



# **ECAR-3727 BASELINE CHARACTERIZATION DATABASE VERIFICATION REPORT - NBG-17 BILLET 830-3**

July 2017

*Changing the World's Energy Future*

David T Rohrbaugh





#### **DISCLAIMER**

This information was prepared as an account of work sponsored by an agency of the U.S. Government. Neither the U.S. Government nor any agency thereof, nor any of their employees, makes any warranty, expressed or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness, of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. References herein to any specific commercial product, process, or service by trade name, trade mark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the U.S. Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the U.S. Government or any agency thereof.



**ECAR-3727 BASELINE CHARACTERIZATION  
DATABASE VERIFICATION REPORT - NBG-17 BILLET  
830-3**

**David T Rohrbaugh**

**July 2017**

**Idaho National Laboratory  
Idaho Falls, Idaho 83415**

**<http://www.inl.gov>**


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



**ENGINEERING CALCULATIONS AND ANALYSIS**

INL/RPT-23-71707


Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3ECAR No.: 3727 Rev. No.: 0 Project No.: 32138 Date: 07/31/2017


Performer: David Rohrbaugh D520  7/10/2017  
(Signature) (Date)

Checker<sup>1</sup>: W. David Swank B120  7/11/17  
(Signature) (Date)

Independent Peer  
Reviewer<sup>2</sup>: N/A


CUI Reviewer: N/A  
Yes ☐ or No<sup>5</sup> ☒

Manager<sup>3</sup>: Scott McBride D520  7.10.17  
(Signature) (Date)

Owner<sup>4</sup>: William Windes B120  7-11-17  
(Signature) (Date)

Nuclear Safety<sup>4</sup>: N/A

Cognizant Engineer<sup>4</sup>: N/A

Quality Assurance: Michelle Sharp H330  7/31/17  
(Signature) (Date)

1. Confirmation of completeness, mathematical accuracy, and correctness of data and appropriateness of assumptions.
2. Concurrence of method or approach. See definition in LWP-10106.
3. Concurrence of procedure compliance. Concurrence with method/approach and conclusion.
4. Concurrence with the document's assumptions and input information. See definition of acceptance in LWP-10200.
5. Does the document contain CUI material? Please check either yes or no.



ECAR No.: 3727      Rev. No.: 0      Project No.: 32138      Date: 07/31/2017

[illegible]



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

1. Quality Level (QL) No.	NA	<b>Professional Engineer's Stamp</b>  <b>NA</b> <b>See LWP-10010 for requirements</b>
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: <p>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.</p> <p>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 830-3 and facilitates release of associated data to the NDMAS custodians.</p>		
8. If revision, please state the reason and list sections and/or pages being affected:  NA		
9. Conclusions/Recommendations: <p>A review of the data spreadsheets compiled from physical and mechanical property measurements on nuclear-grade graphite NBG-17 Billet 830-3 revealed no notable errors or omissions that preclude the transfer of these data to the NDMAS site for storage.</p> <p>In addition to a full visual review of the data files to determine whether obvious errors, such as missing information, were made regarding the data collected, graphical representations were made of individual evaluations in order 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.</p>		



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727      Rev. No.: 0      Project No.: 32138      Date: 07/31/2017

**CONTENTS**

PROJECT ROLES AND RESPONSIBILITIES .....	7
SCOPE AND BRIEF DESCRIPTION .....	8
DESIGN OR TECHNICAL PARAMETER INPUT AND SOURCES .....	8
ASSUMPTIONS .....	8
COMPUTER CODE VALIDATION .....	8
DISCUSSION/ANALYSIS .....	9
Introduction .....	9
Database Analysis .....	9
Compression Specimen Database (NBG-17 830-3C).....	11
Electrical Resistivity, Modulus, CTE.....	16
Density Values .....	19
Flexural Specimen Database (NBG-17 830-3F) .....	19
Density Values .....	23
Tensile Specimen Database (NBG-17 830-3T) .....	26
RE-MACHINED SPECIMEN PROPERTIES.....	29
RE-MACHINED SPLIT DISC TESTING .....	29
RE-MACHINED SPECIMEN DIFFUSIVITY.....	31
SUMMARY .....	31
REFERENCES .....	31
Appendix A Additional Compression Specimen Database Plots (NBG-17 830-3) .....	A-1
Appendix B Additional Flexural Specimen Database Plots (NBG-17 830-3).....	B-1
Appendix C Additional Tensile Specimen Database Plots (NBG-17 830-3).....	C-1



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727      Rev. No.: 0      Project No.: 32138      Date: 07/31/2017

## FIGURES

Figure 1. The three types of mechanical test specimens that will be machined from stock graphite and provide the basis for material property evaluations. ....	10
Figure 2. Individual specimen extraction and tracking identification from NBG-17 Billet 830-3. ....	10
Figure 3. Compressive load at maximum load (N), mean = 9495, standard deviation = 898....	11
Figure 4. Compressive stress at maximum load (MPa), mean = 75.0, standard deviation = 7.1. ....	12
Figure 5. Displacement at maximum load (mm), mean = -0.6824, standard deviation = 0.0798. ....	12
Figure 6. Compressive strain (mm/mm), mean = 0.0269, standard deviation = 0.0031. ....	13
Figure 7. Fracture categorization results and description. ....	14
Figure 8. Maximum load (N), mean = 2389, standard deviation = 176. ....	15
Figure 9. Maximum flexure stress (MPa), mean = 47.5, standard deviation = 3.5.....	15
Figure 10. Electrical resistivity ( $\mu\Omega\text{-m}$ ), mean = 10, standard deviation = 0.5.....	16
Figure 11. Young's modulus by sonic velocity method (GPa), mean = 14.3, standard deviation = 0.3. ....	17
Figure 12. Shear modulus by sonic velocity method (GPa), mean = 4.99, standard deviation = 0.1. ....	17
Figure 13. Young's modulus by sonic resonance method (GPa), mean = 11.4, standard deviation = 0.3.....	18
Figure 14. Mean coefficient of thermal expansion (1/K).....	18
Figure 15. Density ( $\text{g/cm}^3$ ), mean = 1.8214, standard deviation = 0.0113.....	19
Figure 16. Average width (mm), mean = 15.883, standard deviation = 0.0183. ....	20
Figure 17. Average thickness (mm), mean = 8.131, standard deviation = 0.0067.....	21
Figure 18. Average length (mm), mean = 79.377, standard deviation = 0.0075. ....	21
Figure 19. Maximum load (N), mean = -524.6, standard deviation = 30.6. ....	22
Figure 20. Maximum flexure stress (MPa), mean = 30.0, standard deviation = 1.7.....	22
Figure 21. Mid-span deflection at maximum load ( $\mu\text{m}$ ), mean = -0.3733, standard deviation = 0.0242. ....	23
Figure 22. Density ( $\text{g/cm}^3$ ), mean = 1.8266, standard deviation = 0.0108.....	24
Figure 23. Flexural vibration mode modulus (GPa), mean = 11.2, standard deviation = 0.3. ....	25



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727      Rev. No.: 0      Project No.: 32138      Date: 07/31/2017

Figure 24. Torsional vibration mode shear modulus (GPa), mean = 4.6, standard deviation = 0.1. ....	25
Figure 25. Minimum gauge diameter (mm), mean = 8.763, standard deviation = 0.0045.....	26
Figure 26. Maximum load (N), mean = 1256, standard deviation = 84. ....	27
Figure 27. Ultimate tensile strength (MPa), mean = 20.8, standard deviation = 1.4. ....	27
Figure 28. Stress at break (MPa), mean = 20.8, standard deviation = 1.4. ....	28
Figure 29. Average strain at break (%), mean = 0.29, standard deviation = 0.022.....	28
Figure 30. Unstressed specimen remnants from tensile specimens are re-machined into AGC-sized piggyback specimens. ....	29
Figure 31. Disc splitting tensile strength (MPa), mean = 19.6, standard deviation = 1.3. ....	30
Figure 32. Disc splitting compressive load at max load (N), mean = 2670, standard deviation = 171. ....	30
Figure 33. Re-machined specimen diffusivity. ....	31
Figure A-1. Average length (mm), mean = 25.3992, standard deviation = 0.0249. ....	1
Figure A-2. Average diameter (mm), mean = 12.6951, standard deviation = 0.0086. ....	1
Figure A-3. Mass (mg), mean = 5,861.7, standard deviation = 36.6.....	2
Figure A-4. Volume (mm <sup>3</sup> ), mean = 3,218.3, standard deviation = 4.7.....	2
Figure A-5. Mid-span deflection at maximum load (um), mean = -0.4959, standard deviation = 0.1. ....	3
Figure A-6. Elapsed time at maximum load (sec), mean = 82.5, standard deviation = 4.8.....	3
Figure B-1. Elapsed time at maximum load (sec), mean = 38.9, standard deviation = 3.1.....	1
Figure B-2. Mass (mg), mean = 18725.3, standard deviation = 110.1.....	1
Figure B-3. Volume (mm <sup>3</sup> ), mean = 10251.7, standard deviation = 14.9.....	2
Figure C-1. Modulus (automatic Young's) (GPa), mean = 10.3, standard deviation = 0.42.....	1
Figure C-2. Load at break (N), mean = 1255, standard deviation = 84.....	1
Figure C-3. Strain 1 at break (mm/mm), mean = 0.0028, standard deviation = 0.0003. ....	2
Figure C-4. Strain 2 at break (mm/mm), mean = 0.003, standard deviation = 0.0003. ....	2
Figure C-5. Re-machined specimens length (mm), mean = 6.338, standard deviation = 0.0133. ....	3
Figure C-6. Re-machined specimens diameter (mm), mean = 12.722, standard deviation = 0.0064. ....	3
Figure C-7. Re-machined specimens density (g/cm <sup>3</sup> ), mean = 1.8262, standard deviation = 0.0123. ....	4



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727      Rev. No.: 0      Project No.: 32138      Date: 07/31/2017

Figure C-8. Disc splitting extension (mm), mean = 0.6263, standard deviation = 0.0188.....4

Figure C-9. Disc splitting deflection (mm), mean = -0.2356, standard deviation = 0.0185.....5



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727      Rev. No.: 0      Project No.: 32138      Date: 07/31/2017

## PROJECT ROLES AND RESPONSIBILITIES

Project Role	Name (Typed)	Organization	Pages covered (if applicable)
Performer	David Rohrbaugh	D520	All
Checker <sup>a</sup>	W. David Swank	B120	All
Independent Reviewer <sup>b</sup>	NA		
CUI Reviewer <sup>c</sup>	NA		All
Manager <sup>d</sup>	Scott McBride	D520	
Requestor <sup>e</sup>	William Windes	B120	
Nuclear Safety <sup>e</sup>	NA		
Document Owner <sup>e</sup>	William Windes	B120	
Quality Assurance	Michelle Sharp	H330	

### Responsibilities:

- a. Confirmation of completeness, mathematical accuracy, and correctness of data and appropriateness of assumptions.
- b. Concurrence of method or approach. See definition in LWP-10106.
- c. Concurrence with the document's markings in accordance with LWP-11202.
- d. Concurrence of procedure compliance. Concurrence with method/approach and conclusion.
- e. Concurrence with the document's assumptions and input information. See definition of acceptance in LWP-10200.

**NOTE:** *Delete or mark "NA" 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.*



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

## **SCOPE AND BRIEF DESCRIPTION**

This engineering calculations and analysis report provides the results of a validity evaluation of the physical and mechanical property data collected on a billet of nuclear-grade graphite (i.e., NBG-17 Billet 830-3) in support of the ART Baseline Graphite Characterization Program.<sup>1,2</sup> 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,"<sup>3</sup> and their 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, "Baseline Graphite Characterization Plan: Electromechanical Testing"; and PLN-3267, "AGC-2 Characterization Plan."<sup>4</sup>

## **ASSUMPTIONS**

None.

## **COMPUTER CODE VALIDATION**

Data collection and storage are organized as reported in PLN-3467 and INL/EXT-10-19910, *Baseline Graphite Characterization: First Billet*.<sup>5</sup> The 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 life-cycle documentation is recorded in accordance with LWP-20000-01, "Conduct of Research Plan."<sup>6</sup> Validation of commercial packages is handled via integrated system checks specific to each new element or upgrade, as appropriate.



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

## **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 in-service behavior of graphite for the latest proposed designs, two main programs are under way. The first, 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 very high-temperature reactor core.<sup>1</sup> 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 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.<sup>2</sup> 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 currently available.

This report covers the release of physical and mechanical property data from a billet of NBG-17 graphite. The graphite billet, NBG-17 830-3, is a block of vibra-molded graphite with a medium grain structure. 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 consists of tabbed spreadsheets occupied by more than 70,000 cells of individual characteristic or property values and associated tagging information.

This report is intended as a validation review of the graphite billet, NBG-17 830-3. The report is not an analysis of property characteristics or trends beyond the evaluation necessary to determine if the collected data are reflective of the properties of this particular graphite billet. The report is an acceptance of the test methods used, data calculations and conversions being carried out, and review of values from the standpoint of determining whether they reflect anomalous behavior that must be further investigated.

Ultimately, this report provides justification for transfer of this data set into a storage and analysis system that is available for internal and external analysts to utilize 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 are provided in INL/EXT-10-19910.<sup>5</sup> 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 in Figure 2. This figure is representative of a single sub-wedge of graphite from this billet.



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

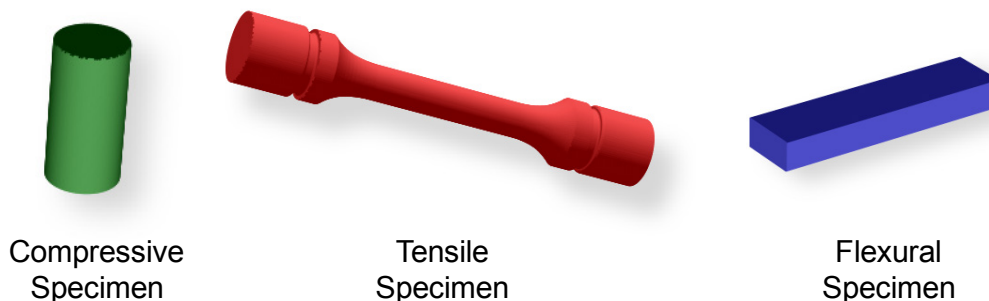


Figure 1. The three types of mechanical test specimens that will be machined from stock graphite and provide the basis for material property evaluations.

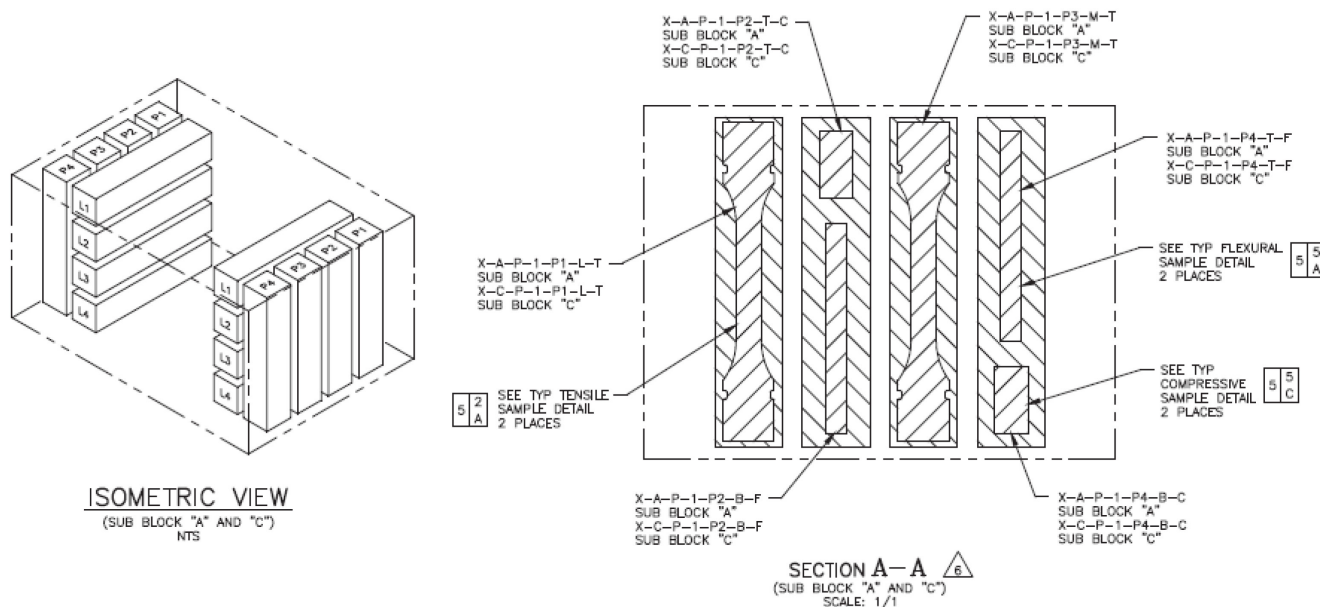


Figure 2. Individual specimen extraction and tracking identification from NBG-17 Billet 830-3.

Sections of this report cover each of the individual databases for this billet, are divided by mechanical test specimen type (i.e., 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 (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 either in the text below or in the three appendices ( $\pm 1$ , 2, and 3 standard deviations are represented by the yellow, orange, and red dotted lines, and the mean is represented by the green line).



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

A goal 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 Database (NBG-17 830-3C)

#### Compression Testing

Compression testing was performed per ASTM C695-15<sup>7</sup> and PLN-3467. Figure 3 shows the maximum applied load for each of the 189 compression specimens from Billet 830-3. As was mentioned previously, some variation in graphite properties is expected, 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 (Figure 3), 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. Although Figures 3–6 show data points that lie outside of 3 standard deviations from the mean, there are no supporting data that necessitate the removal of these data from the database. These slightly lower or slightly higher data are therefore attributed to material variation or flaws within the specimens.

