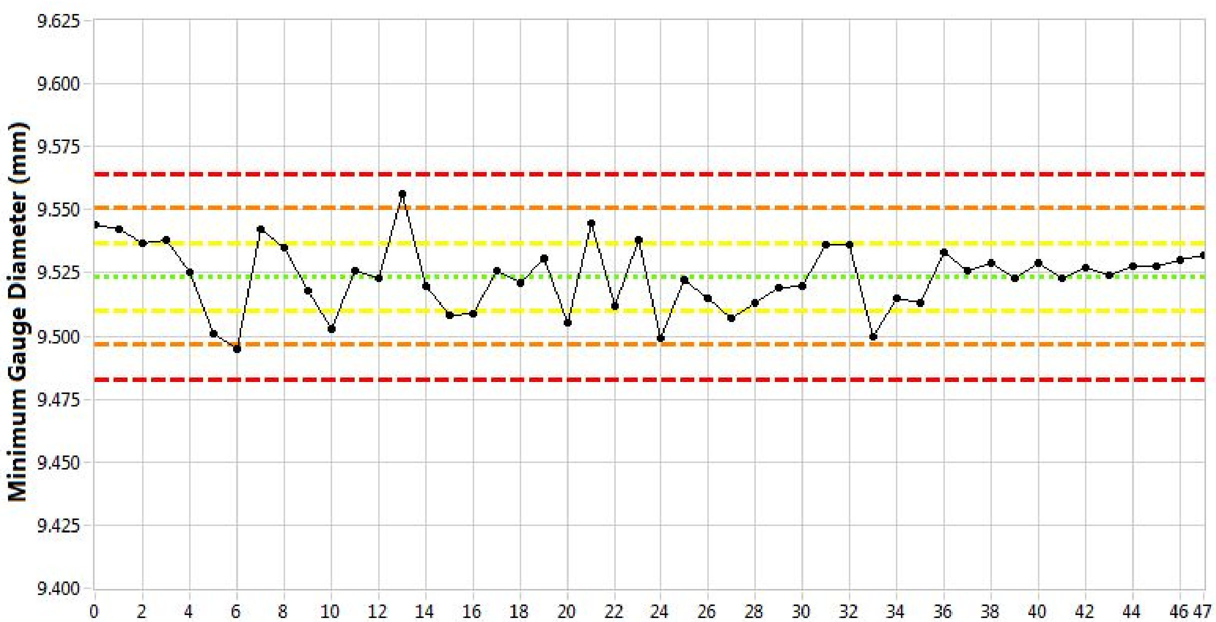


Figure 15. Minimum gauge diameter (mm), mean = 9.523, standard deviation = 0.0135.

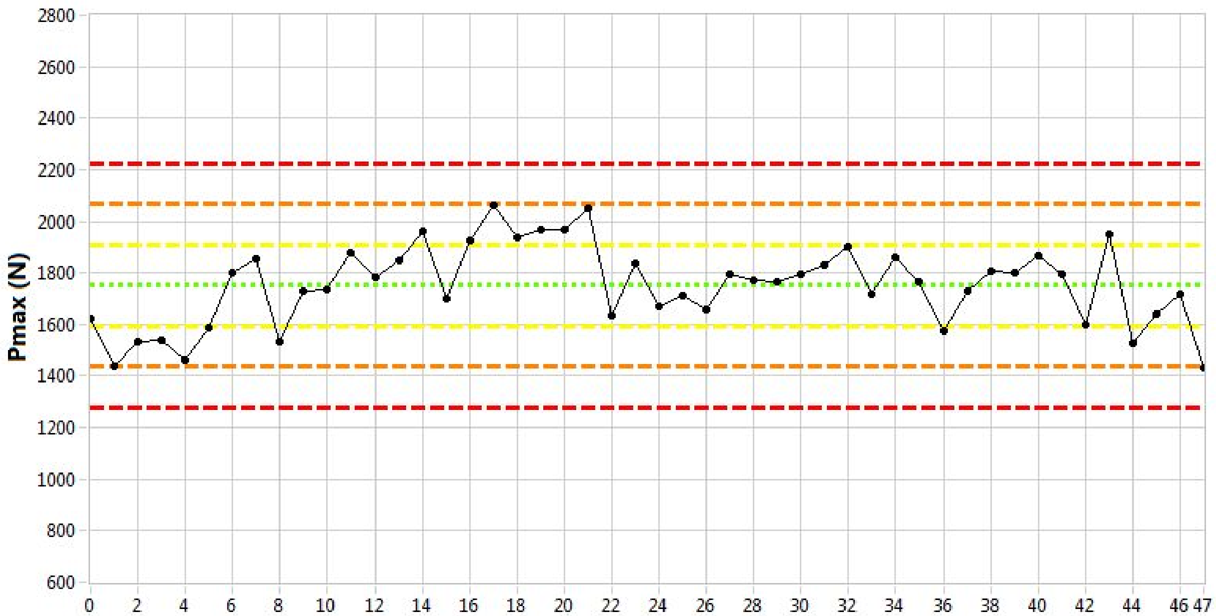


Figure 16. Max load (N), mean = 1751, standard deviation = 157.2.

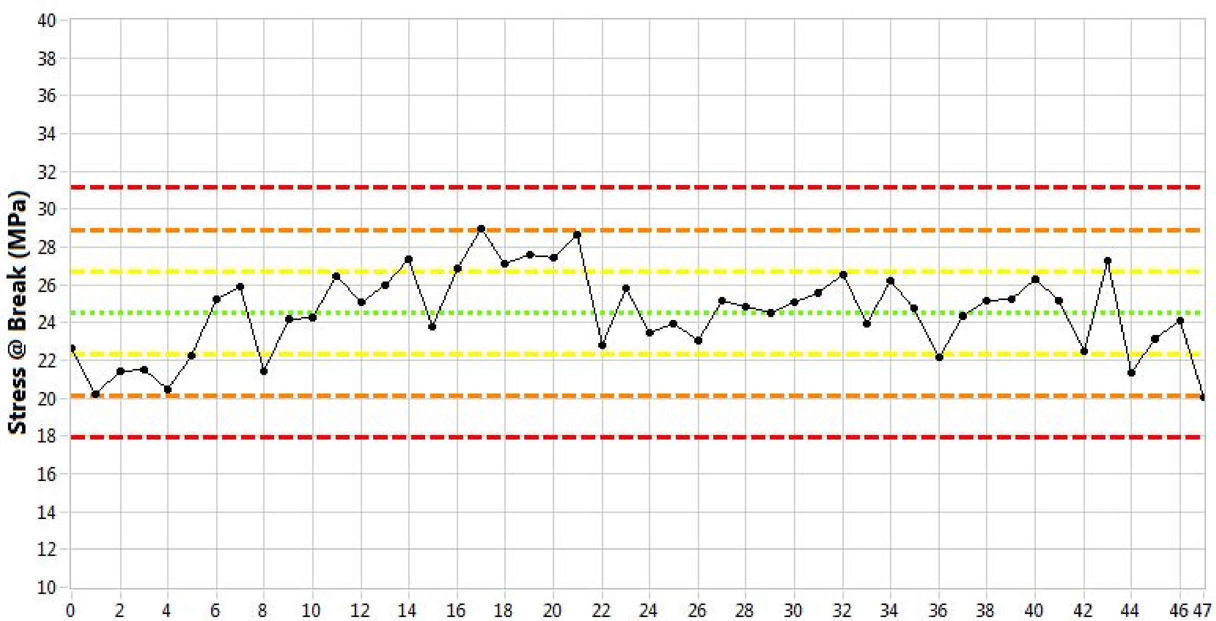


Figure 17. Stress at break (MPa), mean = 24.5, standard deviation = 2.20.

Further comparisons and verification can be made with extensometer-based measured deflection (shown in Figure 18), which will reflect an additional correlation with stress values through material elastic constants. Comparing the extreme values again shows this relationship to be valid.