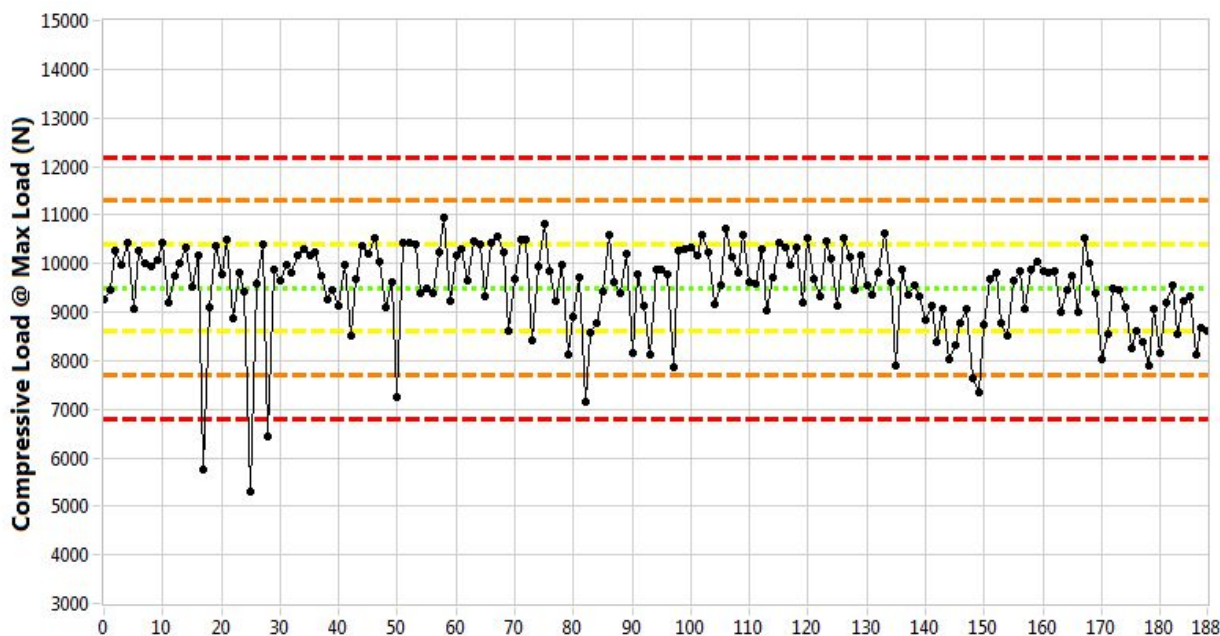


Figure 3. Compressive load at maximum load (N), mean = 9495, standard deviation = 898.



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

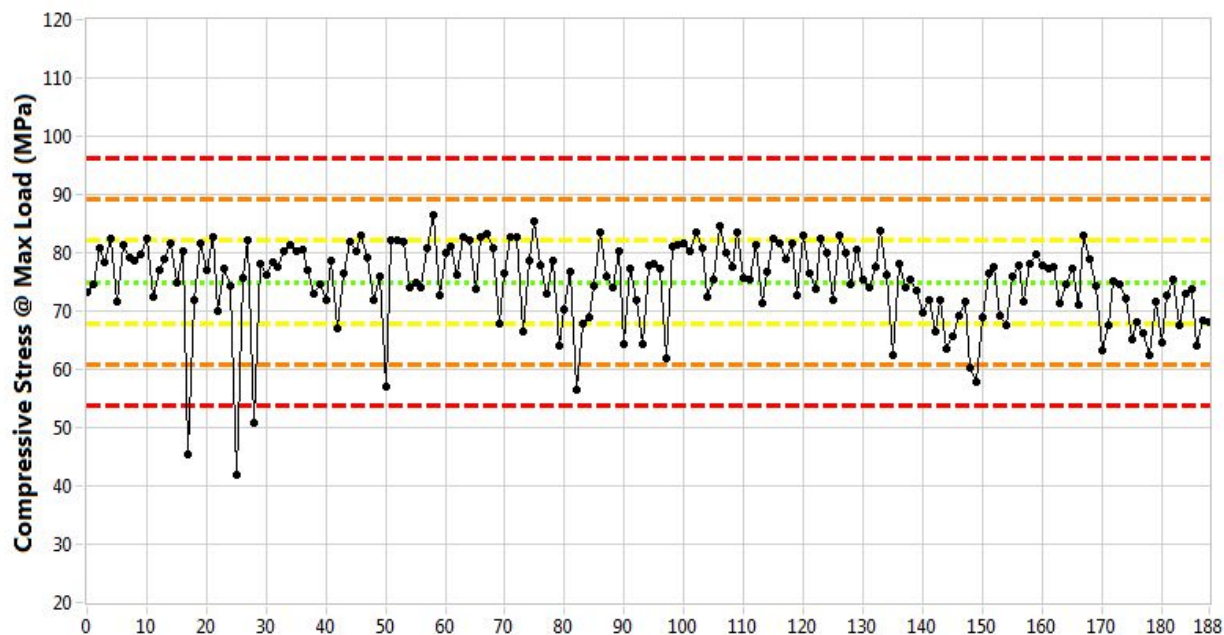


Figure 4. Compressive stress at maximum load (MPa), mean = 75.0, standard deviation = 7.1.

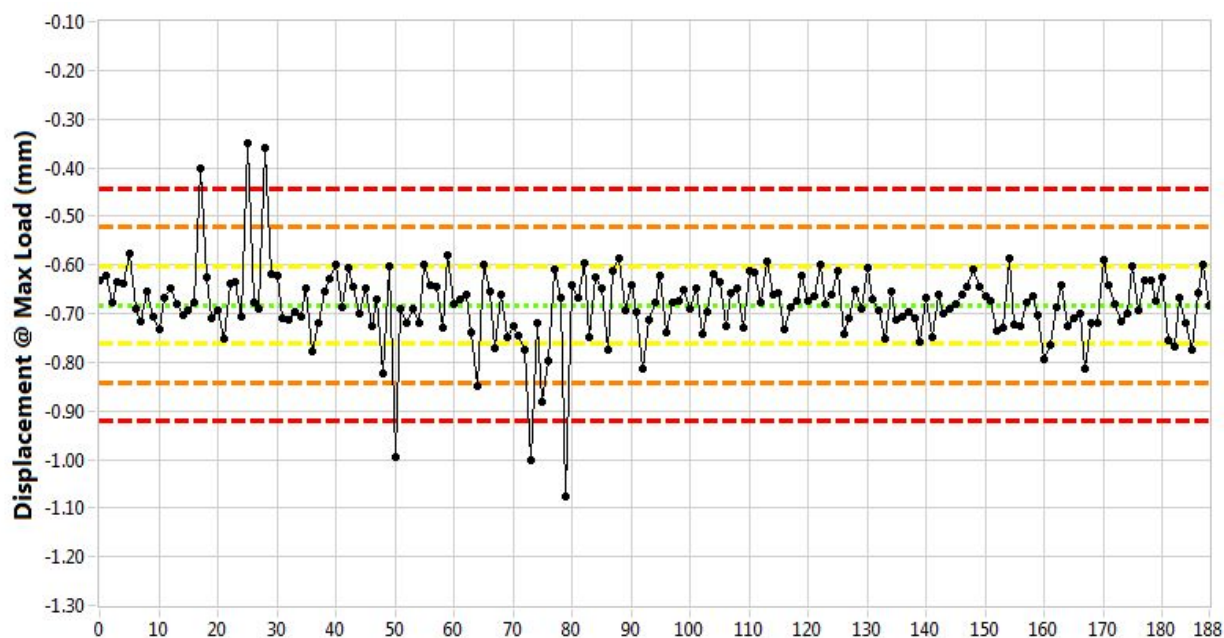


Figure 5. Displacement at maximum load (mm), mean = -0.6824, standard deviation = 0.0798.



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

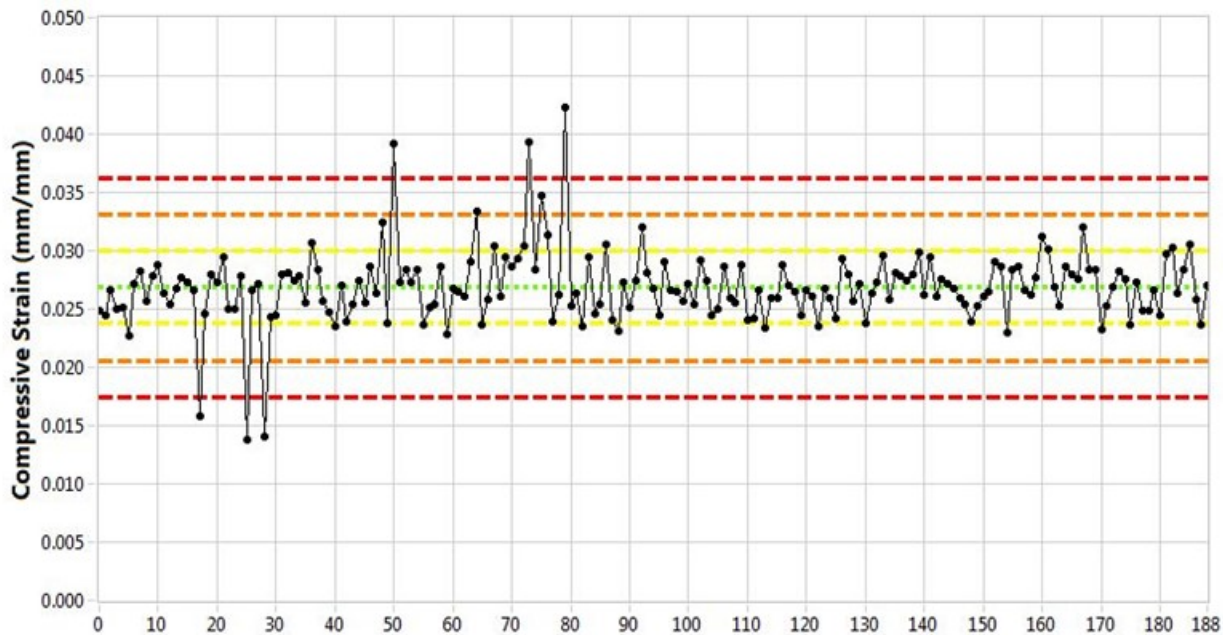


Figure 6. Compressive strain (mm/mm), mean = 0.0269, standard deviation = 0.0031.

### 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 is a screen shot of this categorization, along with distribution of recorded fracture categories for each of the 189 compressive specimens from NBG-17 Billet 830-3 (with no anomalous values indicative of an unallowable characterization).



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

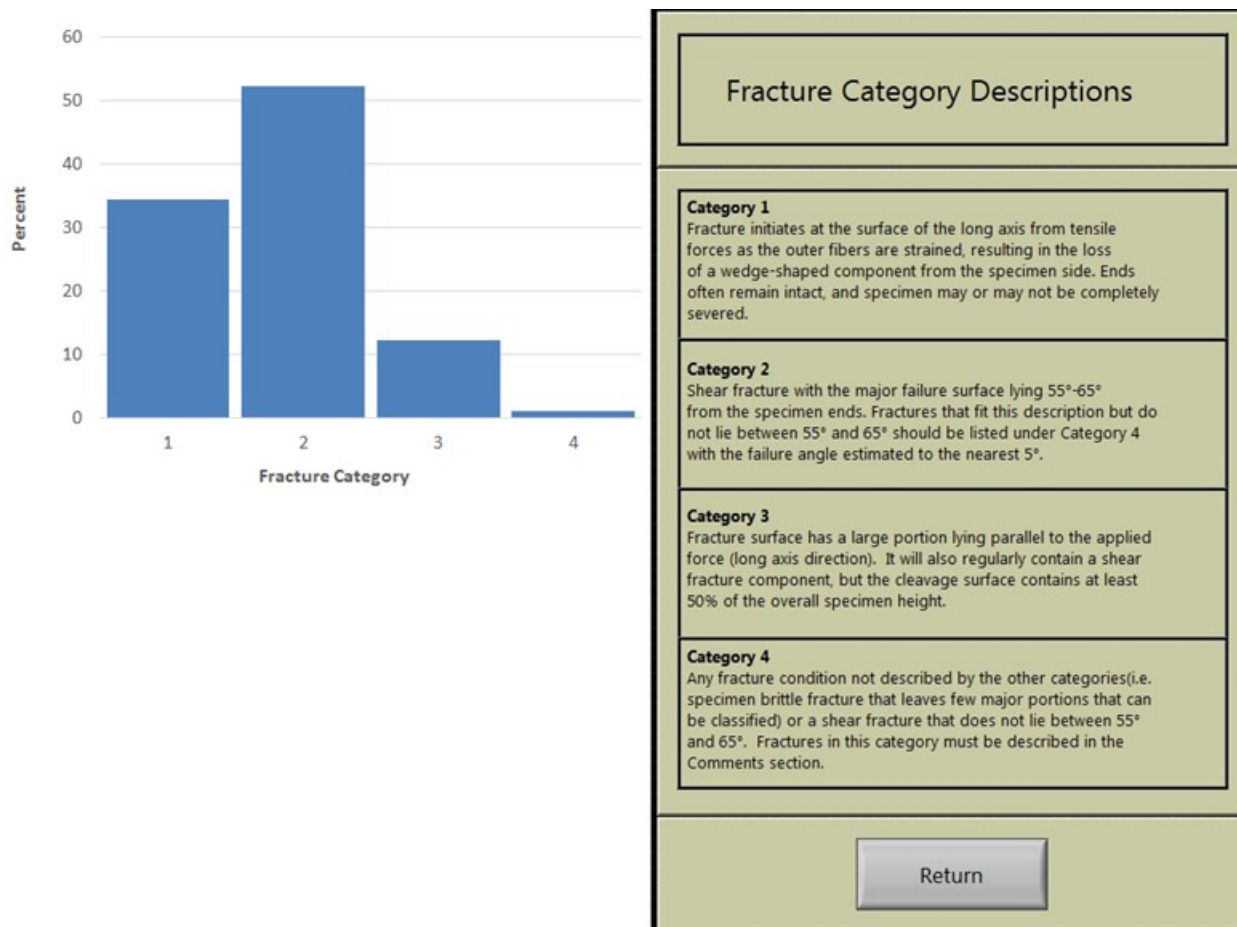


Figure 7. Fracture categorization results and description.

### Three-Point Bend Testing

Thirty-three of the compression specimens were held back from compression testing, specifically for three-point bend testing. These data were subsequently kept in the compression data spreadsheet file. The three-point bend test was recently adopted for evaluating carbon and graphite products under ASTM D7972-14.<sup>8</sup> The constraints prescribed by the standard are discussed in PLN-3348. Figure 8 and Figure 9 show the relationship between the maximum load and maximum stress for the 33 specimens that were broken using the three-point bend method.



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

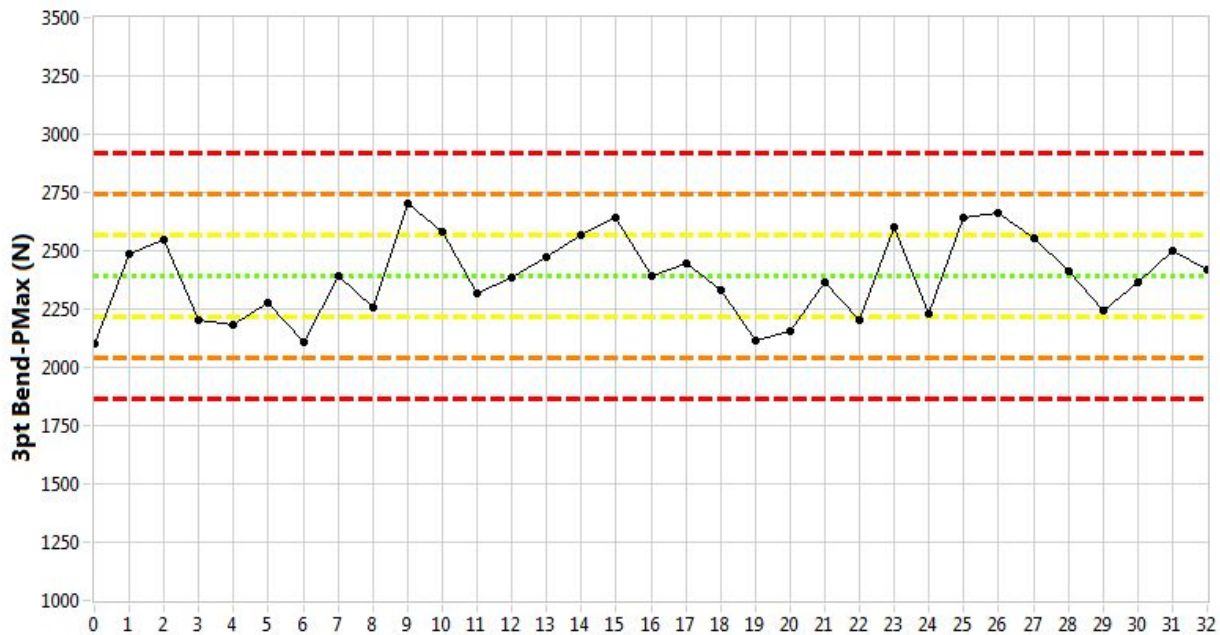


Figure 8. Maximum load (N), mean = 2389, standard deviation = 176.

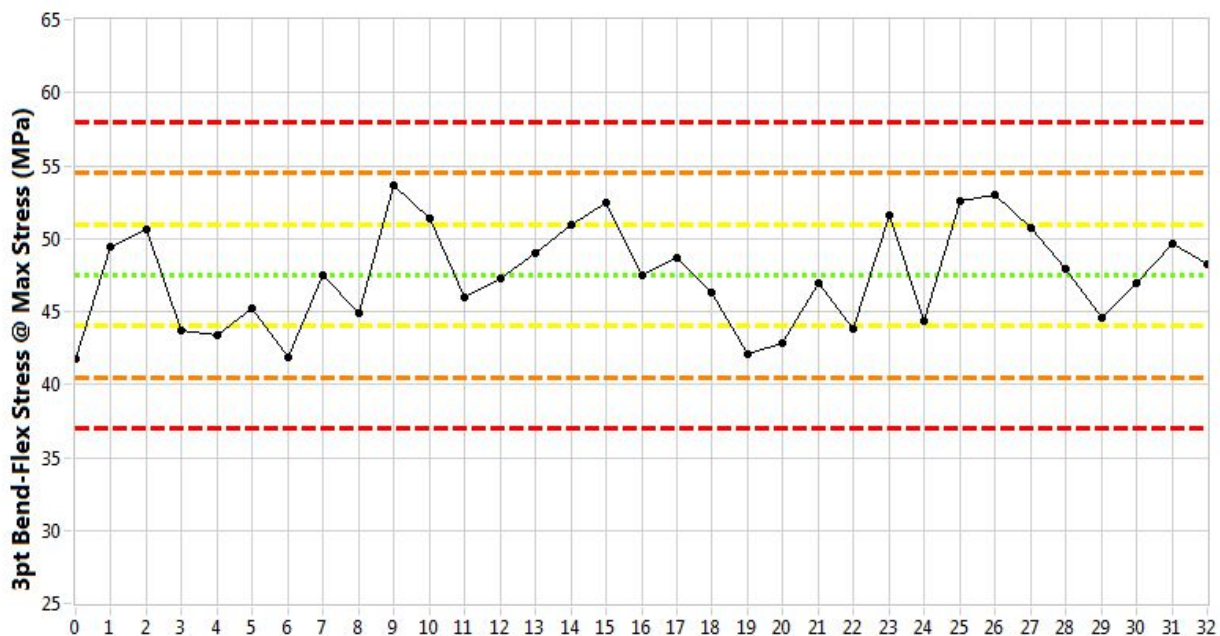


Figure 9. Maximum flexure stress (MPa), mean = 47.5, standard deviation = 3.5.



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

### Electrical Resistivity, Modulus, CTE

Electrical resistivity, Young's and shear modulus by sonic velocity, Young's modulus by sonic resonance, and coefficient of thermal expansion tests were performed on the 61 three-point bend specimens before they were broken. These tests were carried out via the appropriate ASTM standards.<sup>9,10,11,12,13</sup> Charts of those data are shown in Figure 10 through Figure 14.

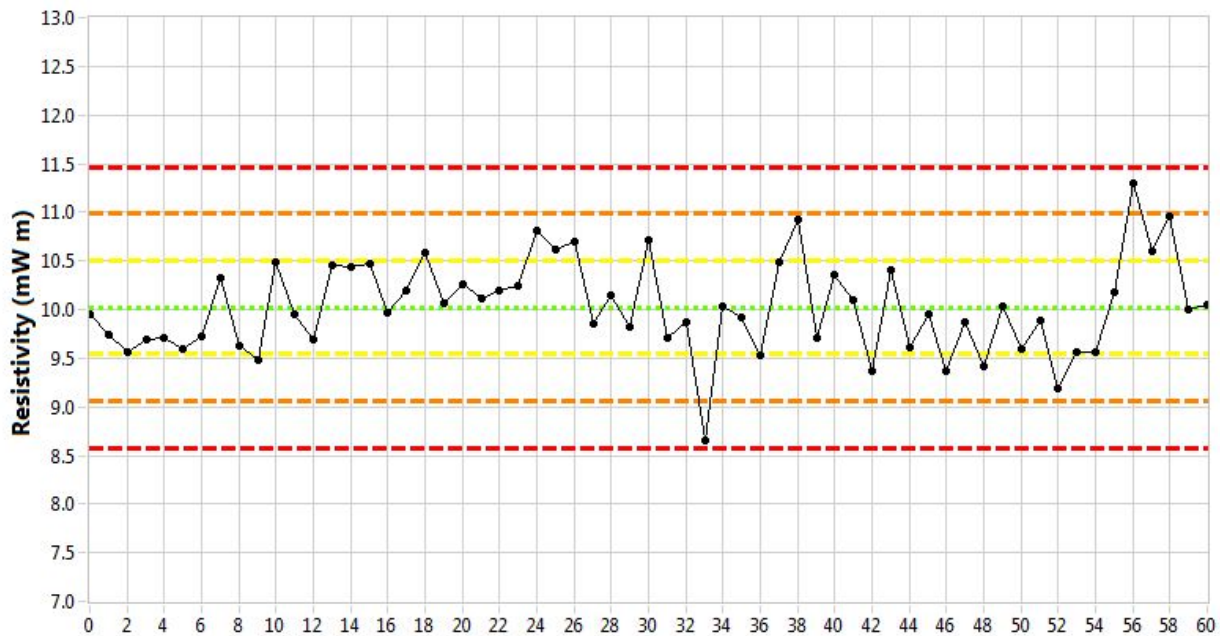


Figure 10. Electrical resistivity ( $\mu\Omega\text{-m}$ ), mean = 10, standard deviation = 0.5.



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

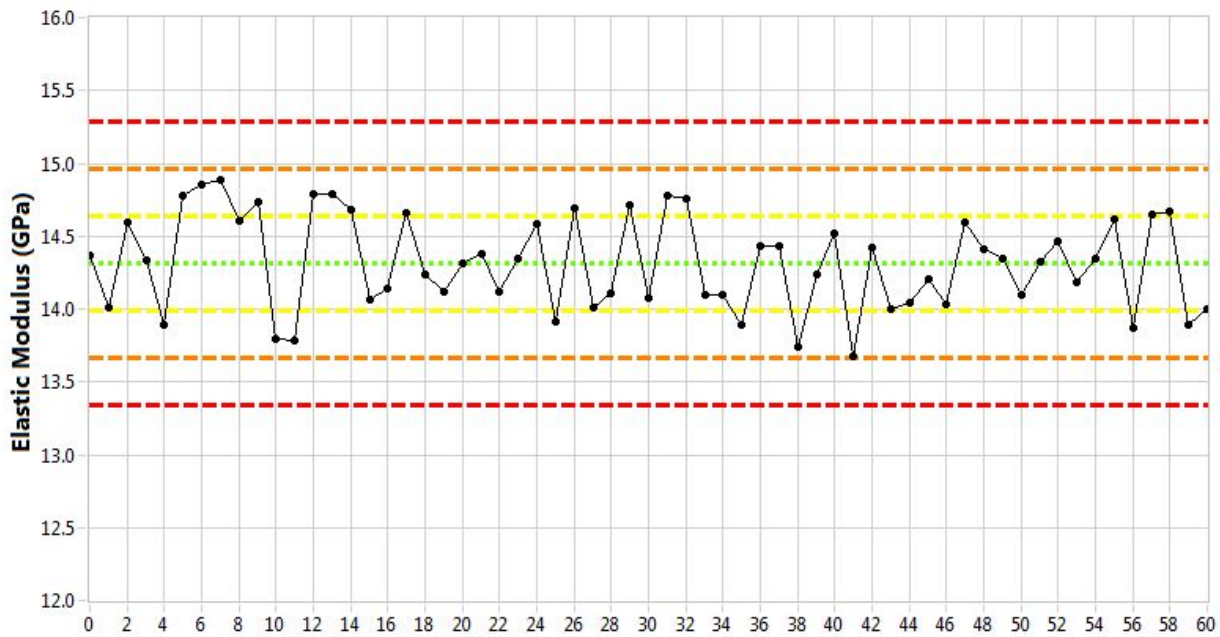


Figure 11. Young's modulus by sonic velocity method (GPa), mean = 14.3, standard deviation = 0.3.

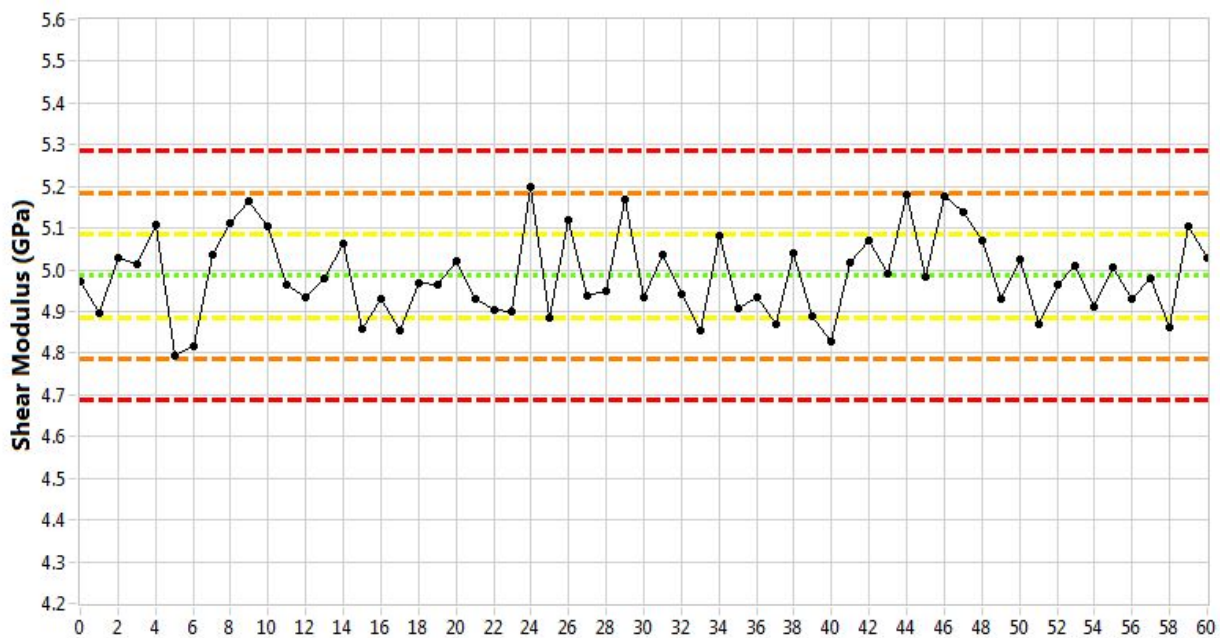


Figure 12. Shear modulus by sonic velocity method (GPa), mean = 4.99, standard deviation = 0.1.



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

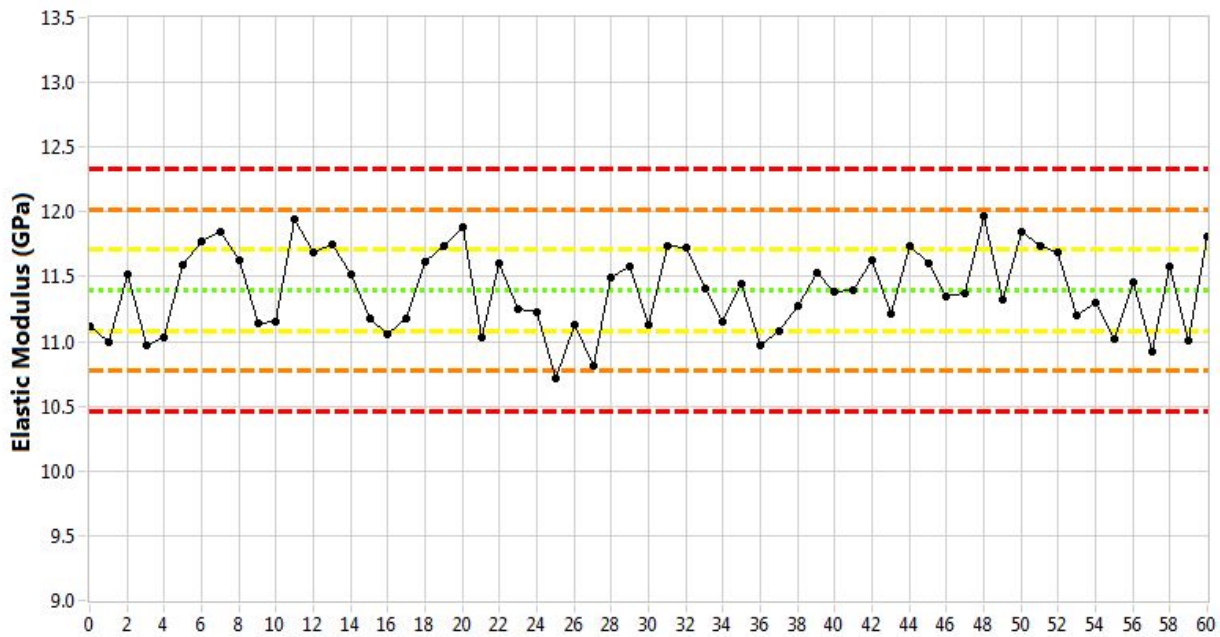


Figure 13. Young's modulus by sonic resonance method (GPa), mean = 11.4, standard deviation = 0.3.

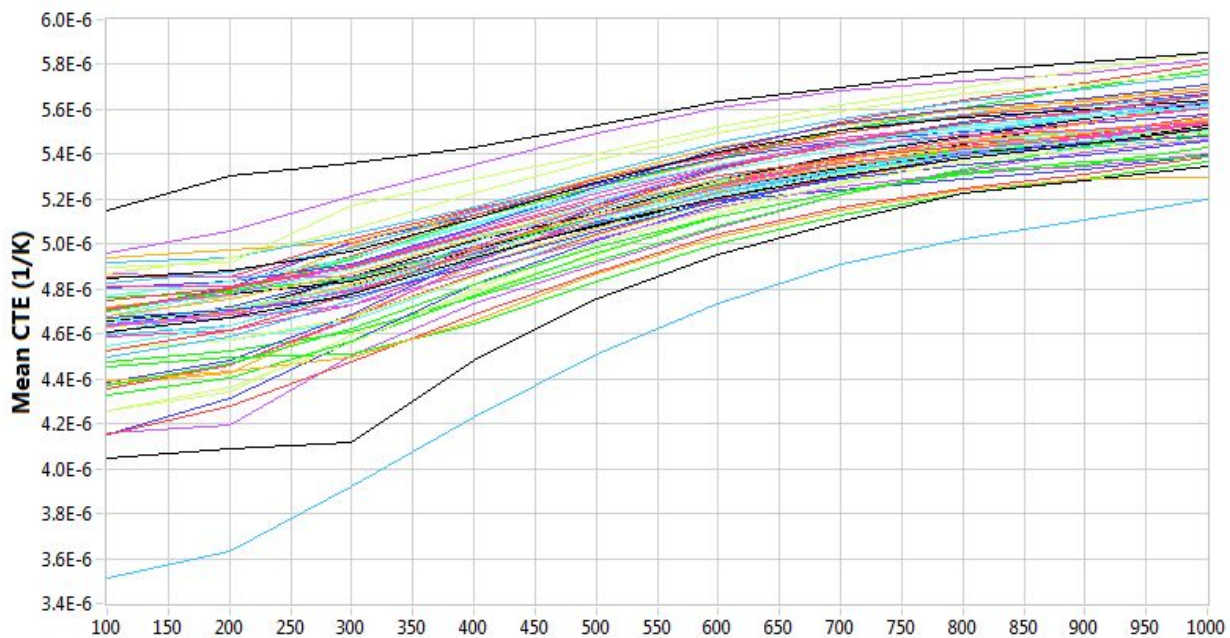


Figure 14. Mean coefficient of thermal expansion (1/K).



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

## Density Values

The relatively simple geometric shape of the compressive specimens provides an opportunity to collect density data (per ASTM C559-90<sup>14</sup>) 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 (Figure 15) show no anomalous values other than the expected material and measurement variation.

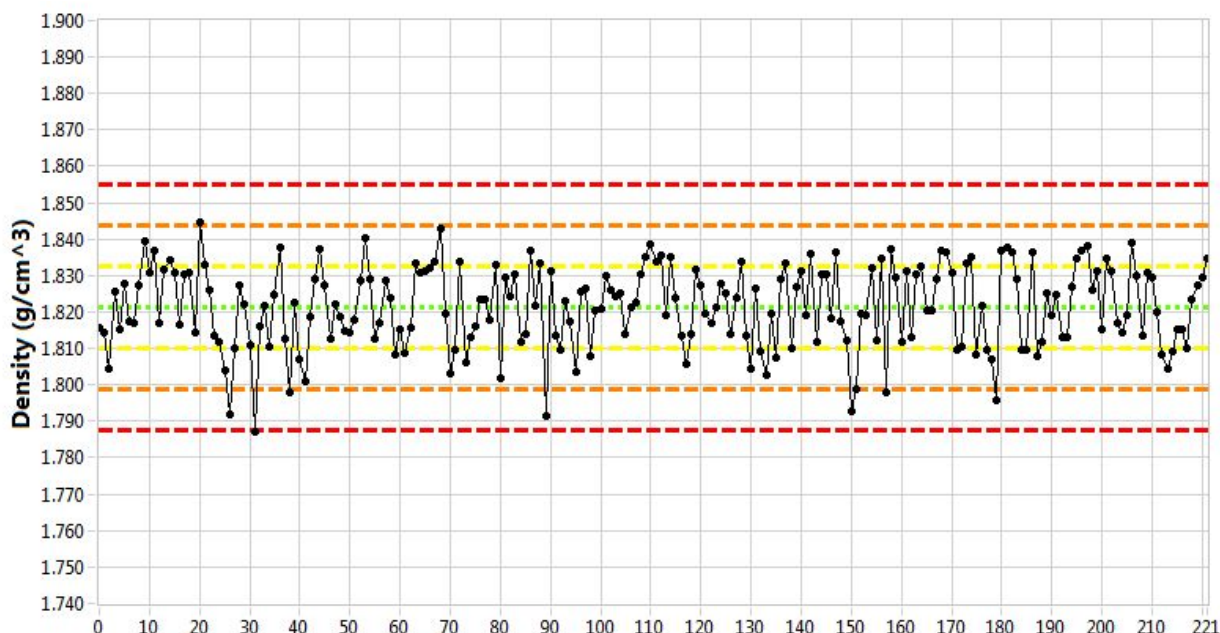


Figure 15. Density ( $\text{g}/\text{cm}^3$ ), mean = 1.8214, standard deviation = 0.0113.

## Flexural Specimen Database (NBG-17 830-3F)

### Flexural Testing

Flexural testing was performed per ASTM C651-91,<sup>15</sup> with clarifications to ambiguities in the standard identified in PLN-3467.<sup>3</sup> 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. Additional verification of test conditions can be accomplished through an analysis of the physical characteristics of the specimens. Figure 16, Figure 17, and Figure 18 show the measured width, thickness, and length for all flexural specimens tested. These plots show five points that lie more than 3 standard deviations below the sample mean. Regardless of this fact, these five specimens all met the size requirements imposed by the ASTM standard. Moreover, their corresponding flexure test results, such as maximum load, maximum stress, and deflection, all fell within a reasonable envelope. Because of this, the flexure data were deemed to be acceptable and are included in the data set. These outlying



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

dimensional measurements are highlighted in the flexural test results plots in Figure 16, Figure 17, and Figure 18.

Figure 19 and Figure 20 also show the relationship between flexural load and recorded flexural stress for the 224 specimens tested in flexure from NBG-17 Billet 830-3. Further comparisons and verification can be made with measured deflection (Figure 21), which will reflect an additional correlation with stress values through material elastic constants.