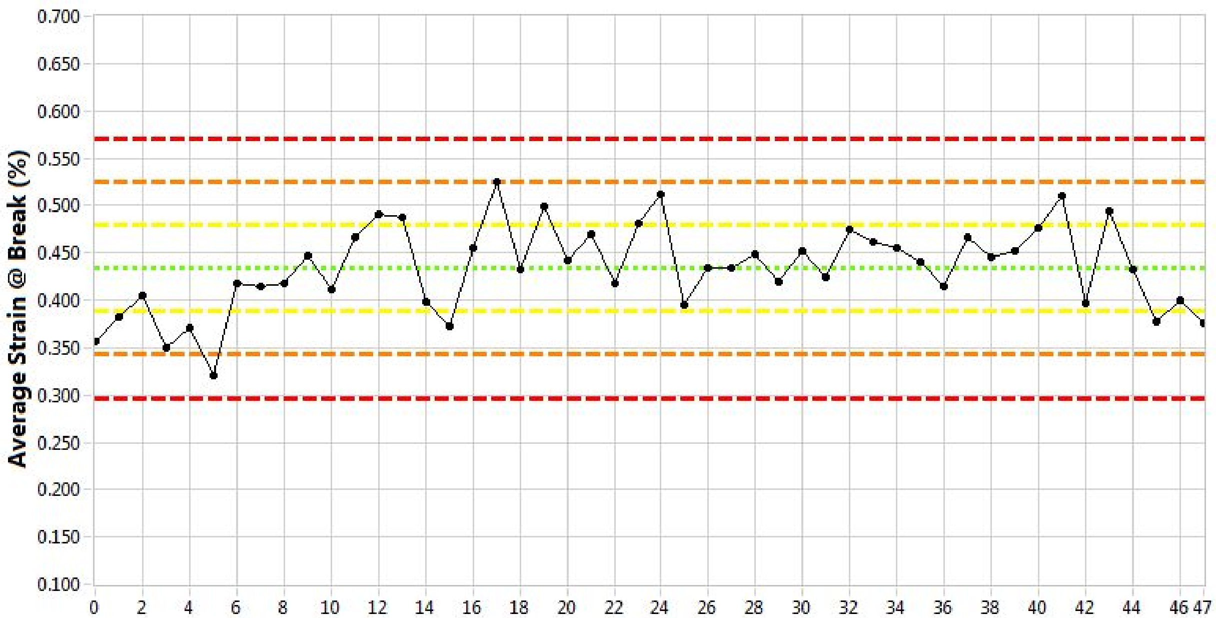


Figure 18. Average strain at break (%), mean = 0.43, standard deviation = 0.046.

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 19) 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.

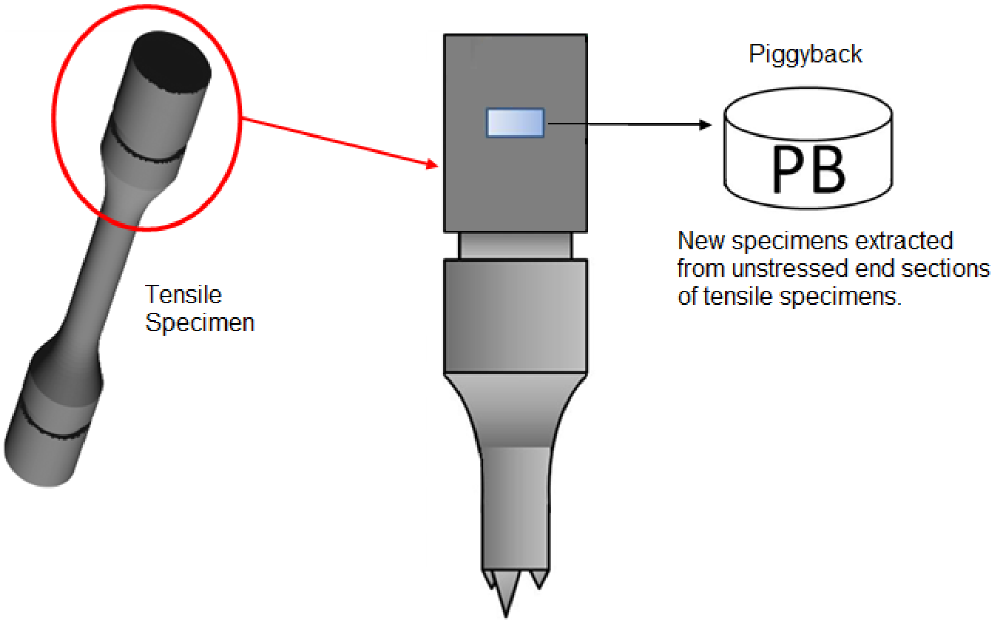


Figure 19. Unstressed specimen remnants from tensile specimens are re-machined into AGC geometries.

RE-MACHINED SPLIT DISC TESTING

Disc splitting tensile strength testing was performed in accordance with PLN-3348, Revision 4, Section 6.1.1.5. This allows for a direct comparison of tensile data to data that were acquired through strict application of ASTM C749-08.[11] Figure 20 and Figure 21 show strength and load data from the split disc testing. While the mean strength value calculated from the split disc testing was slightly lower than that from the traditional tensile testing (Figure 22), there was less spread in the data.

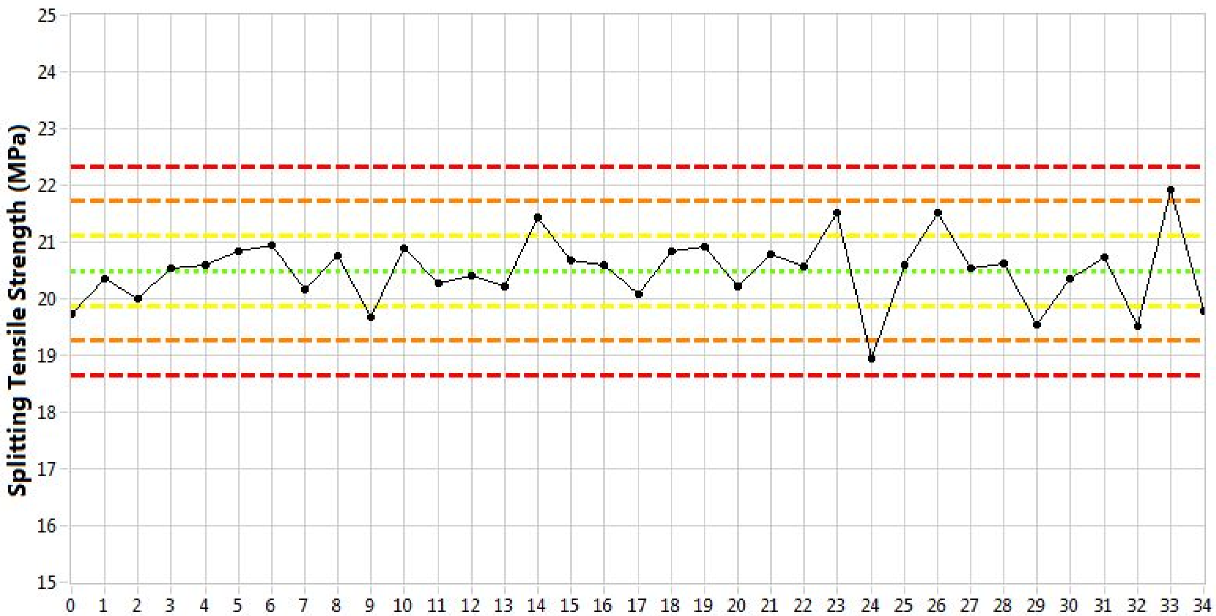


Figure 20. Splitting disc tensile strength (MPa), mean = 20.5, standard deviation = 0.62.