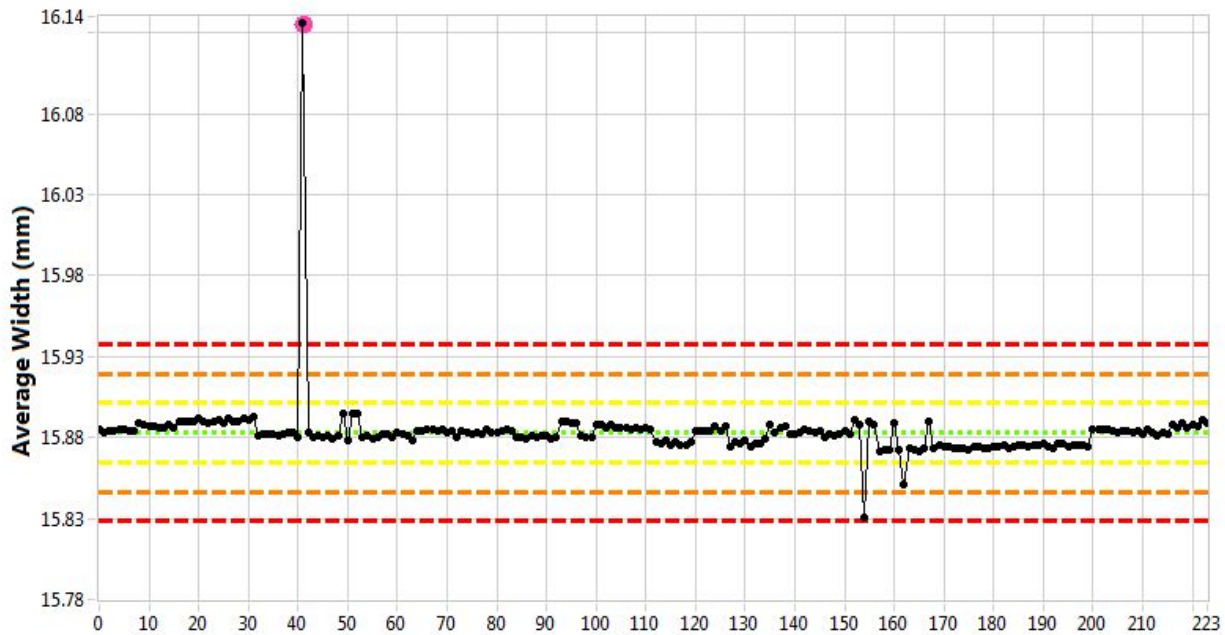


Figure 16. Average width (mm), mean = 15.883, standard deviation = 0.0183.



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

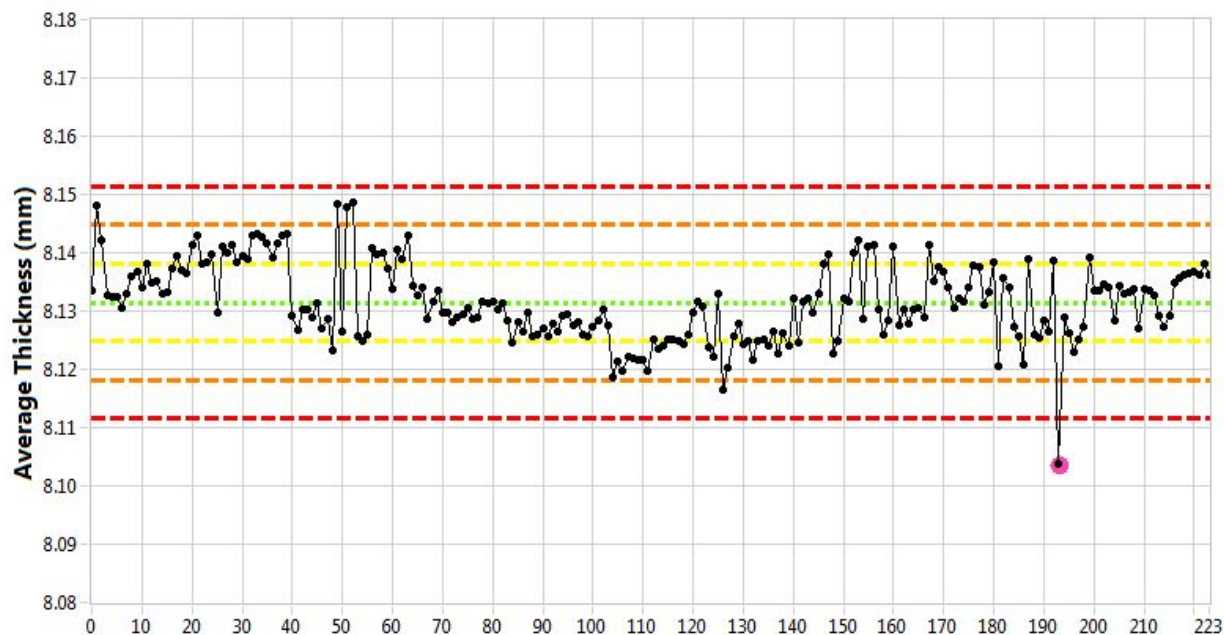


Figure 17. Average thickness (mm), mean = 8.131, standard deviation = 0.0067.

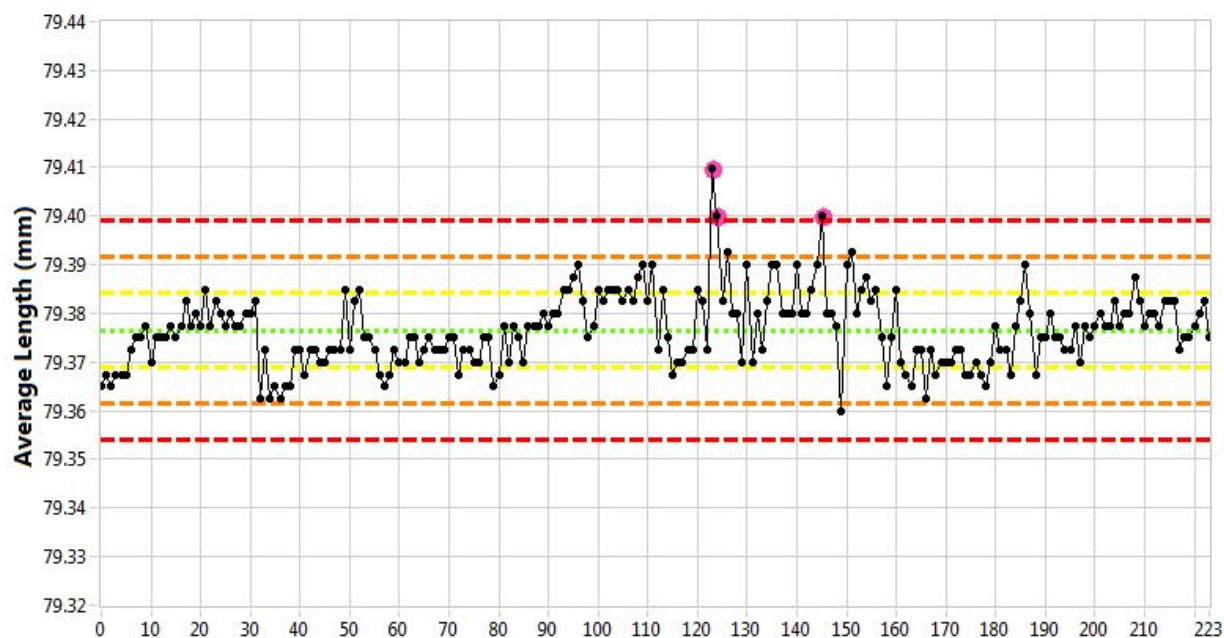


Figure 18. Average length (mm), mean = 79.377, standard deviation = 0.0075.



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

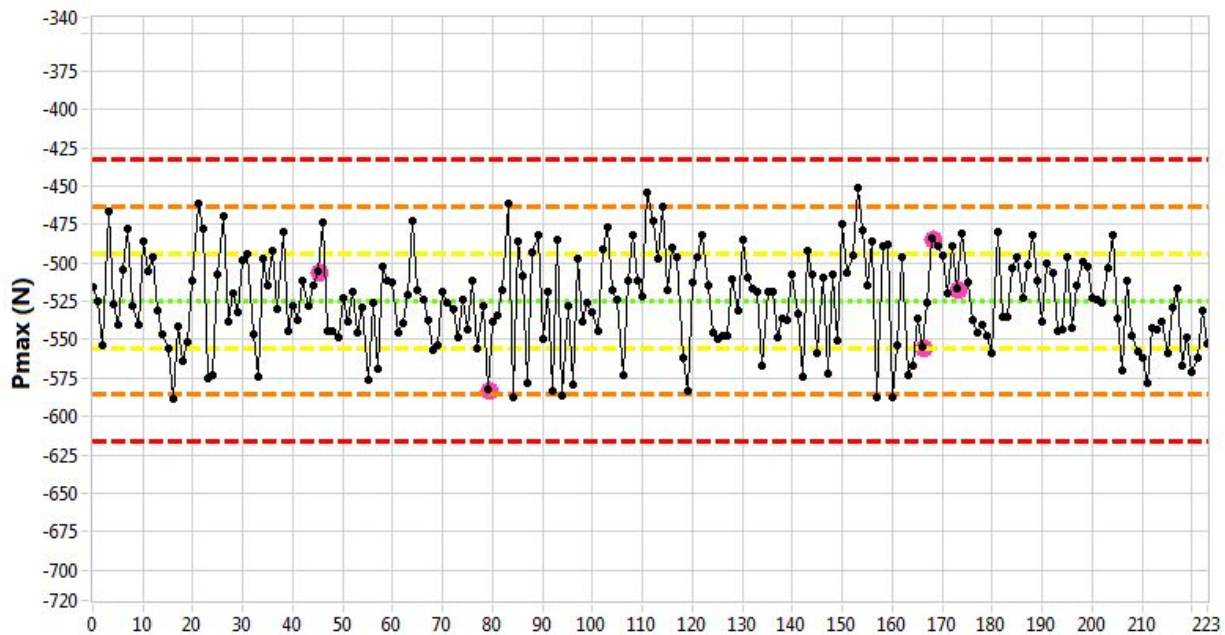


Figure 19. Maximum load (N), mean = -524.6, standard deviation = 30.6.

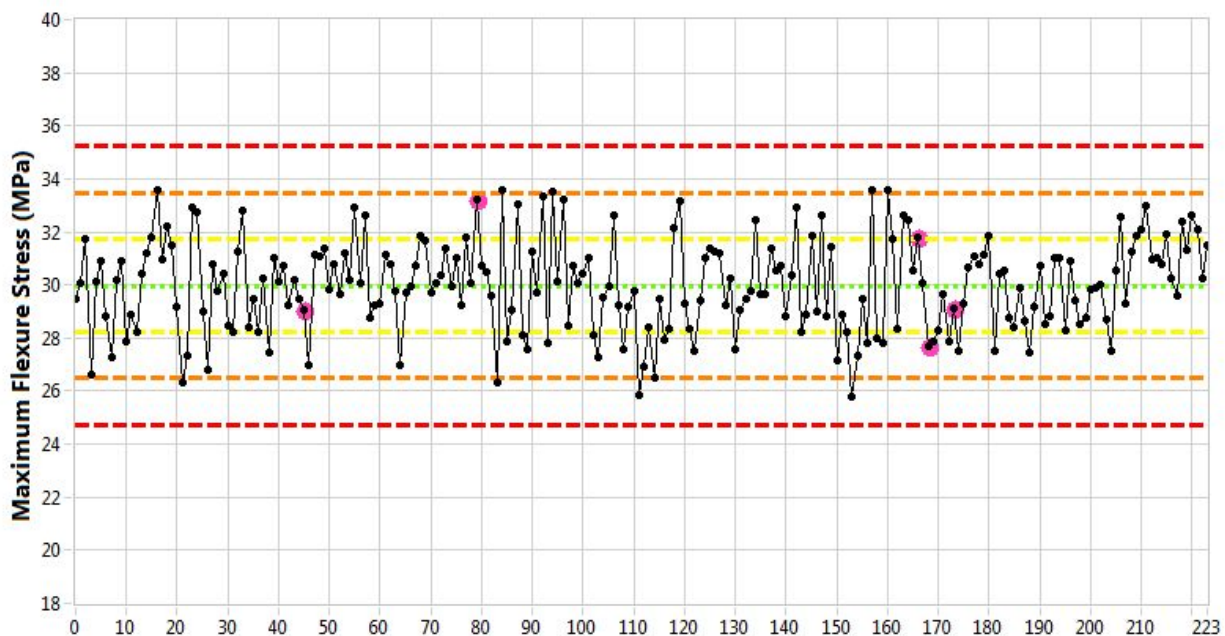


Figure 20. Maximum flexure stress (MPa), mean = 30.0, standard deviation = 1.7.



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

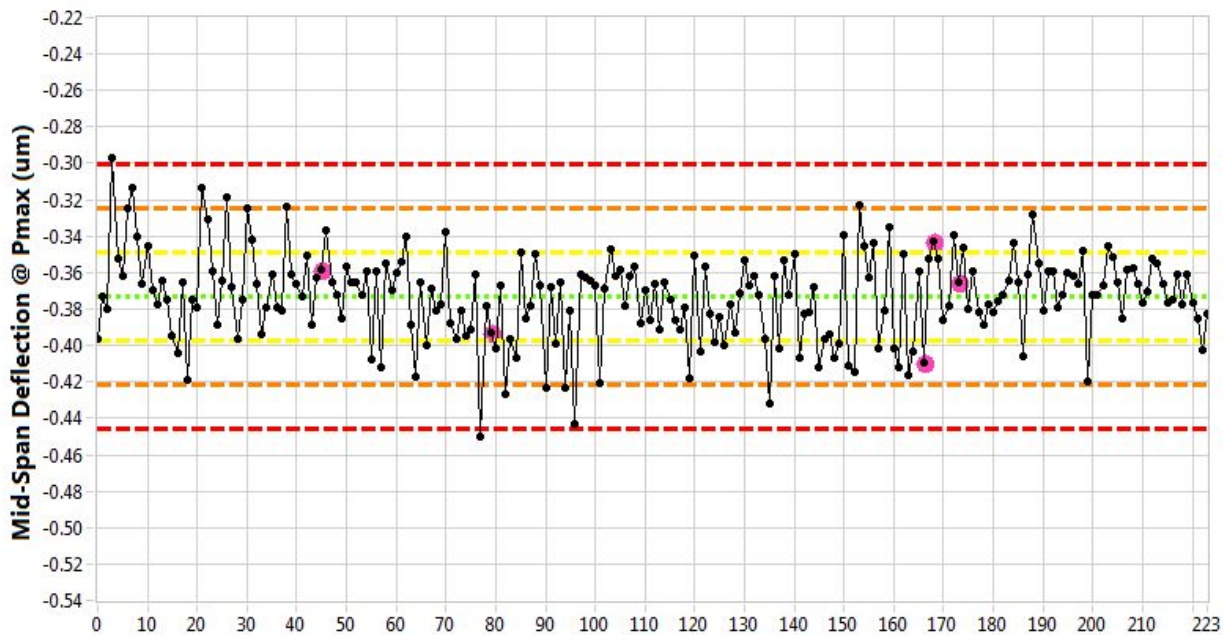


Figure 21. Mid-span deflection at maximum load ( $\mu\text{m}$ ), mean = -0.3733, standard deviation = 0.0242.

## Density Values

Similar to the compression specimens, the flexural specimens' geometry facilitated an opportunity to make density measurements. Figure 22 shows density from the flexural specimens. All flexural specimens' data and associated deviations compare well with the compression specimens' density data.



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

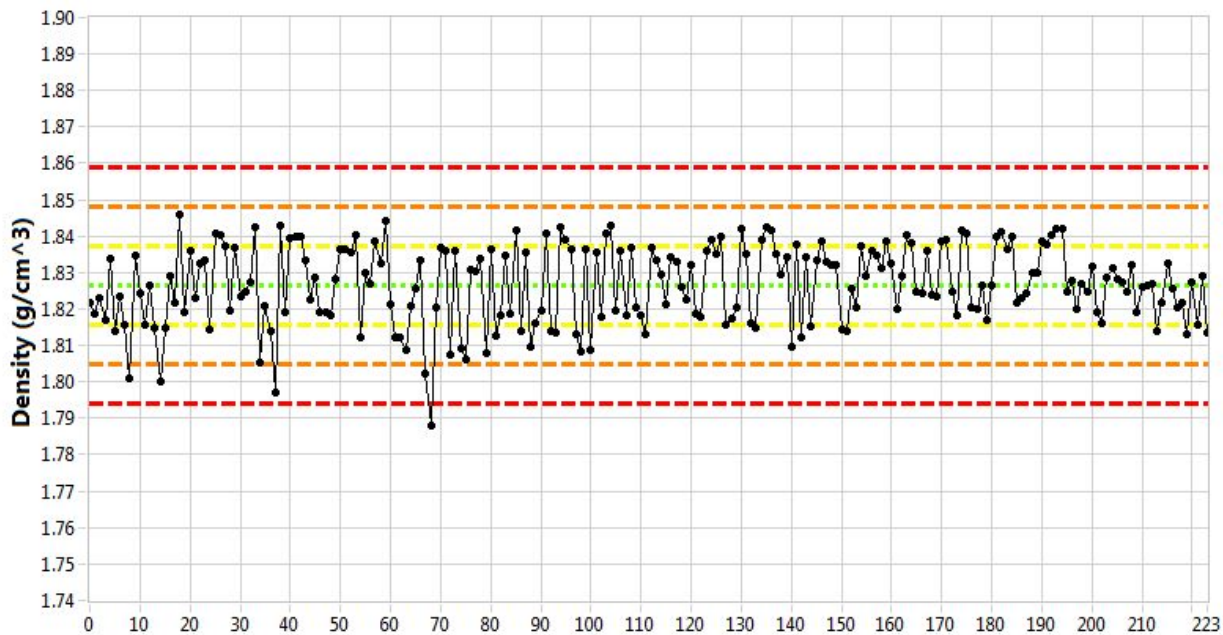


Figure 22. Density (g/cm<sup>3</sup>), mean = 1.8266, standard deviation = 0.0108.

## 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<sup>10</sup>). Values for fundamental frequency-based moduli, both in flexural and torsional modes (shown in Figure 23 and Figure 24), are calculated from the equations provided in ASTM C1259-08.<sup>11</sup> These data all fell within  $\pm 3$  standard deviations from their respective means.