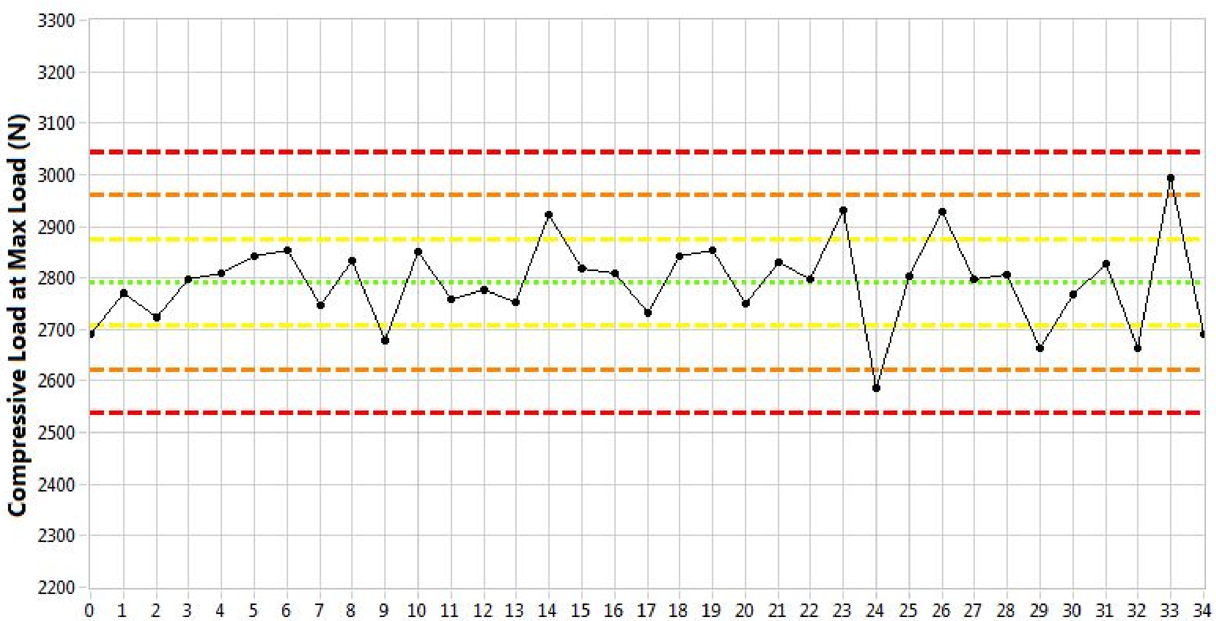


Figure 21. Splitting disc compressive load at max load (N), mean = 2791.5, standard deviation = 84.5.

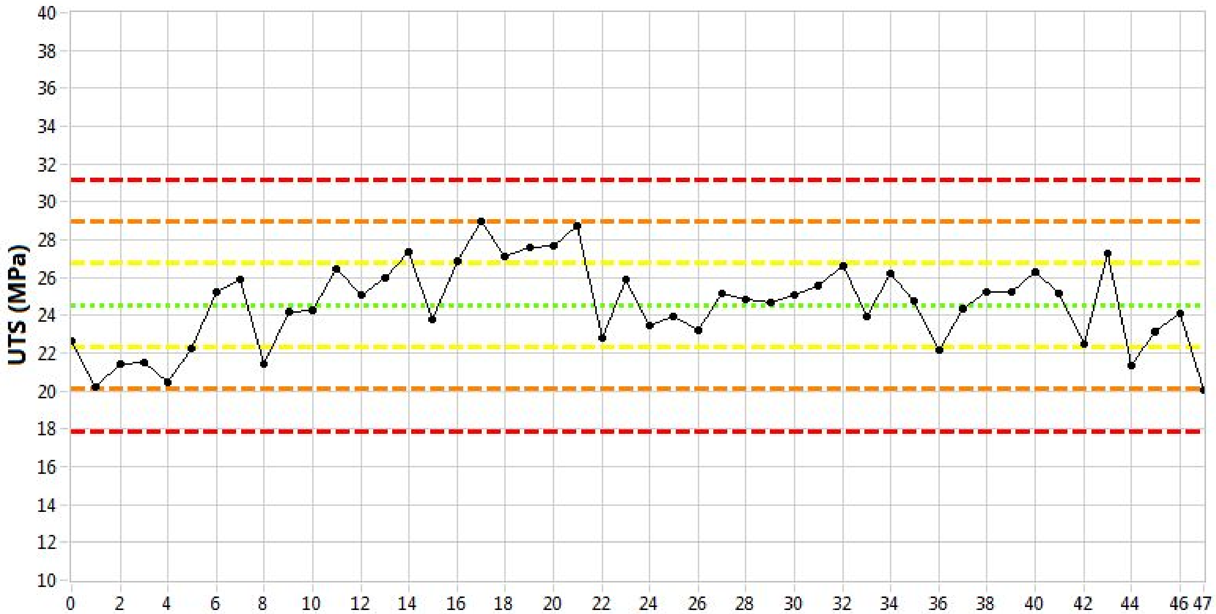


Figure 22. Ultimate tensile strength (MPa), mean = 24.5, standard deviation = 2.21.

RE-MACHINED SPECIMEN DIFFUSIVITY

Thermal diffusivity values are collected from the re-machined tensile specimens per ASTM E1461-07.[12] 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 23.

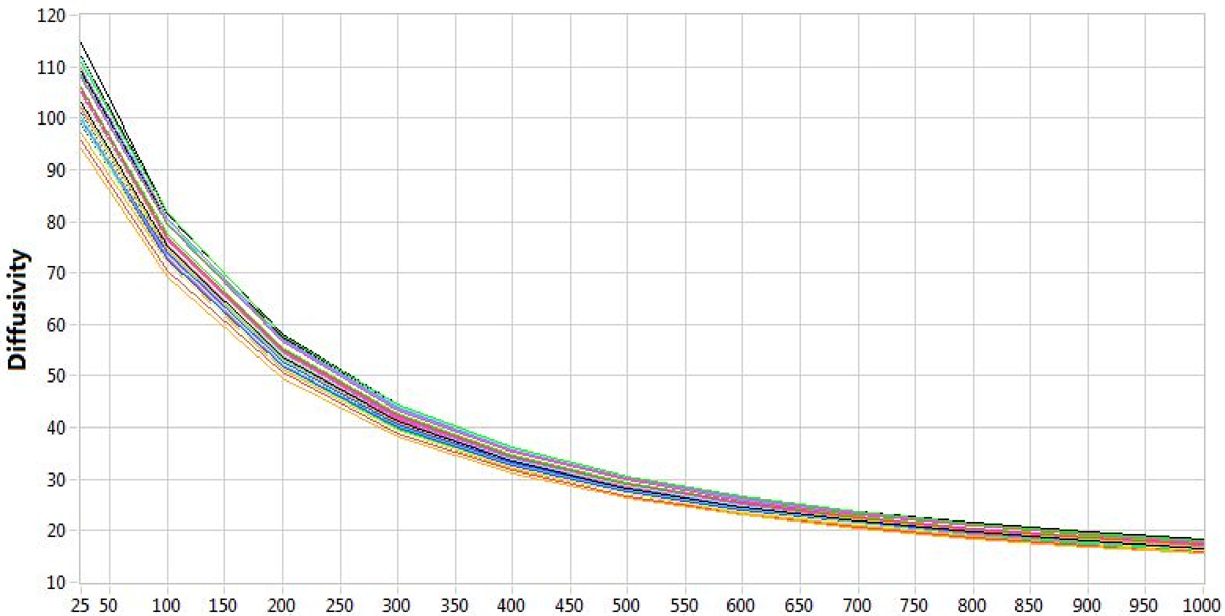


Figure 23. Diffusivity (mm^2/sec).

SUMMARY

The comprehensive data sets for the IG-110 billet 110-08-9-052-7 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: compressive, flexural, and 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 or not obvious errors were made with the data collected, such as 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.

REFERENCES

1. PLN-2497, 2010, “Graphite Technology Development Plan,” Rev. 1, October 2010.

2. PLN-3348, 2011, “Graphite Mechanical Testing,” Rev. 2, April 2011.

3. PLN-3467, 2011, “Baseline Graphite Characterization Plan: Electromechanical Testing,” Rev. 1, August 2011.

4. PLN-3267, 2010, “AGC-2 Characterization Plan,” Rev. 0, March 2010.

5. Carroll, Mark, Joe Lord, and David Rohrbaugh, 2010, *Baseline Graphite Characterization: First Billet*, INL/EXT-10-19910, September 2010.