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

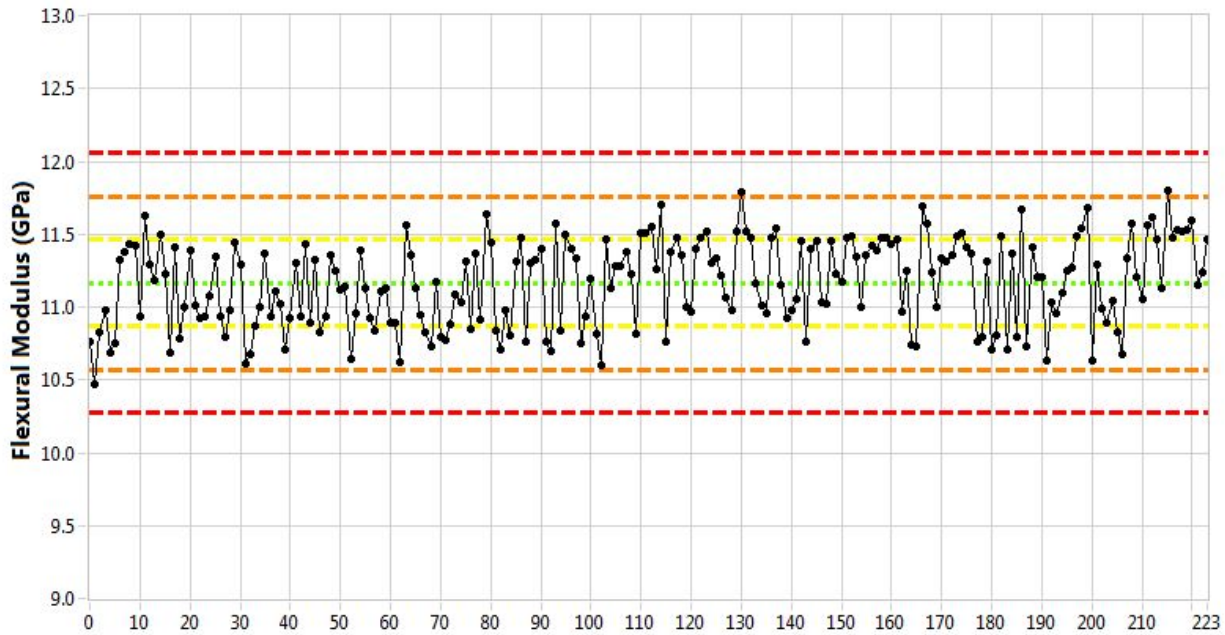


Figure 23. Flexural vibration mode modulus (GPa), mean = 11.2, standard deviation = 0.3.

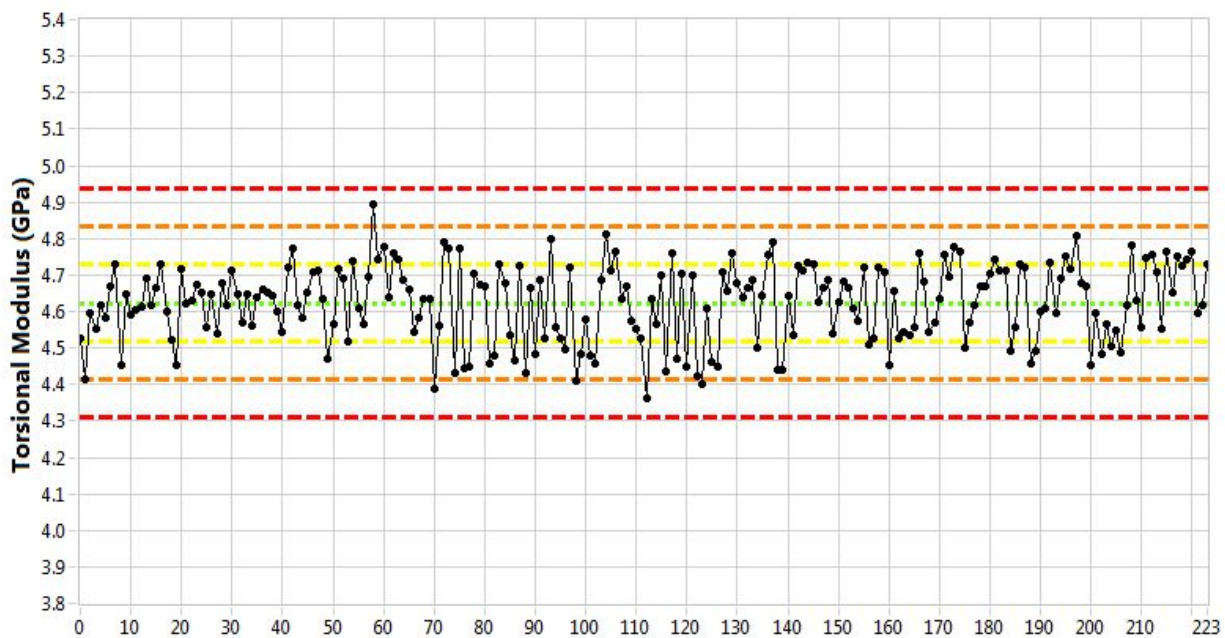


Figure 24. Torsional vibration mode shear modulus (GPa), mean = 4.6, standard deviation = 0.1.



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

## Tensile Specimen Database (NBG-17 830-3T)

### Tensile Testing

Tensile testing was performed per ASTM C749-08.<sup>16</sup> 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. The custom measurement software used to capture tensile gauge diameters is programed to flag any measurement that deviates from the ASTM standard. Figure 25 shows that the 197 tensile specimens fell within the tolerances defined by the ASTM standard.

Figure 26 and Figure 27 show the relationship between tensile load and recorded tensile strength for all of the specimens tested in uniaxial tension from the NBG-17 Billet 830-3. Figure 28 shows the recorded tensile stress for the tested specimens. Further comparisons and verification can be made with extensometer-based measured strain (shown in Figure 29), which will reflect an additional correlation with stress values through material elastic constants. Comparing the extreme values again shows this relationship to be valid.

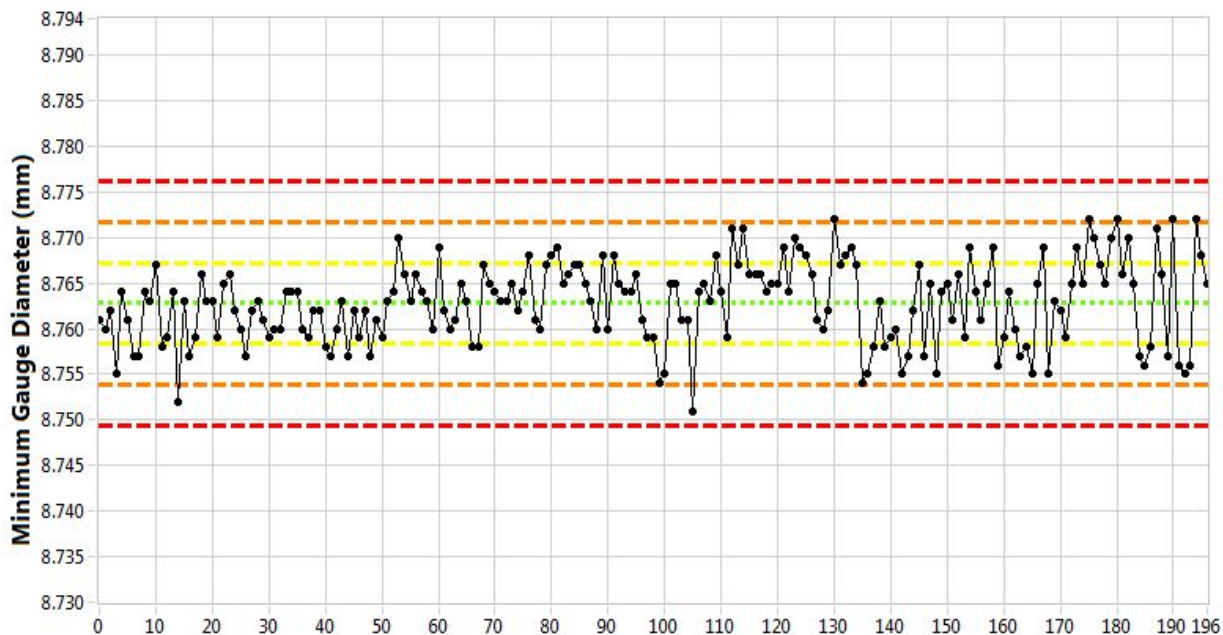


Figure 25. Minimum gauge diameter (mm), mean = 8.763, standard deviation = 0.0045.



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

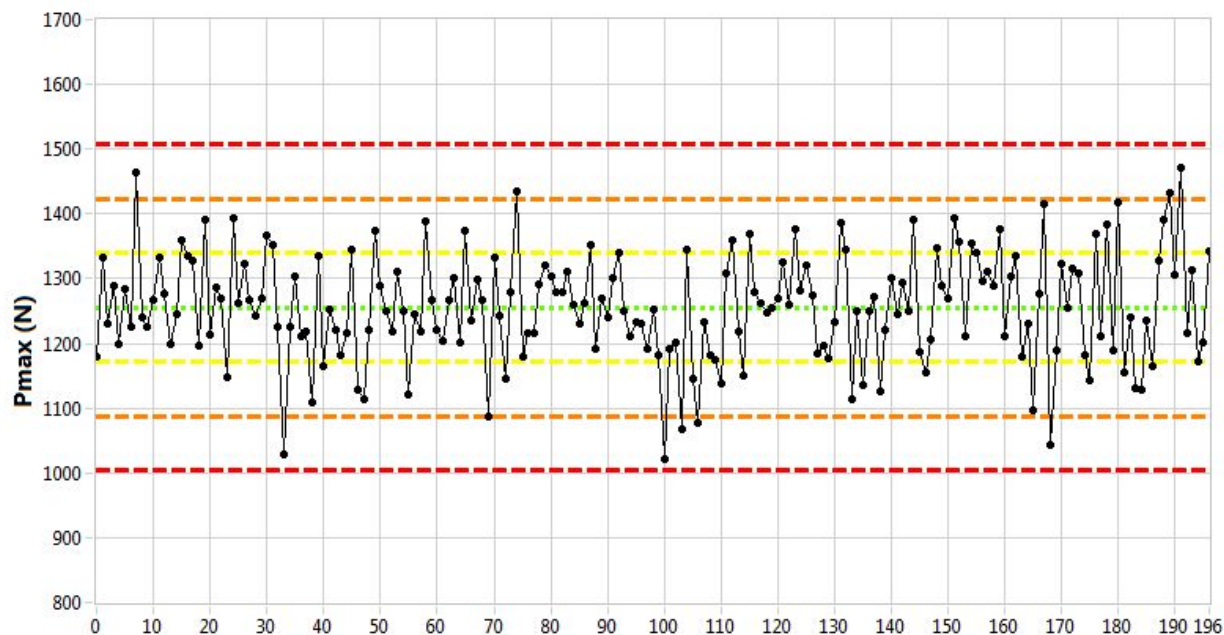


Figure 26. Maximum load (N), mean = 1256, standard deviation = 84.

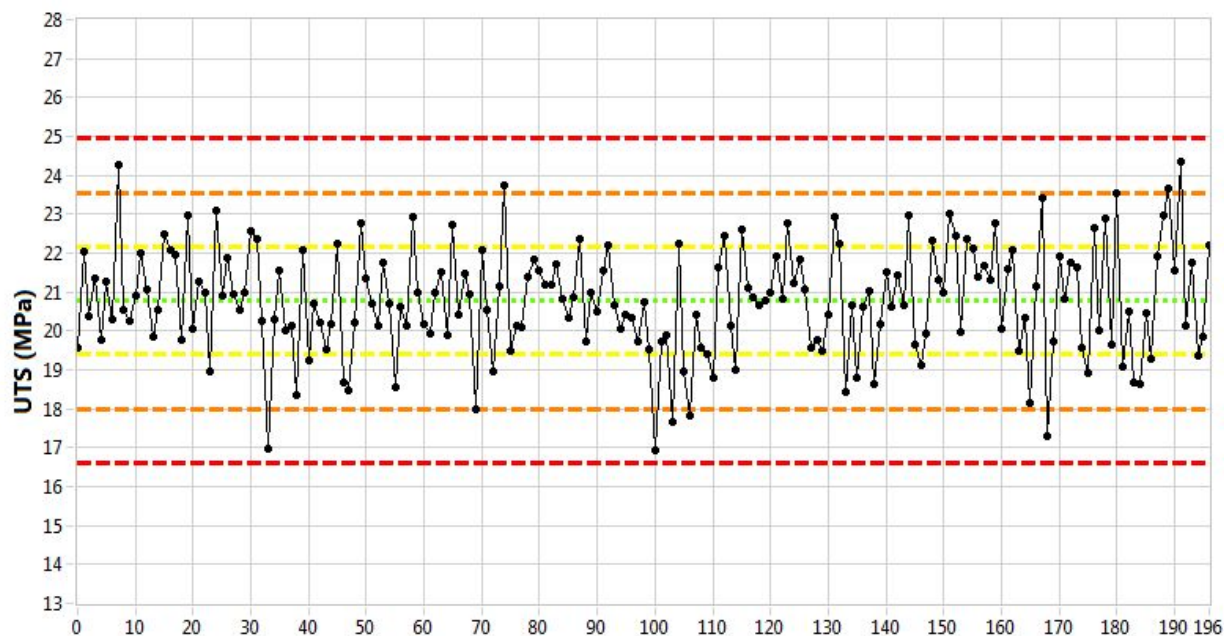


Figure 27. Ultimate tensile strength (MPa), mean = 20.8, standard deviation = 1.4.



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

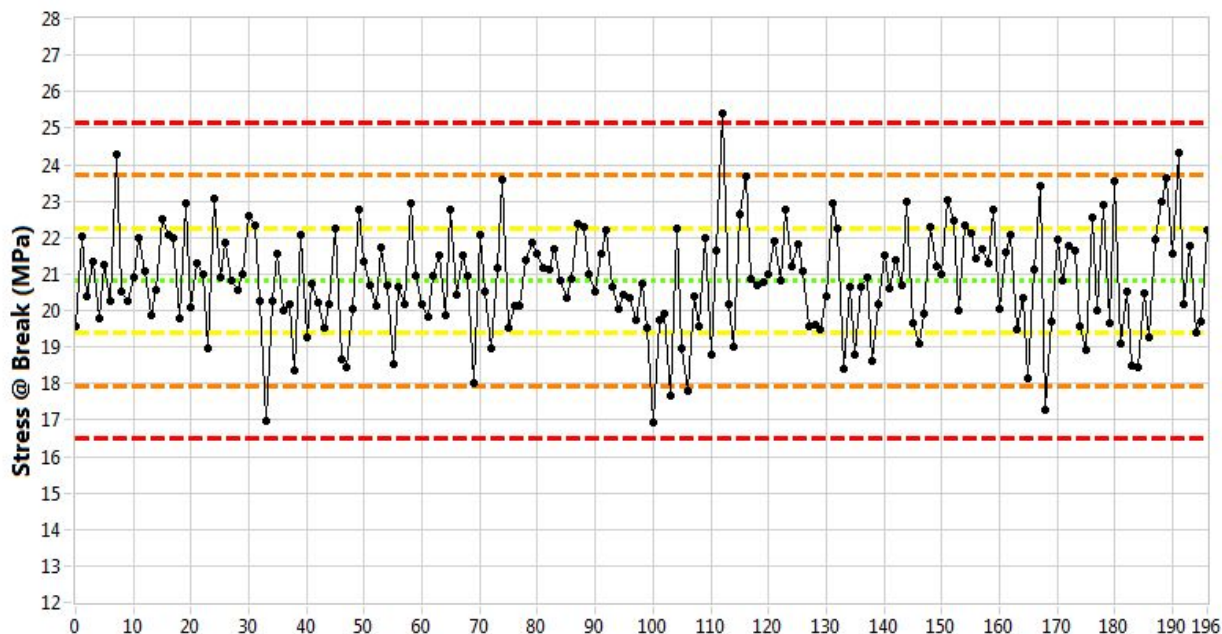


Figure 28. Stress at break (MPa), mean = 20.8, standard deviation = 1.4.

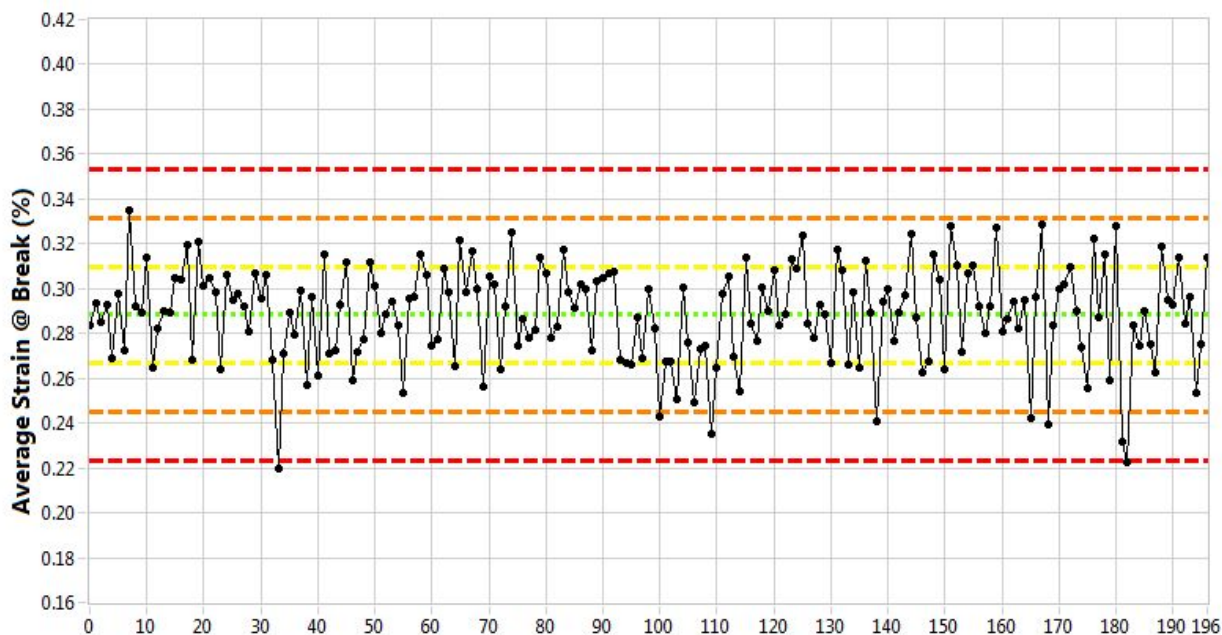


Figure 29. Average strain at break (%), mean = 0.29, standard deviation = 0.022.



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

## 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 30) to the same dimensions as AGC piggyback specimens. A random cross-section of tensile specimens was re-machined in order to repeat tests on AGC-sized specimens (i.e., diffusivity and split disc testing). Using actual test specimens for re-machining allows continued use of the specimen identification and tracking code system, because specimens are machined from tracked locations and can reuse the identification code.

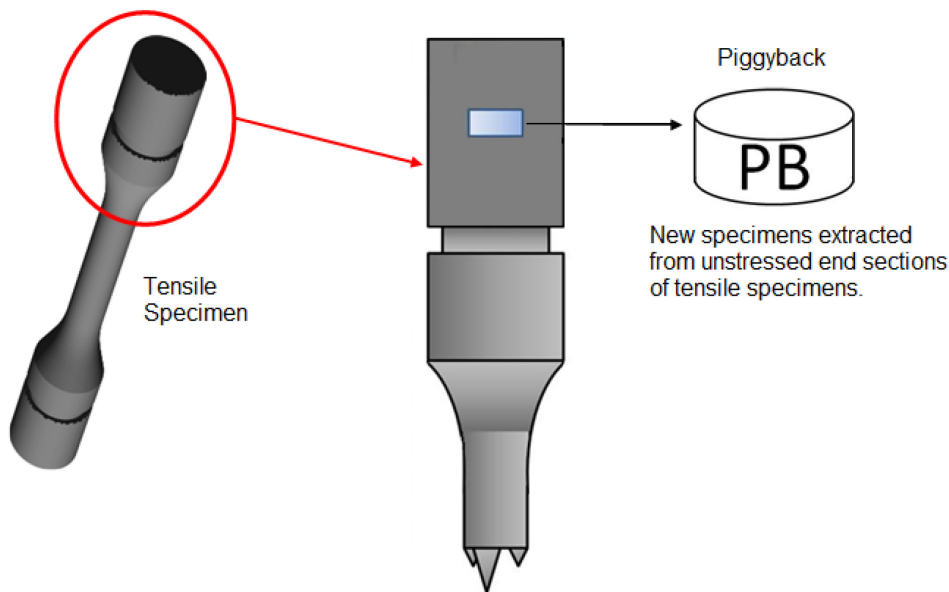


Figure 30. Unstressed specimen remnants from tensile specimens are re-machined into AGC-sized piggyback specimens.

## RE-MACHINED SPLIT DISC TESTING

Split disc tensile strength testing was performed in accordance with PLN-3348, Revision 4, Subsection 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. Figure 31 and Figure 32 show strength and load data from the split disc testing. The mean strength value calculated from the split disc testing was approximately 5% lower than that from traditional tensile testing (Figure 27) and had a slightly lower standard deviation.



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

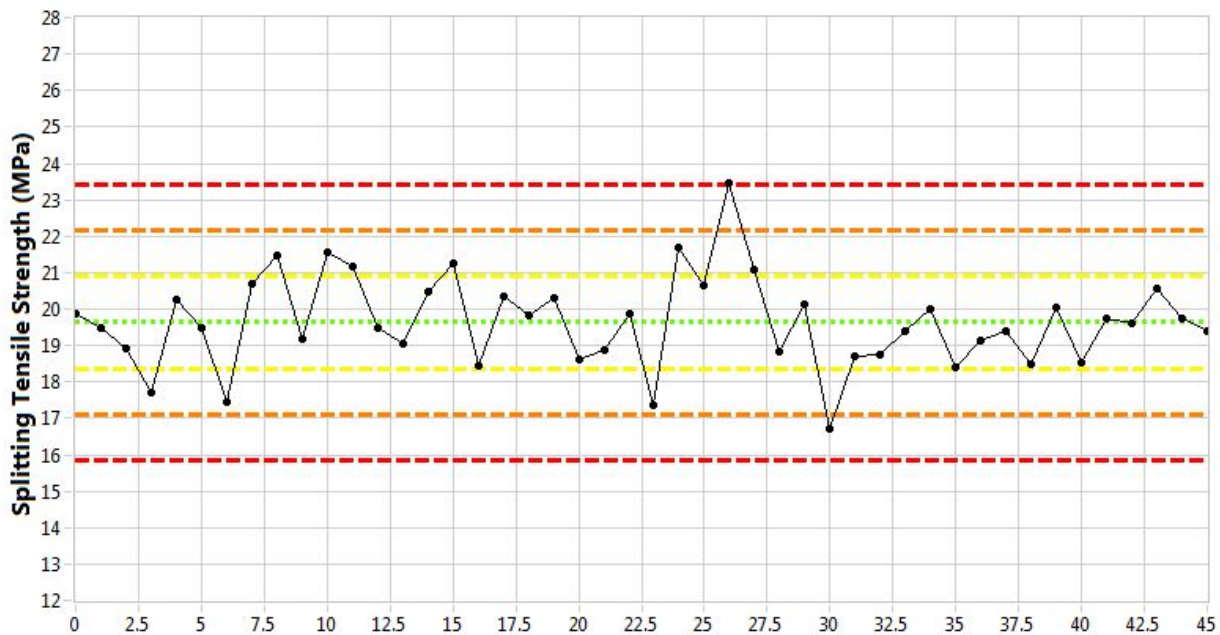


Figure 31. Disc splitting tensile strength (MPa), mean = 19.6, standard deviation = 1.3.

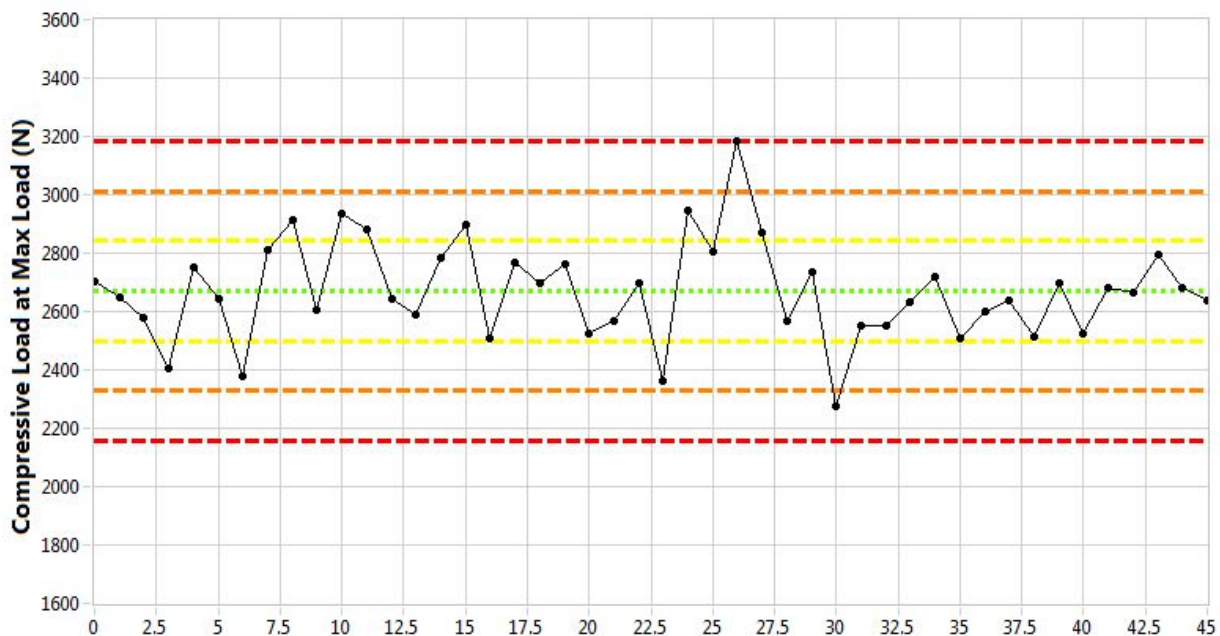


Figure 32. Disc splitting compressive load at max load (N), mean = 2670, standard deviation = 171.



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

## RE-MACHINED SPECIMEN DIFFUSIVITY

Thermal diffusivity values are collected from the re-machined tensile specimens per ASTM E1461-07.<sup>17</sup> 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 33.

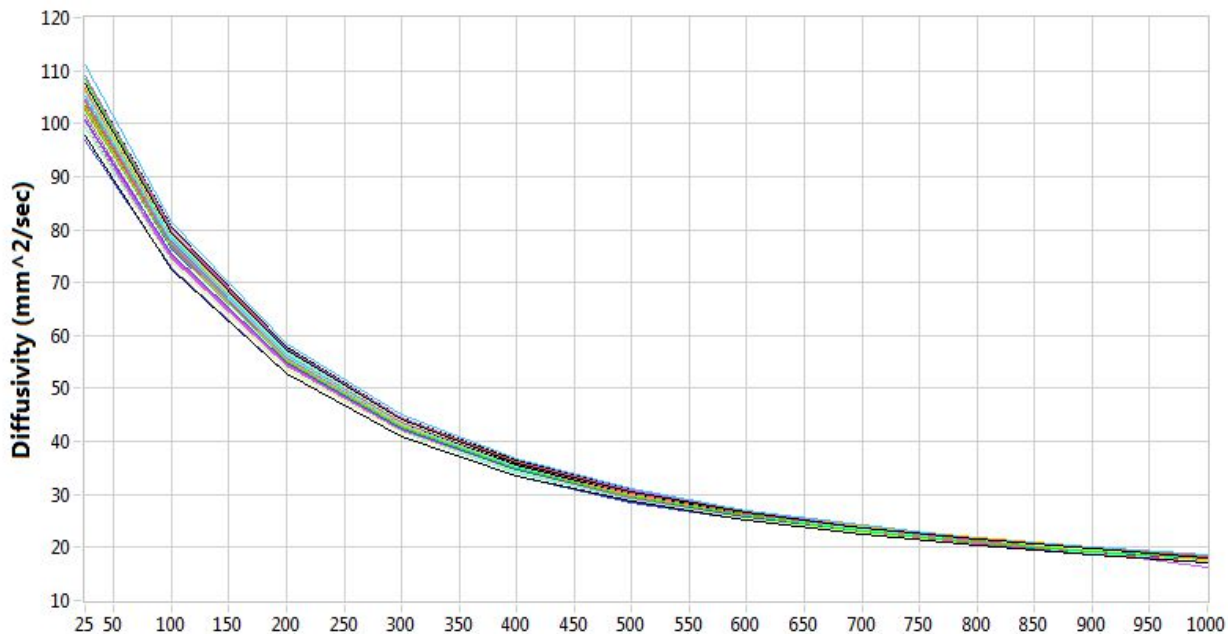


Figure 33. Re-machined specimen diffusivity.

## SUMMARY

Comprehensive data sets for NBG-17 Billet 830-3 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 if 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.

## REFERENCES

1. PLN-2497, 2010, "Graphite Technology Development Plan," Rev. 1, October 4, 2010.



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727      Rev. No.: 0      Project No.: 32138      Date: 07/31/2017

2. PLN-3348, 2011, "Graphite Mechanical Testing," Rev. 4, March 16, 2017.
3. PLN-3467, 2011, "Baseline Graphite Characterization Plan: Electromechanical Testing," Rev. 2, June 22, 2015.
4. PLN-3267, 2010, "AGC-2 Characterization Plan," Rev. 0, March 19, 2010.
5. Mark Carroll, Joe Lord, and David Rohrbaugh, 2010, *Baseline Graphite Characterization: First Billet*, INL/EXT-10-19910, September 2010.
6. LWP-20000-01, "Conduct of Research Plan," Rev. 0, August 2015
7. ASTM C695-15, "Standard Test Method for Compressive Strength of Carbon and Graphite," ASTM International, 2015.
8. ASTM D7972-14, "Standard Test Method for Flexural Strength of Manufactured Carbon and Graphite Articles Using Three-Point Loading at Room Temperature," ASTM International, 2014.
9. ASTM C769-09, "Standard Test Method for Sonic Velocity in Manufactured Carbon and Graphite Material for Use in Obtaining an Approximate Young's Modulus," ASTM International, 2009.
10. ASTM 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.
11. ASTM 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.
12. ASTM C611-05, "Standard Test Method for Electrical Resistivity of Manufactured Carbon and Graphite Articles at Room Temperature," ASTM International, 2005.
13. ASTM E228-06, "Standard Test Method for Linear Thermal Expansion of Solid Materials with a Push Rod Dilatometer," ASTM International, 2006.
14. ASTM C559-90 (Reapproved 2005), "Standard Test Method for Bulk Density by Physical Measurements of Manufactured Carbon and Graphite Articles," ASTM International, 2005.
15. ASTM 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.
16. ASTM C749-08, "Standard Test Method for Tensile Stress-Strain of Carbon and Graphite," ASTM International, 2008.
17. ASTM E1461-07, "Standard Test Method for Thermal Diffusivity by the Flash Method," ASTM International, 2007.



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

## Appendix A

### Additional Compression Specimen Database Plots (NGB-17 830-3)

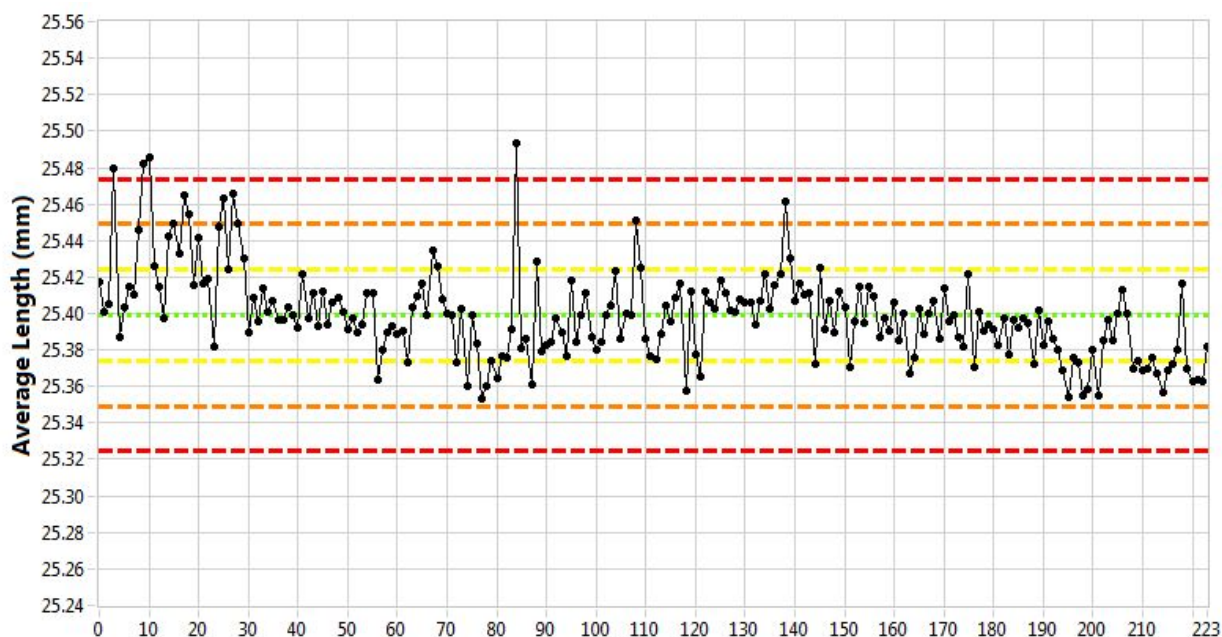


Figure A-1. Average length (mm), mean = 25.3992, standard deviation = 0.0249.

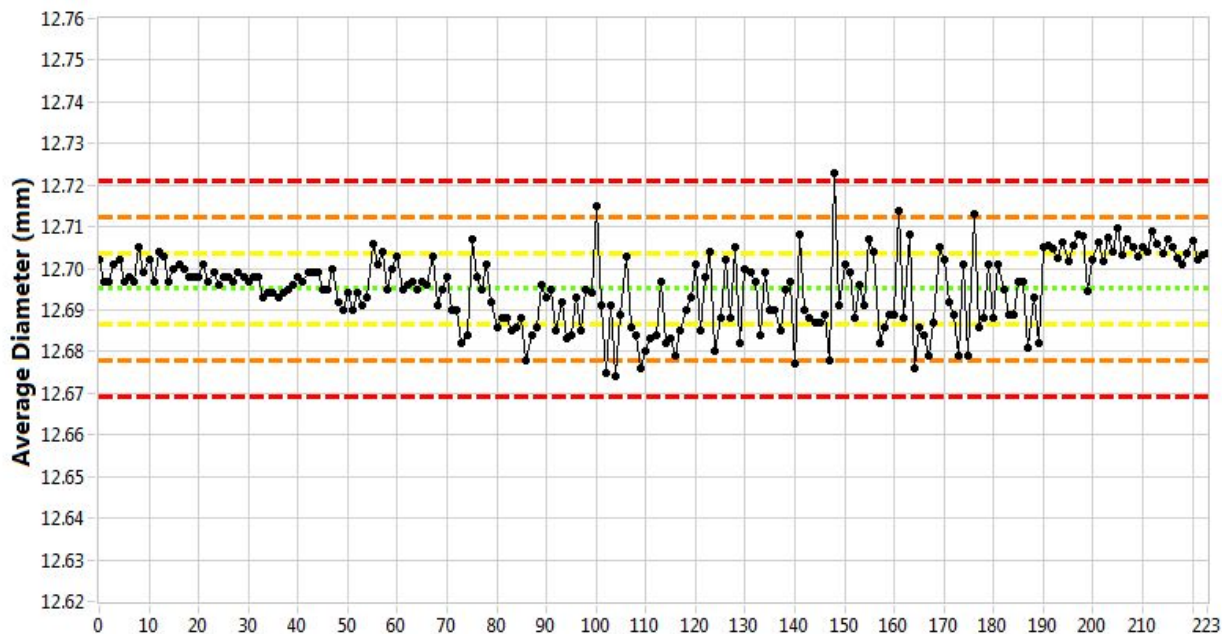


Figure A-2. Average diameter (mm), mean = 12.6951, standard deviation = 0.0086.



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

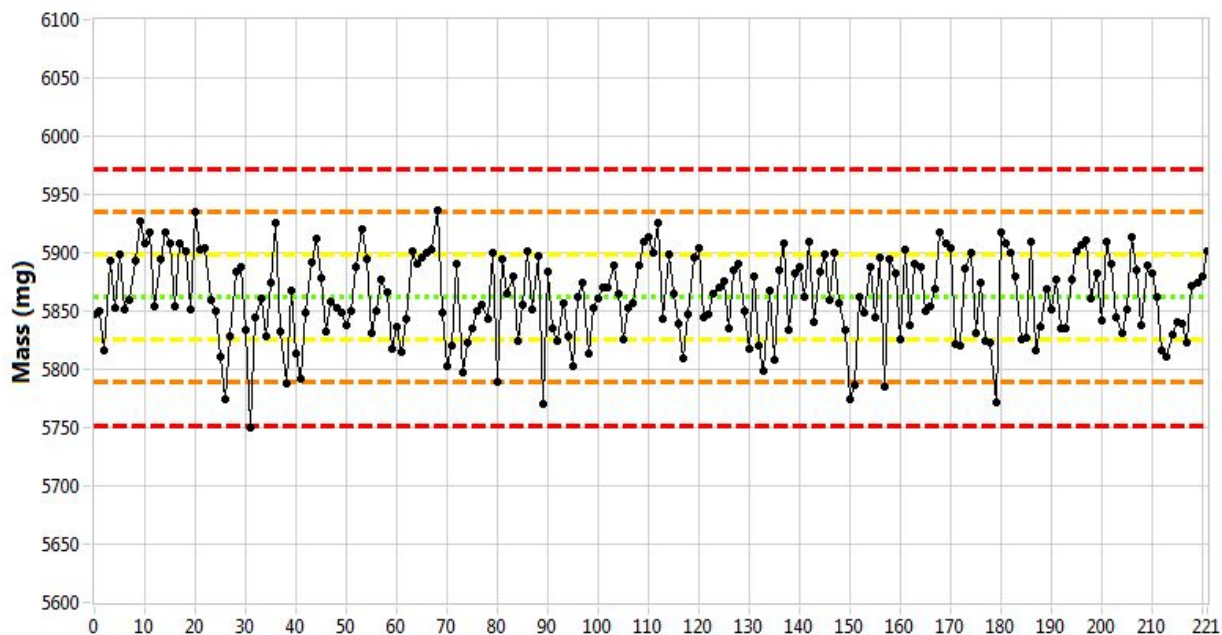


Figure A-3. Mass (mg), mean = 5,861.7, standard deviation = 36.6.