6. ASTM Standard C695-91 (Reapproved 2010), “Standard Test Method for Compressive Strength of Carbon and Graphite,” ASTM International, 2010.

7. ASTM Standard C559-90 (Reapproved 2005), “Standard Test Method for Bulk Density by Physical Measurements of Manufactured Carbon and Graphite Articles,” ASTM International, 2005.

8. ASTM Standard C651-91 (Reapproved 2005), “Standard Test Method for Flexural Strength of Manufactured Carbon and Graphite Articles Using Four-Point Loading at Room Temperature,” ASTM International, 2005.

9. ASTM Standard C747-93 (Reapproved 2005), “Standard Test Method for Moduli of Elasticity and Fundamental Frequencies of Carbon and Graphite Materials by Sonic Resonance,” ASTM International, 2005.

10. ASTM Standard C1259-08, “Standard Test Method for Dynamic Young’s Modulus, Shear Modulus, and Poisson’s Ratio for Advanced Ceramics by Impulse Excitation of Vibration,” ASTM International, 2008.

11. ASTM Standard C749-08, “Standard Test Method for Tensile Stress-Strain of Carbon and Graphite,” ASTM International, 2008.

12. ASTM Standard E1461-07, “Standard Test Method for Thermal Diffusivity by the Flash Method,” ASTM International, 2007.

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Appendix A

Additional Compression Specimen Database Plots (IG-110 08-9-052-7C)

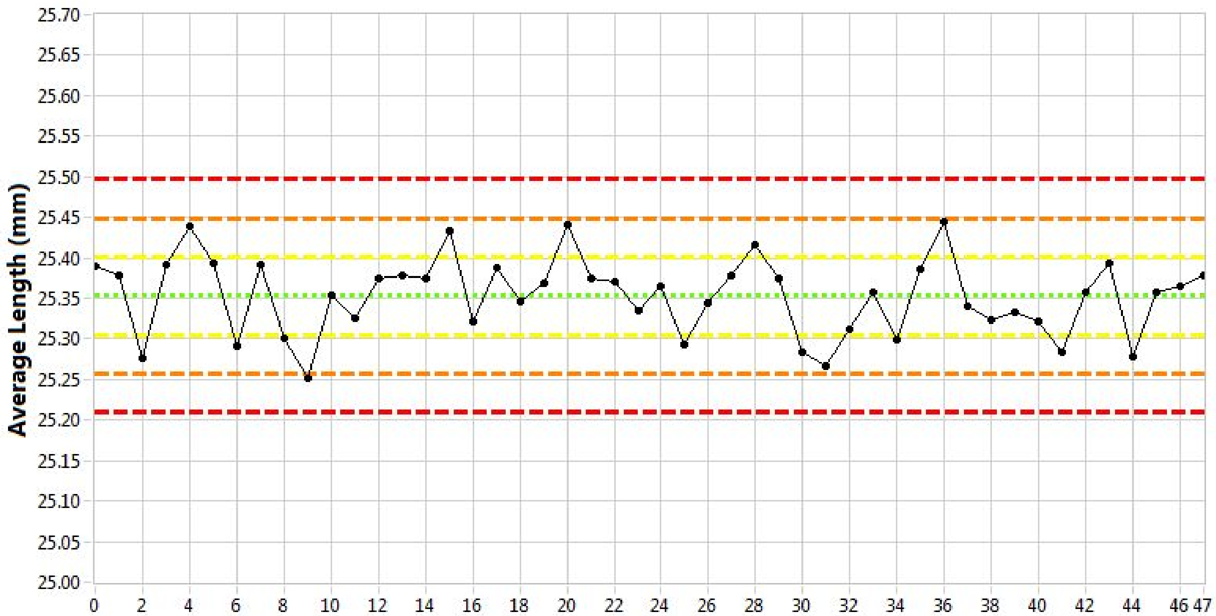

Figure A-1. Average length (mm), mean = 25.353, standard deviation = 0.048.

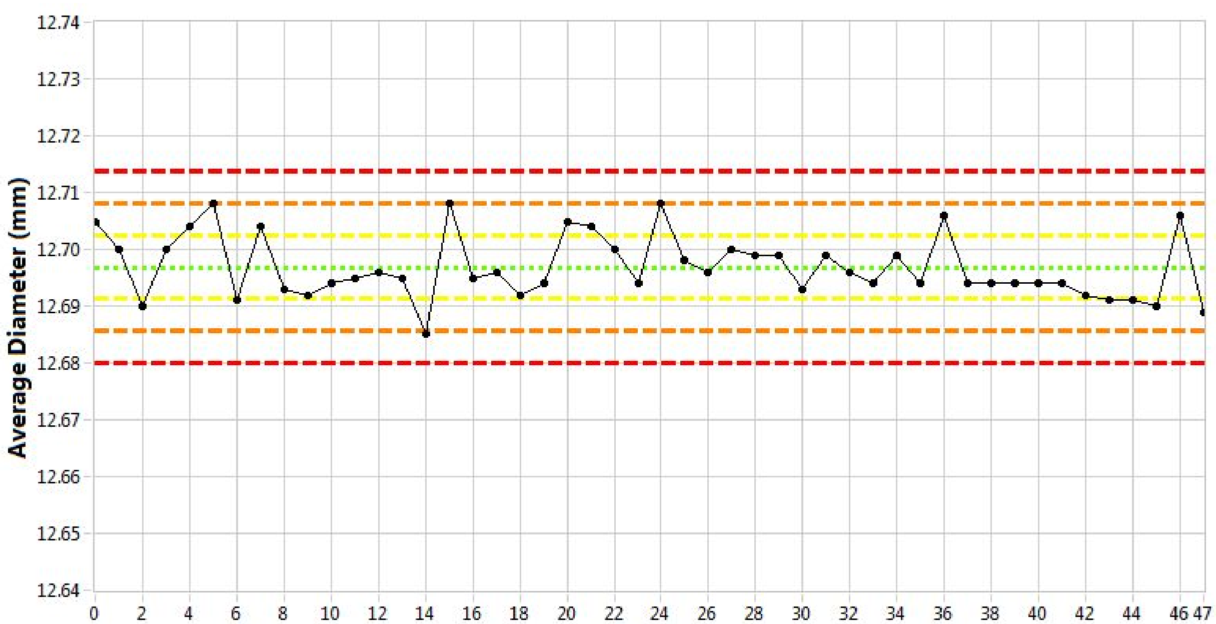

Figure A-2. Average diameter (mm), mean = 12.6968, standard deviation = 0.0056.

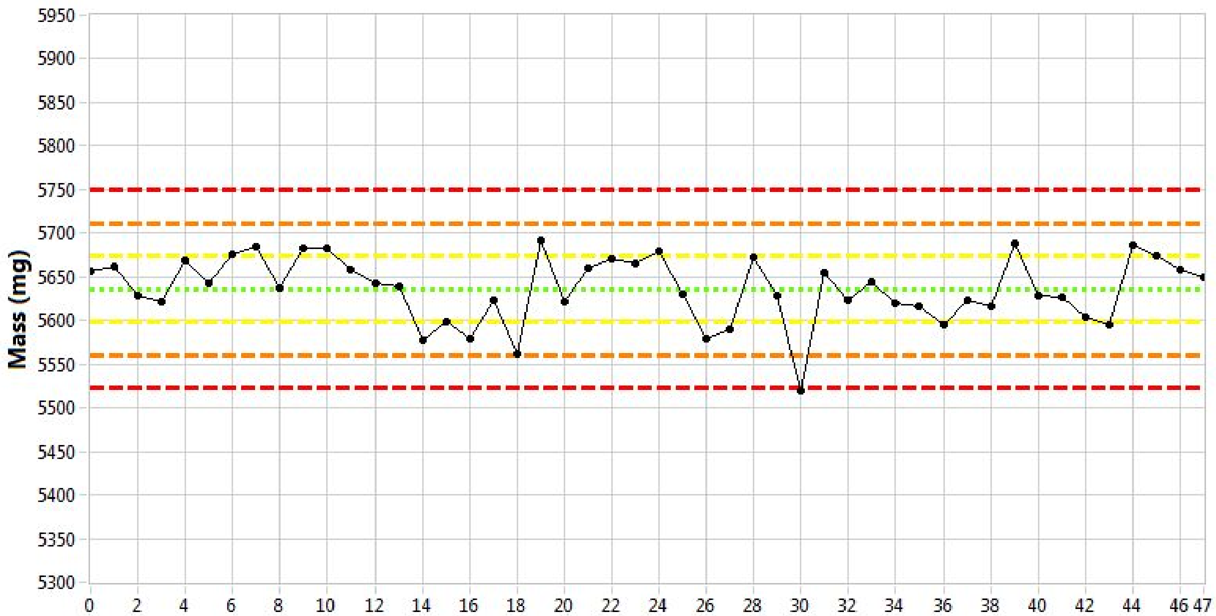

Figure A-3. Mass (mg), mean = 5636.2, standard deviation = 37.81.

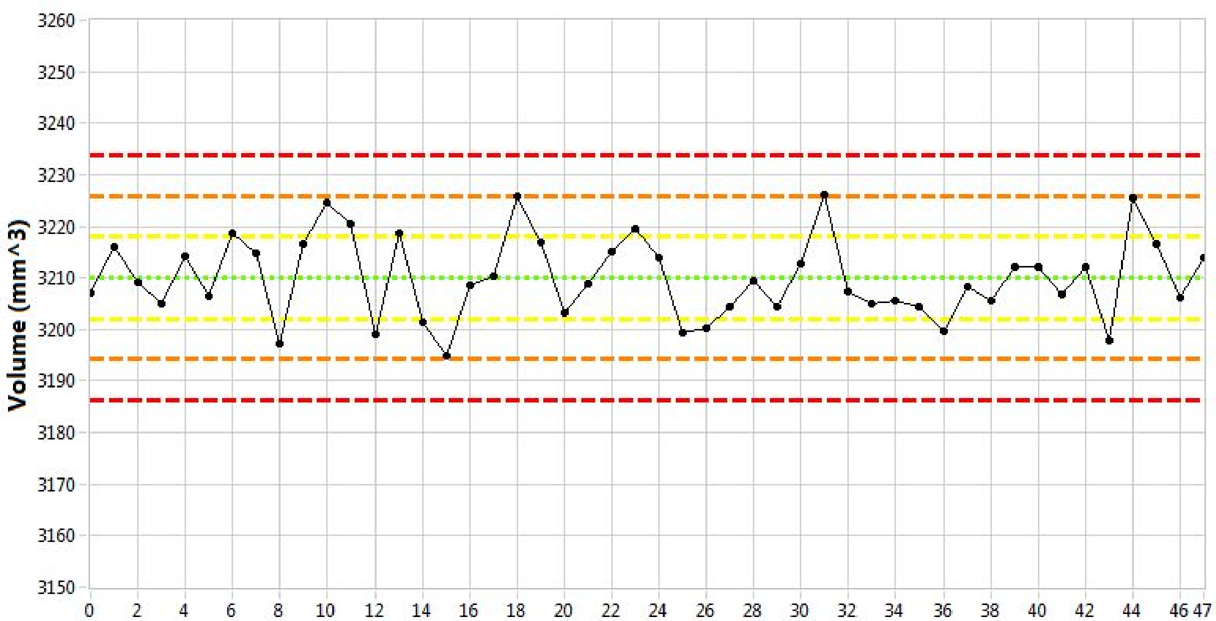

Figure A-4. Volume (mm^3), mean = 3210.0, standard deviation = 7.91.

Appendix B

Additional Flexural Specimen Database Plots (IG-110 08-9-052-7F)

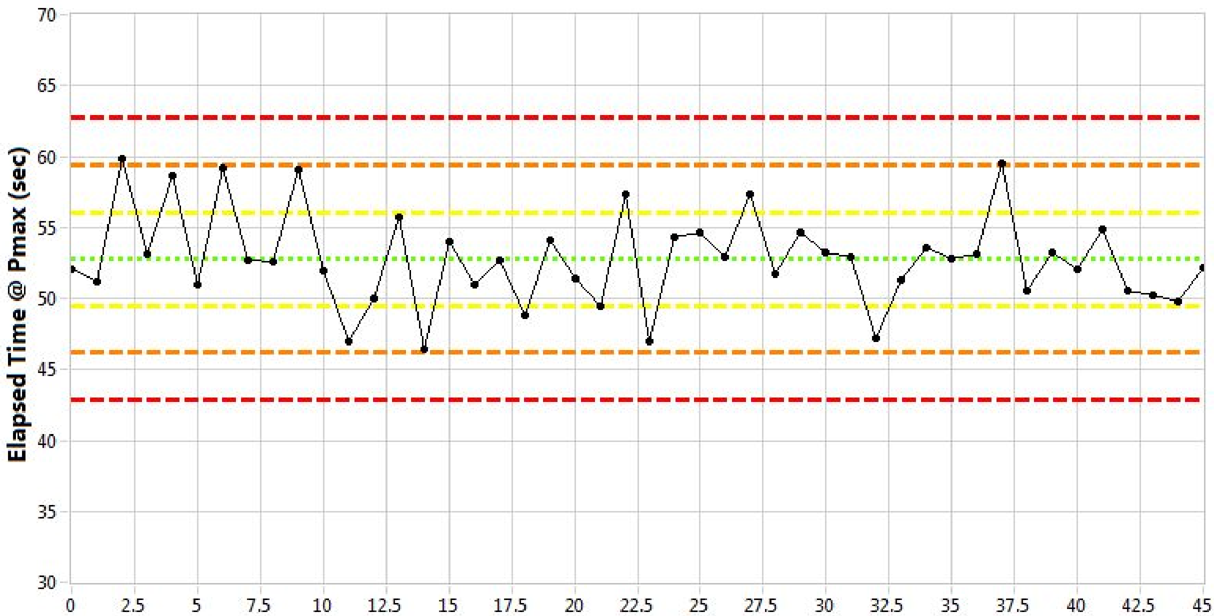

Figure B-1. Elapsed time at max load (sec), mean = 52.8, standard deviation = 3.31.

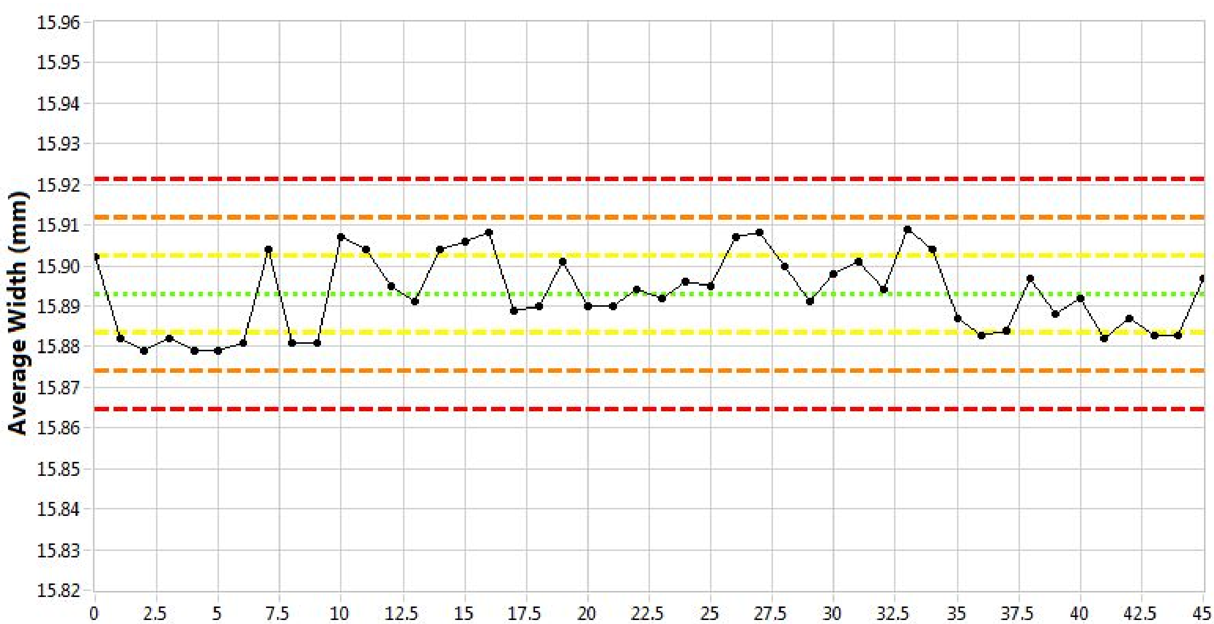

Figure B-2. Minimum Width (mm), mean = 15.893, standard deviation = 0.0094.