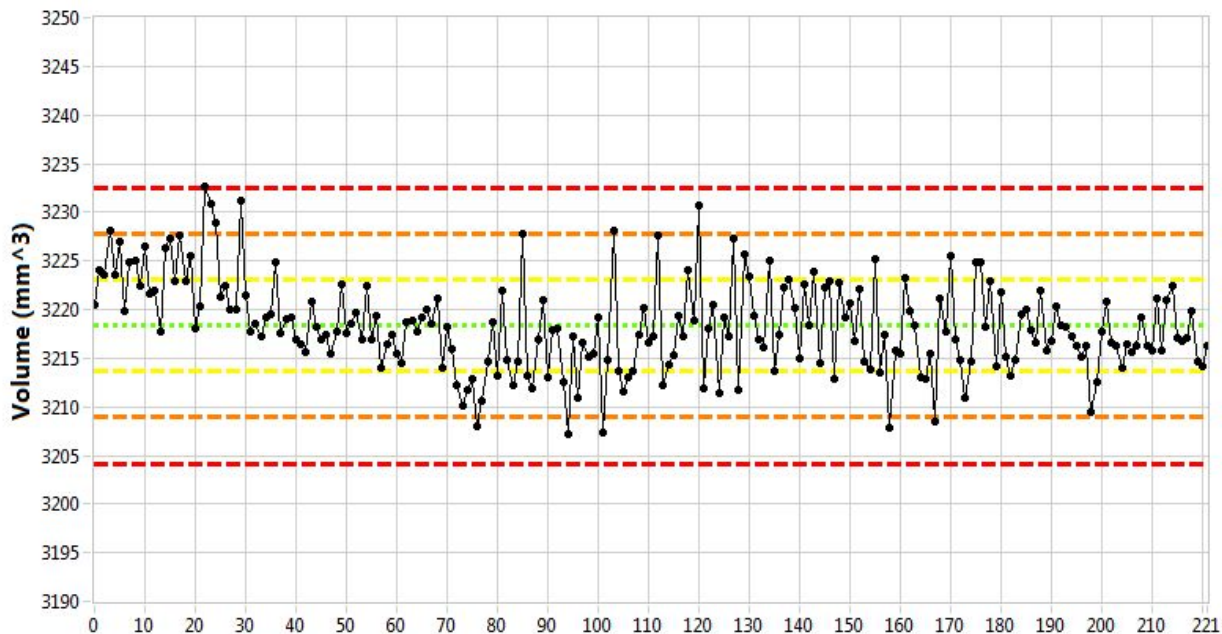


Figure A-4. Volume (mm<sup>3</sup>), mean = 3,218.3, standard deviation = 4.7.



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

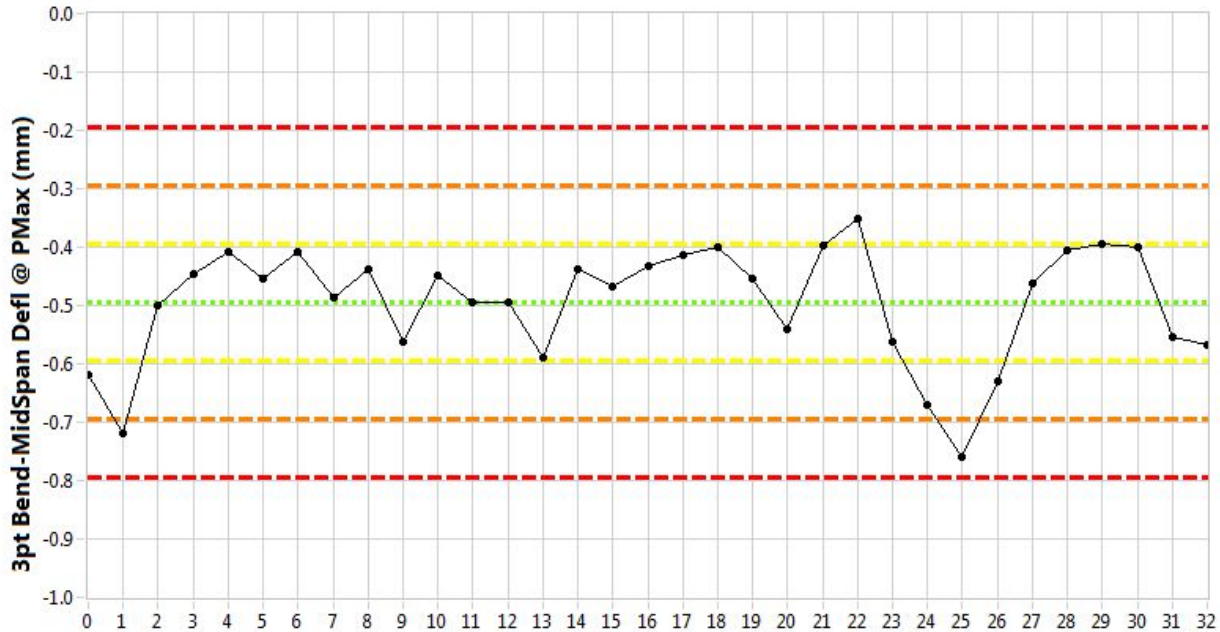


Figure A-5. Mid-span deflection at maximum load (um), mean = -0.4959, standard deviation = 0.1.

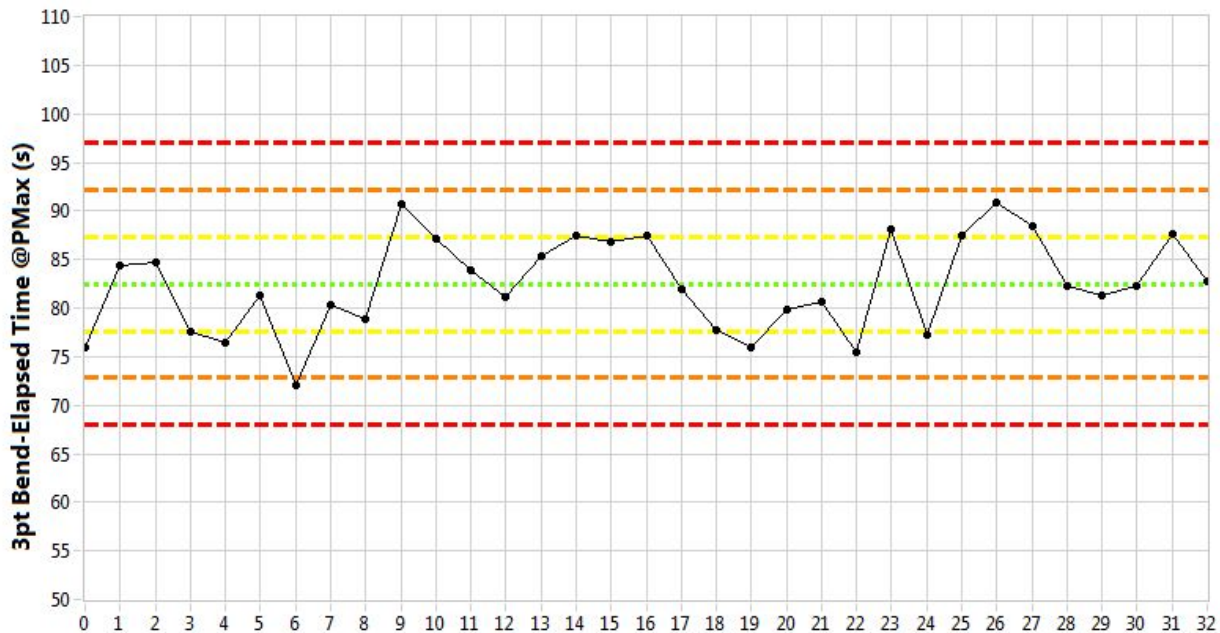


Figure A-6. Elapsed time at maximum load (sec), mean = 82.5, standard deviation = 4.8.



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

## Appendix B

### Additional Flexural Specimen Database Plots (NBG-17 830-3)

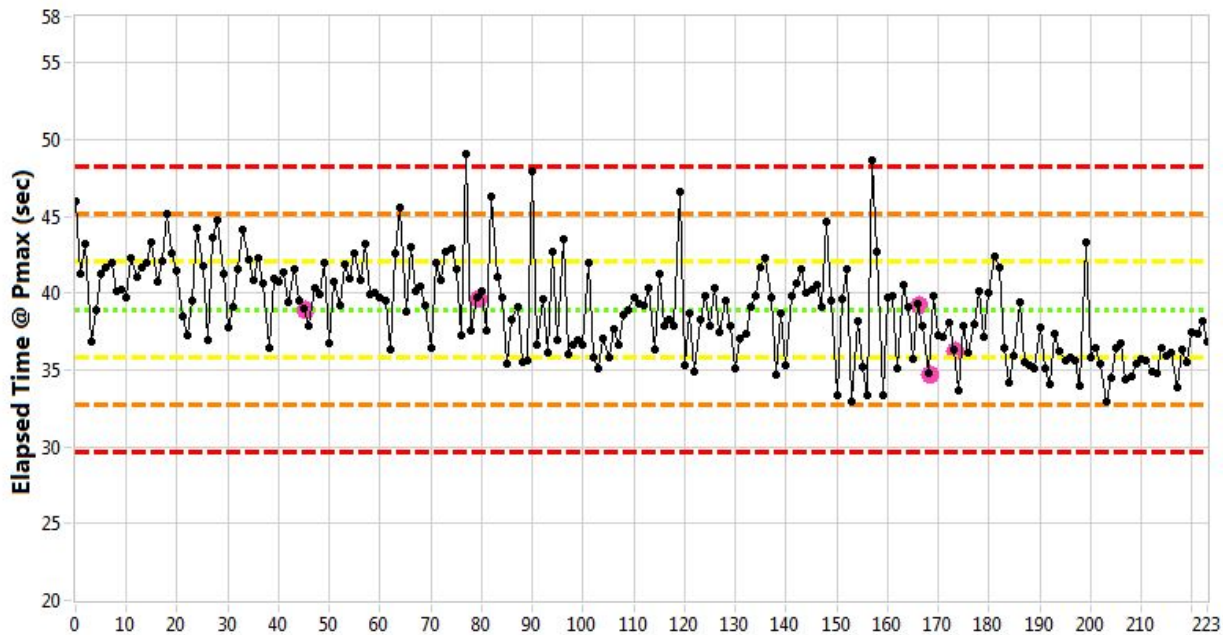


Figure B-1. Elapsed time at maximum load (sec), mean = 38.9, standard deviation = 3.1.

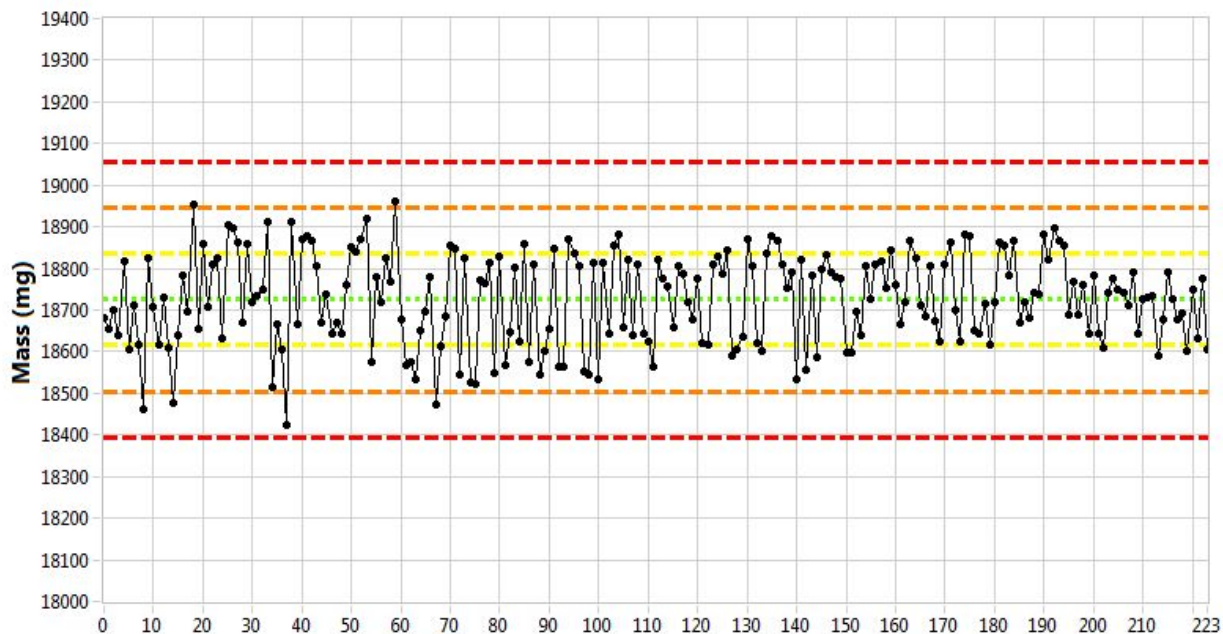


Figure B-2. Mass (mg), mean = 18725.3, standard deviation = 110.1.



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017



Figure B-3. Volume (mm<sup>3</sup>), mean = 10251.7, standard deviation = 14.9.



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

## Appendix C

### Additional Tensile Specimen Database Plots (NBG-17 830-3)

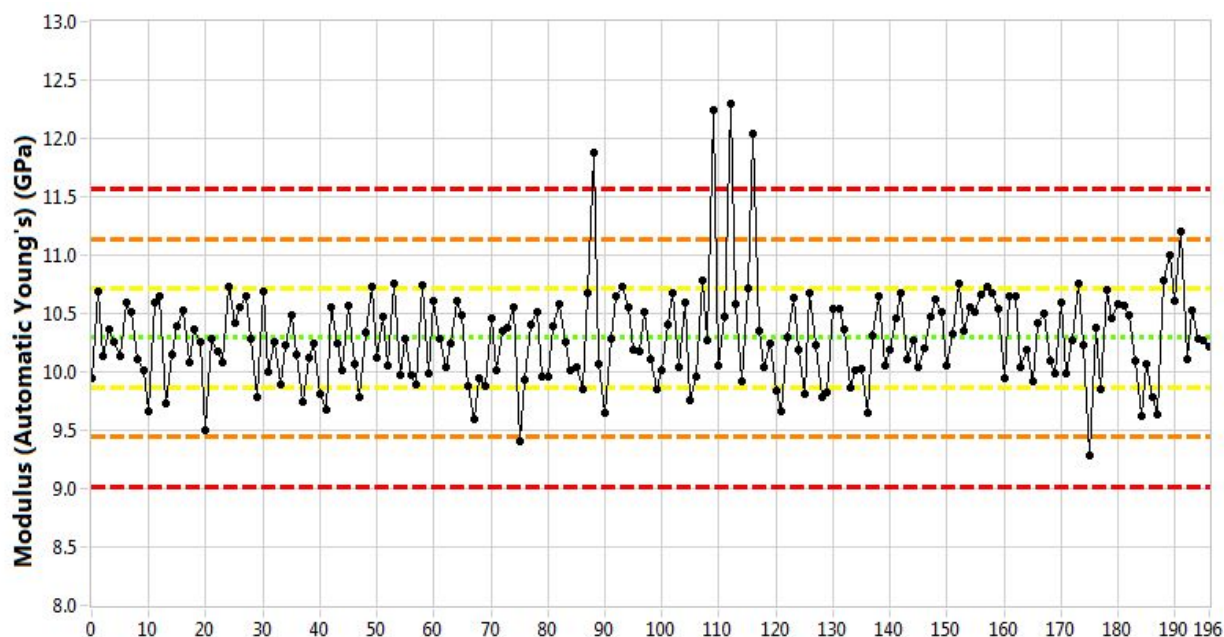


Figure C-1. Modulus (automatic Young's) (GPa), mean = 10.3, standard deviation = 0.42.

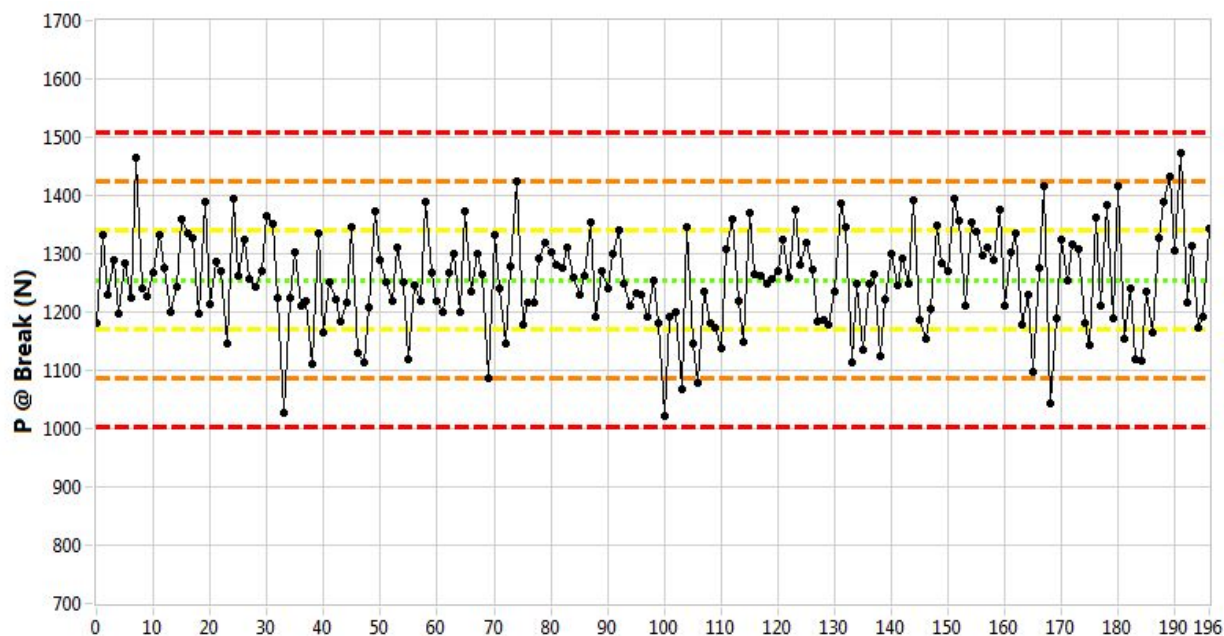


Figure C-2. Load at break (N), mean = 1255, standard deviation = 84.