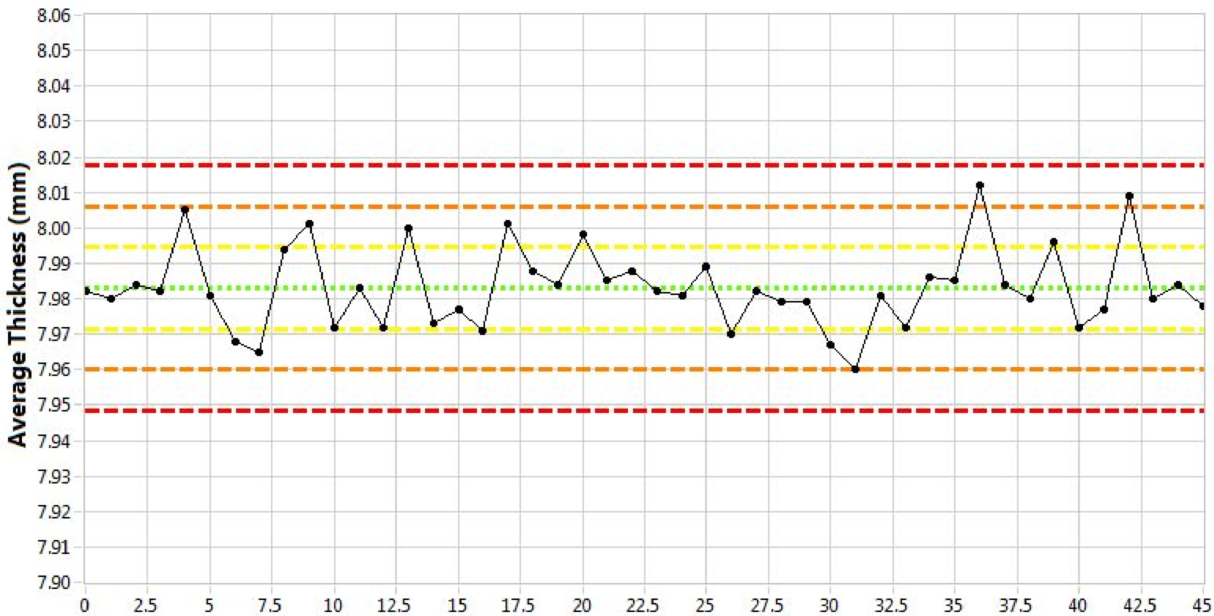

Figure B-3. Minimum thickness (mm), mean = 7.983, standard deviation = 0.0115.

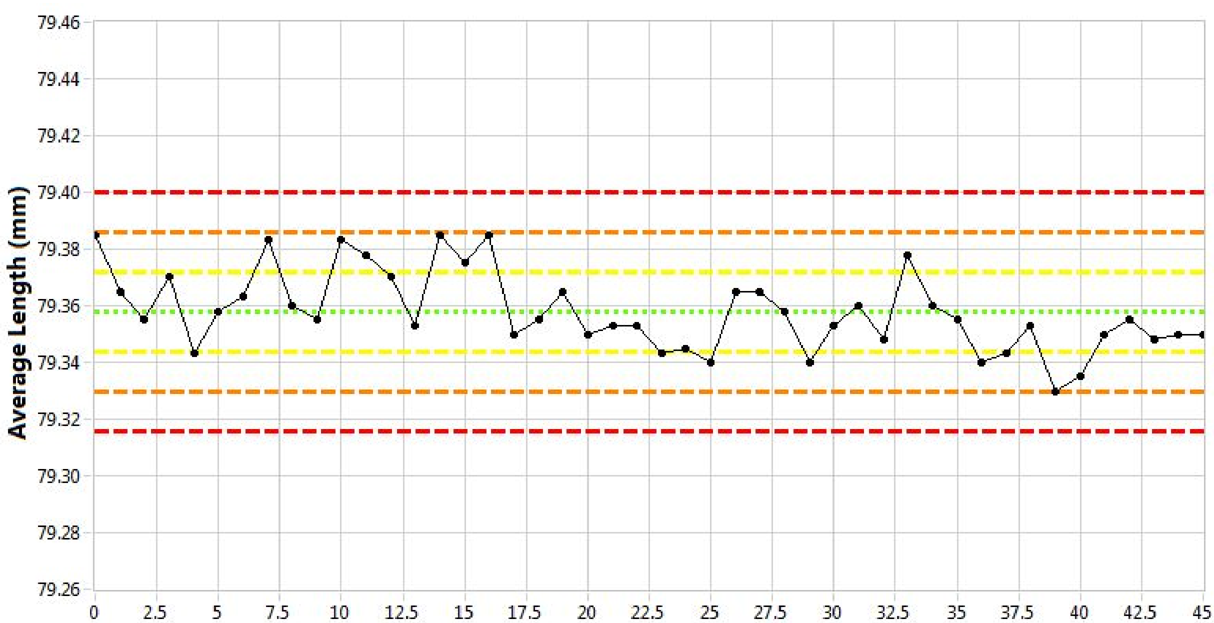

Figure B-4. Minimum length (mm), mean = 79.358, standard deviation = 0.0140.

Appendix C

Additional Tensile Specimen Database Plots (IG-110 08-9-052-7T)

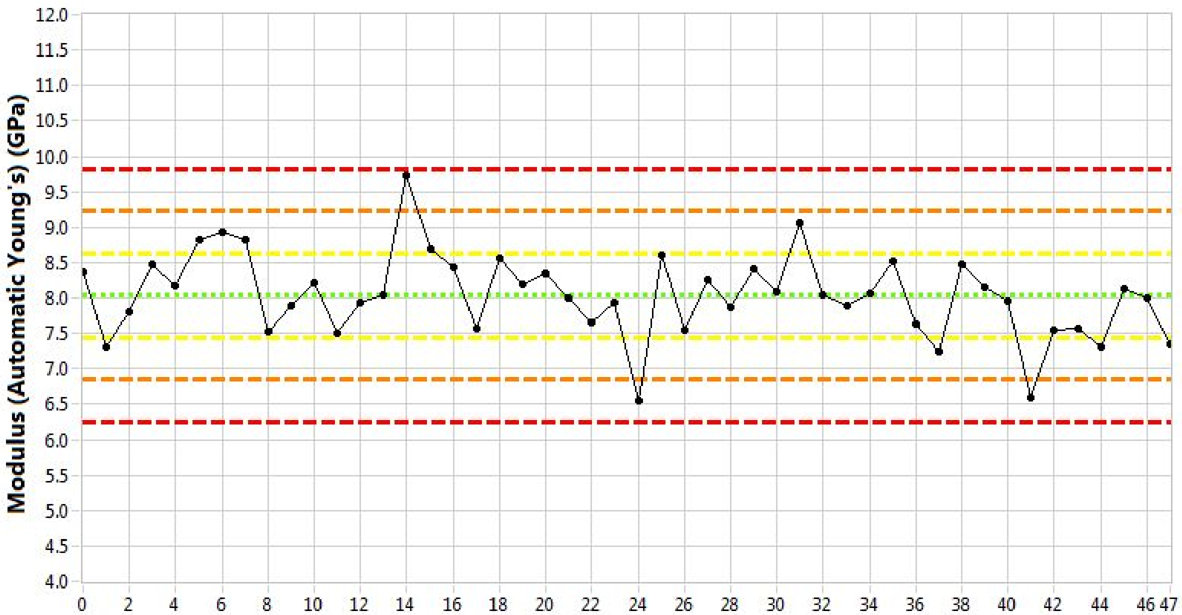

Figure C-1. Modulus (Automatic Young's) (GPa), mean = 8.0, standard deviation = 0.60.

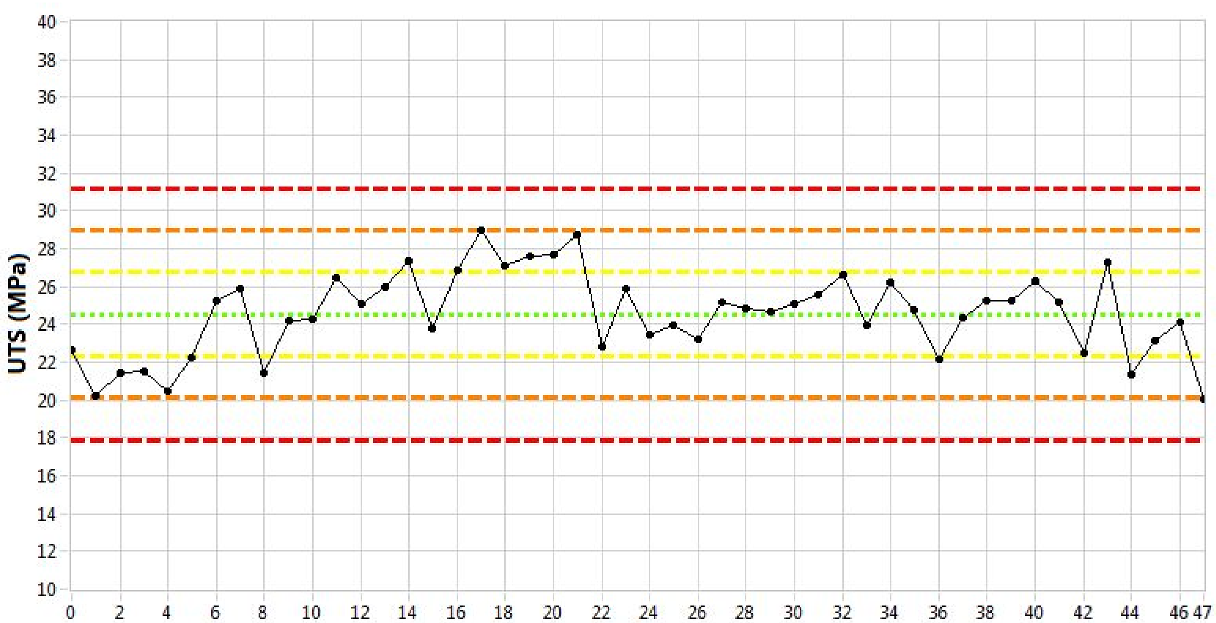

Figure C-2. Ultimate tensile strength (MPa), mean = 24.5, standard deviation = 2.21.

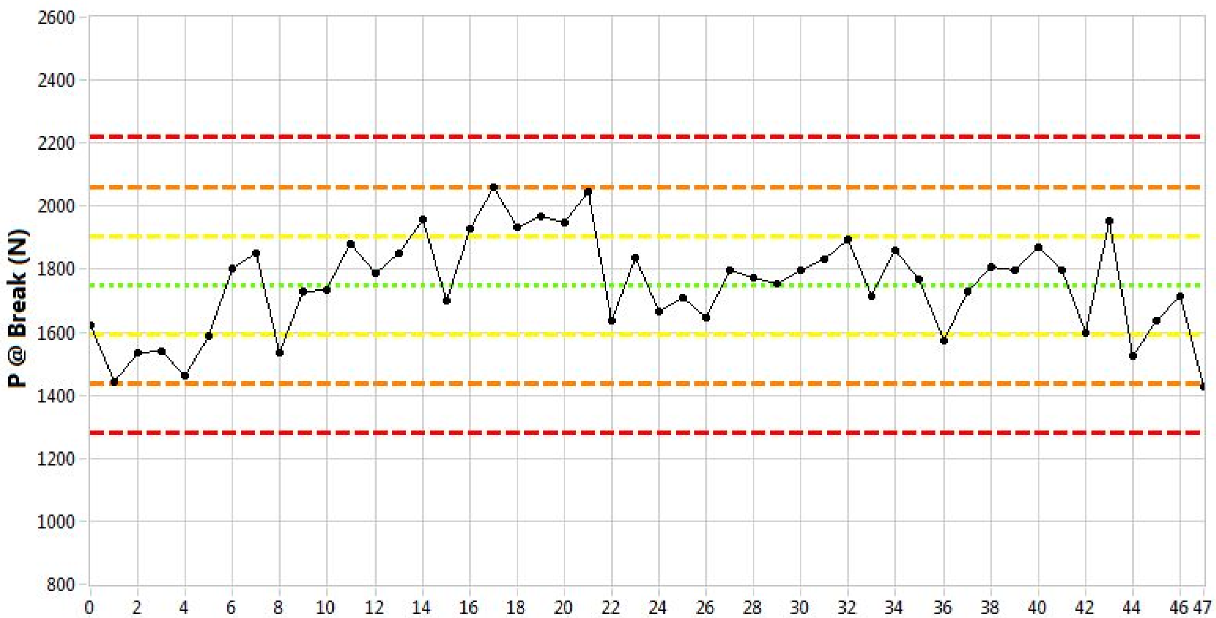

Figure C-3. Load at break (N), mean = 1750, standard deviation = 156.2.

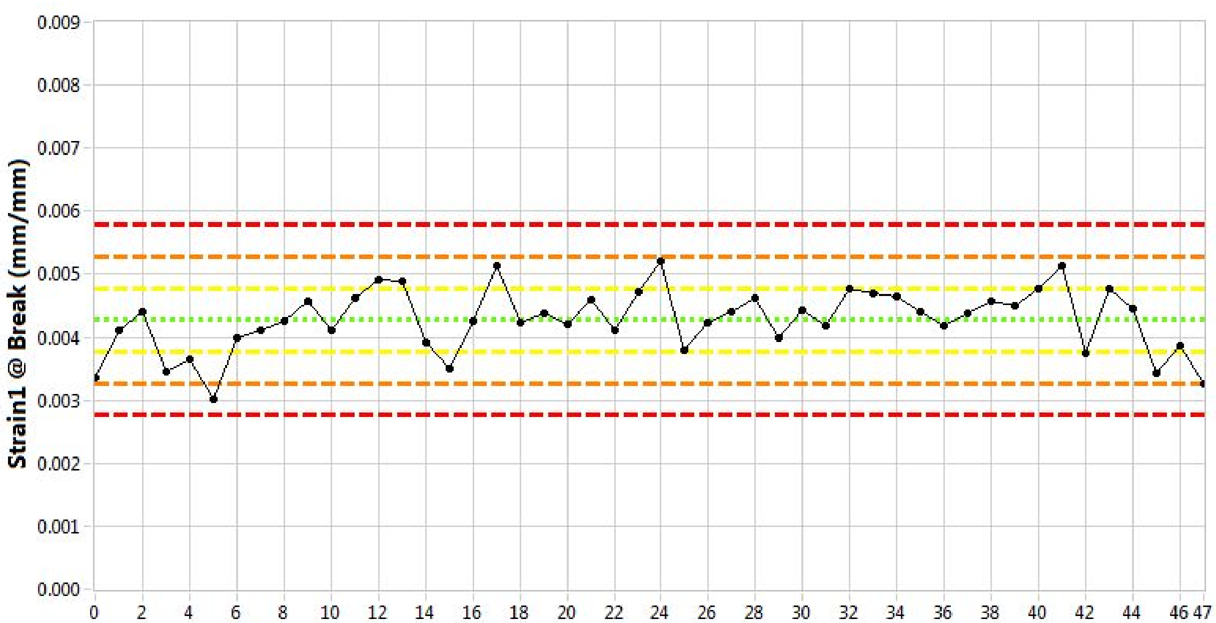

Figure C-4. Strain1 at break (mm/mm), mean = 0.0043, standard deviation = 0.000503.