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

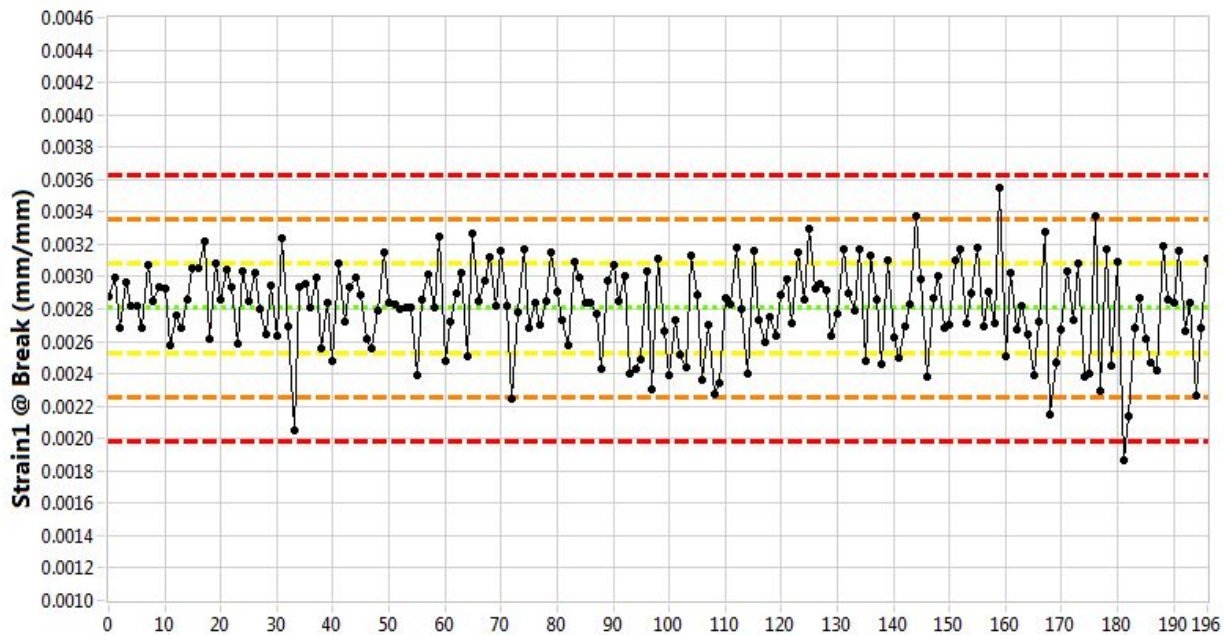


Figure C-3. Strain 1 at break (mm/mm), mean = 0.0028, standard deviation = 0.0003.

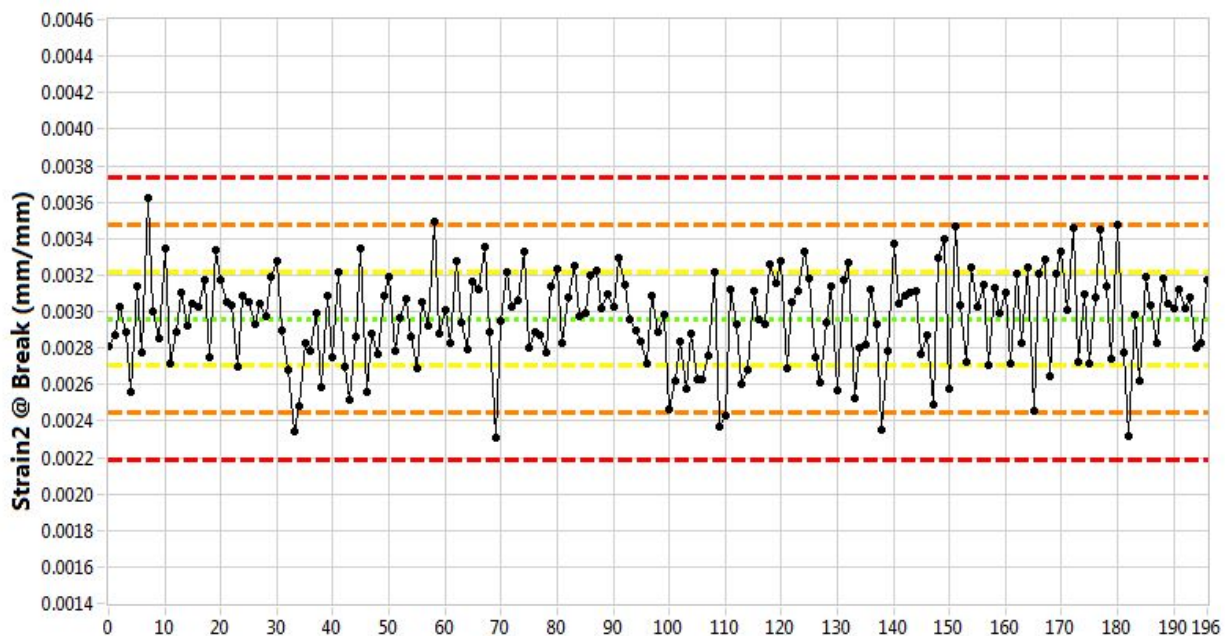


Figure C-4. Strain 2 at break (mm/mm), mean = 0.003, standard deviation = 0.0003.



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

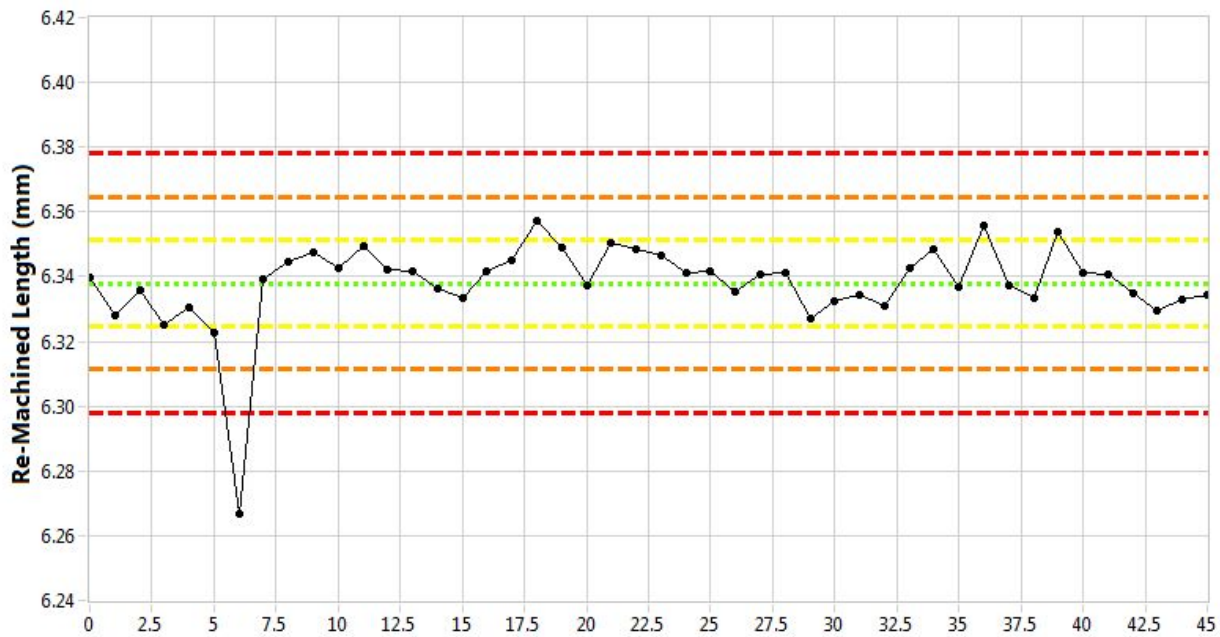


Figure C-5. Re-machined specimens length (mm), mean = 6.338, standard deviation = 0.0133.

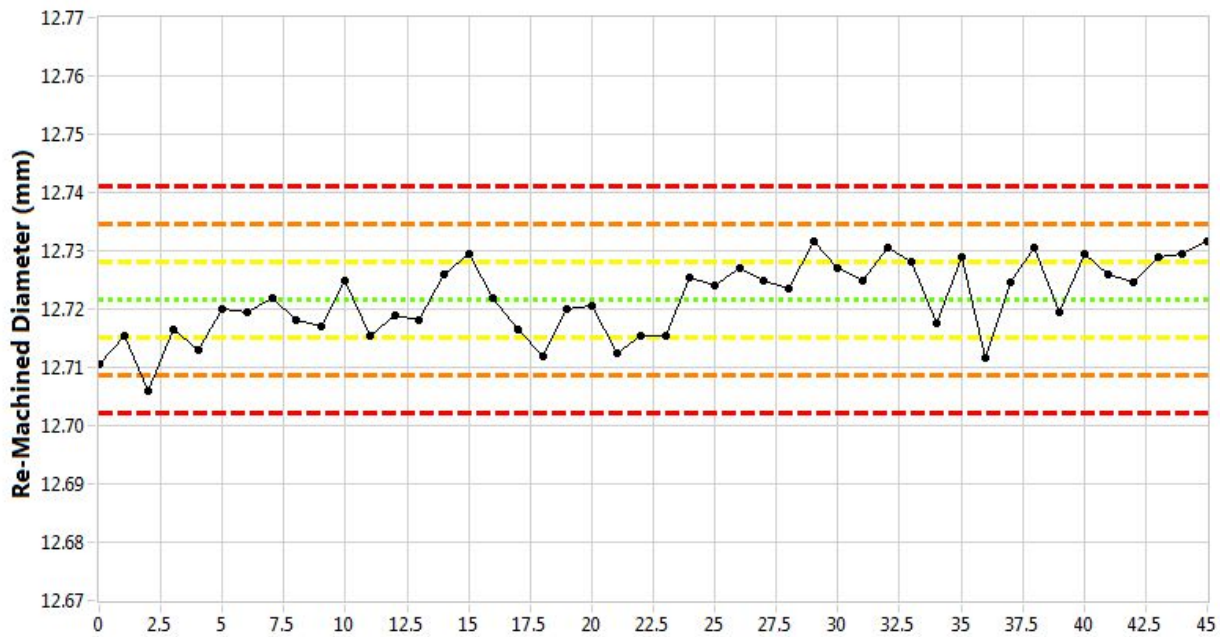


Figure C-6. Re-machined specimens diameter (mm), mean = 12.722, standard deviation = 0.0064.



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

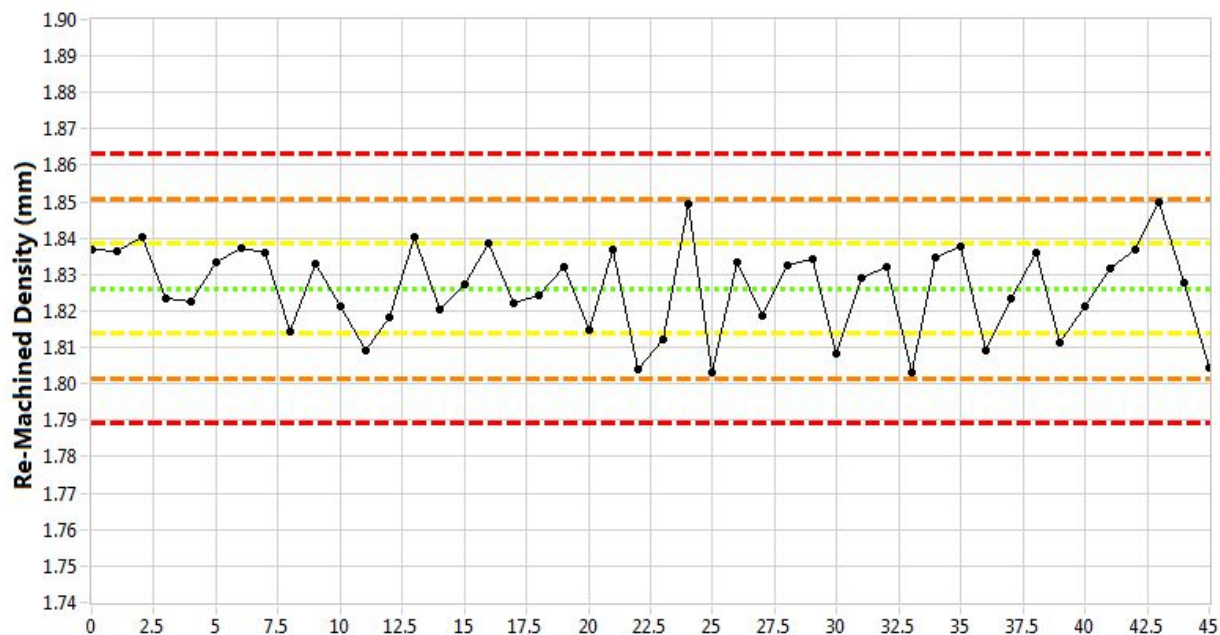


Figure C-7. Re-machined specimens density ( $\text{g}/\text{cm}^3$ ), mean = 1.8262, standard deviation = 0.0123.

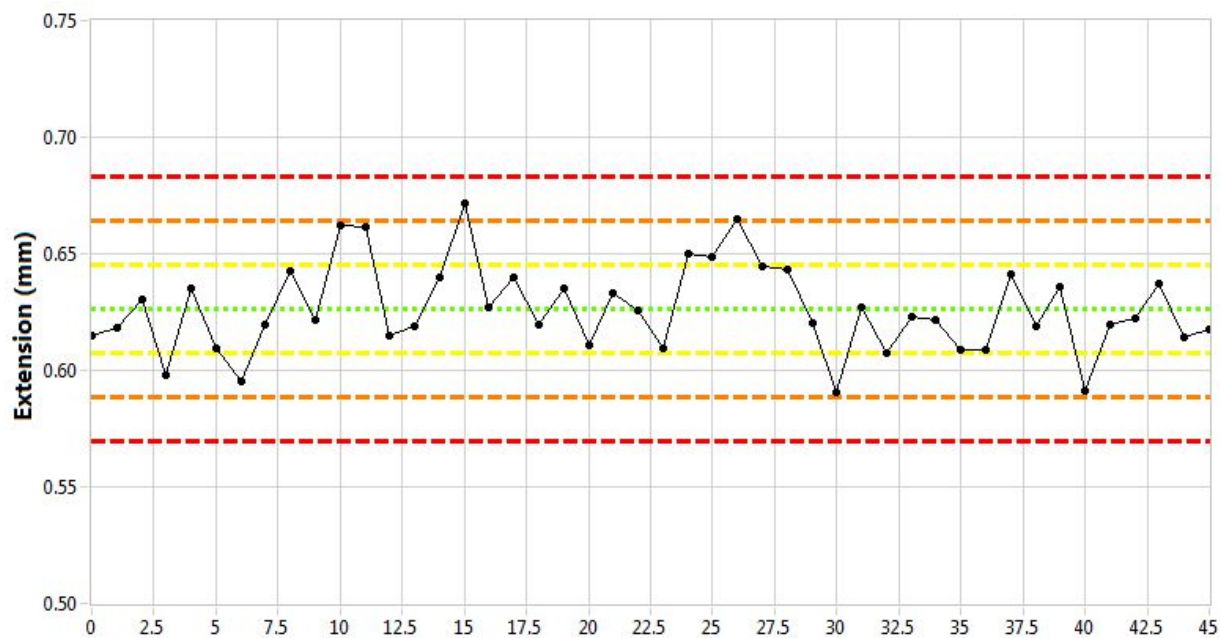


Figure C-8. Disc splitting extension (mm), mean = 0.6263, standard deviation = 0.0188.



Title: Baseline Characterization Database Verification Report – NBG-17 Billet 830-3

ECAR No.: 3727

Rev. No.: 0

Project No.: 32138

Date: 07/31/2017

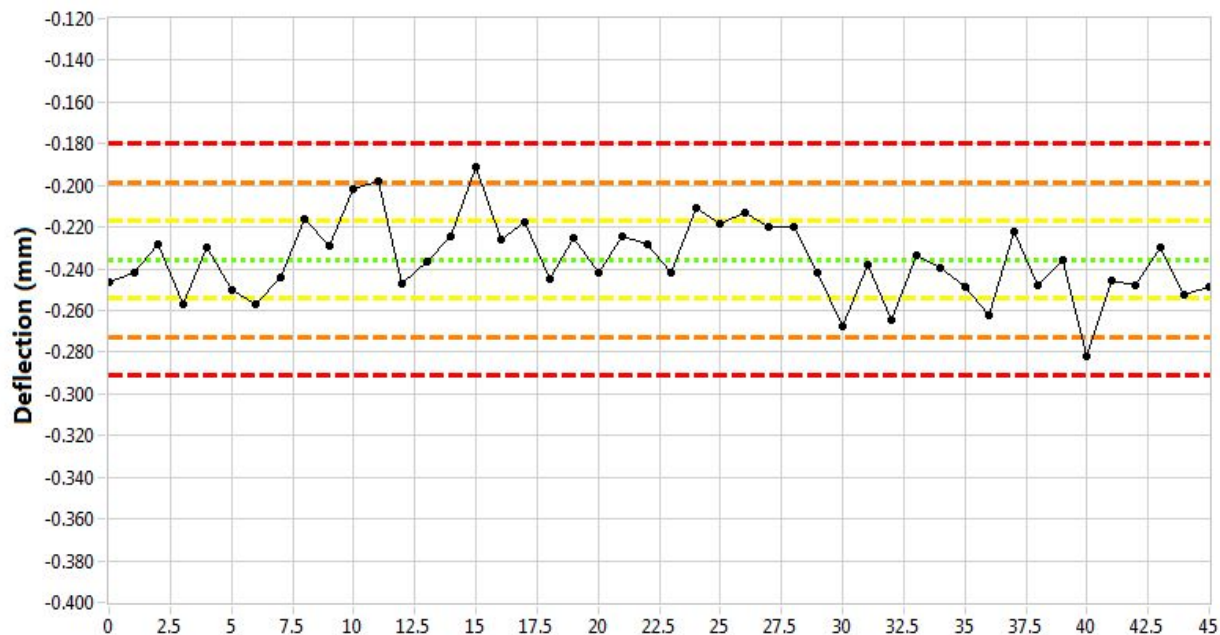


Figure C-9. Disc splitting deflection (mm), mean = -0.2356, standard deviation = 0.0185.