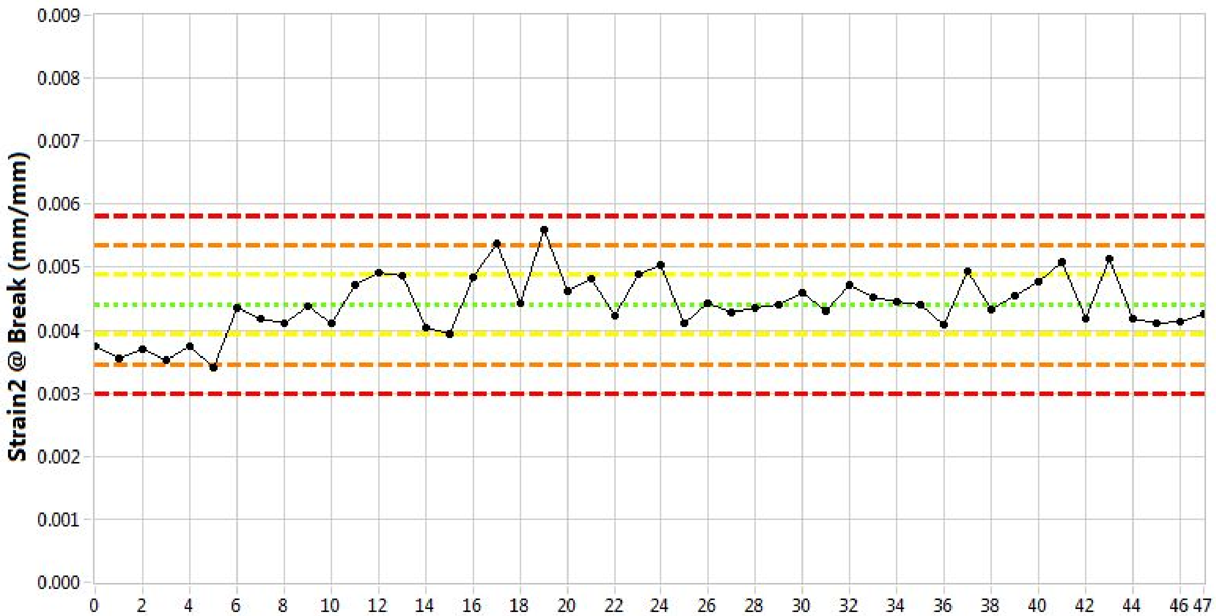

Figure C-5. Stain2 at break (mm/mm), mean = 0.0044, standard deviation = 0.0005.

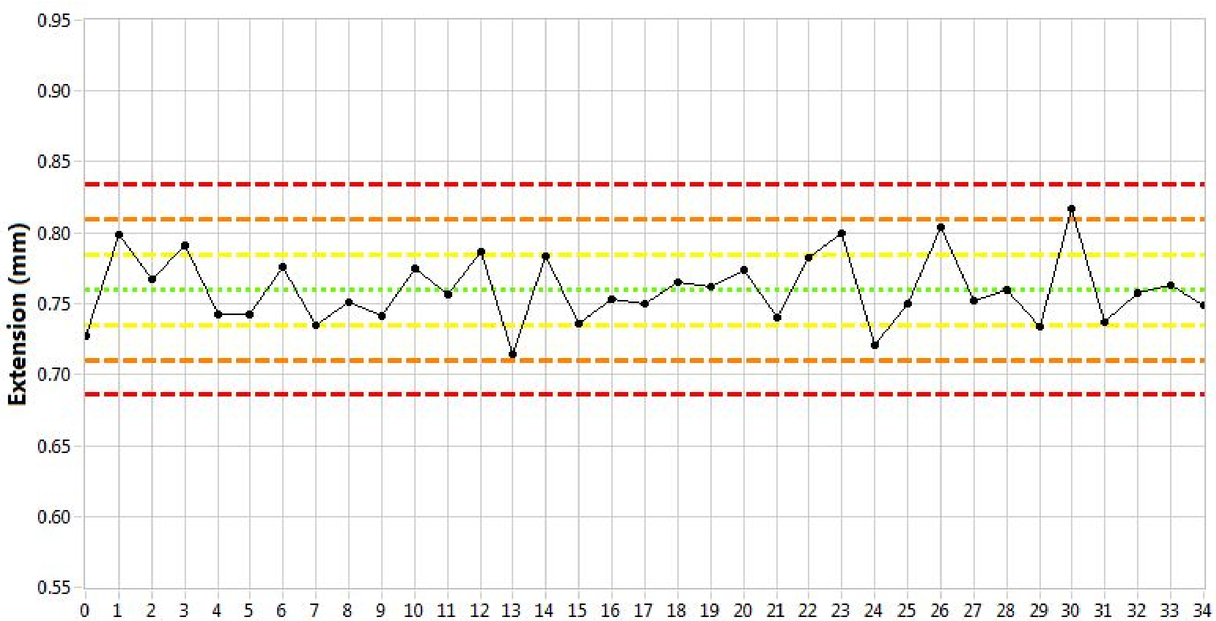

Figure C-6. Extension (mm), mean = 0.7599, standard deviation = 0.0247.

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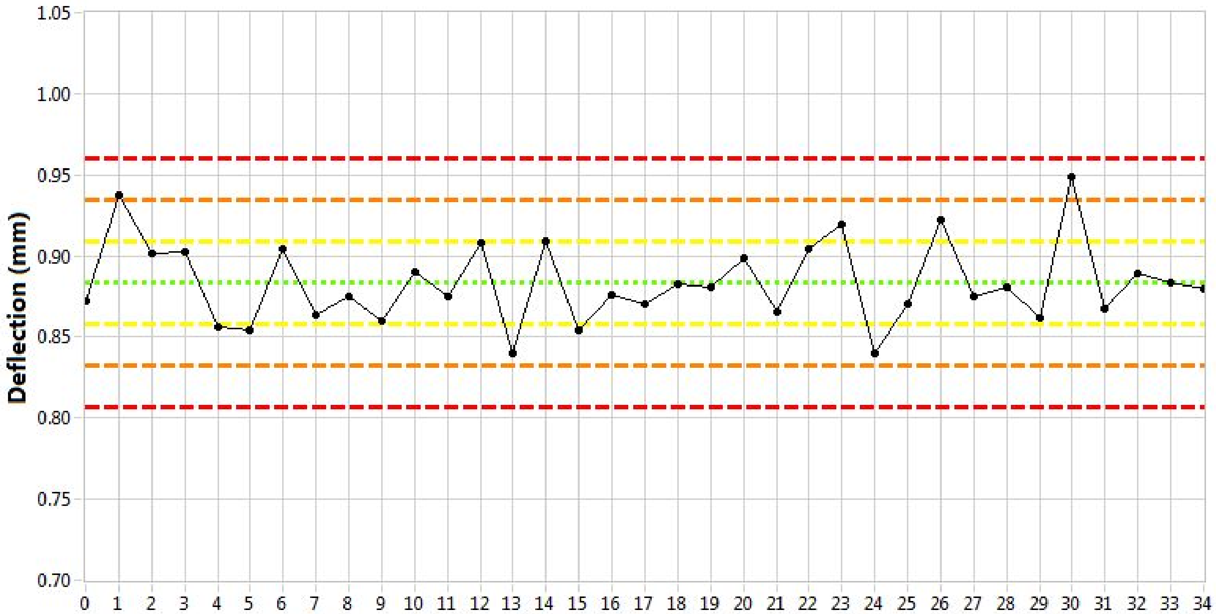

Figure C-7. Deflection (mm), mean = 0.8834, standard deviation = 0.0255.