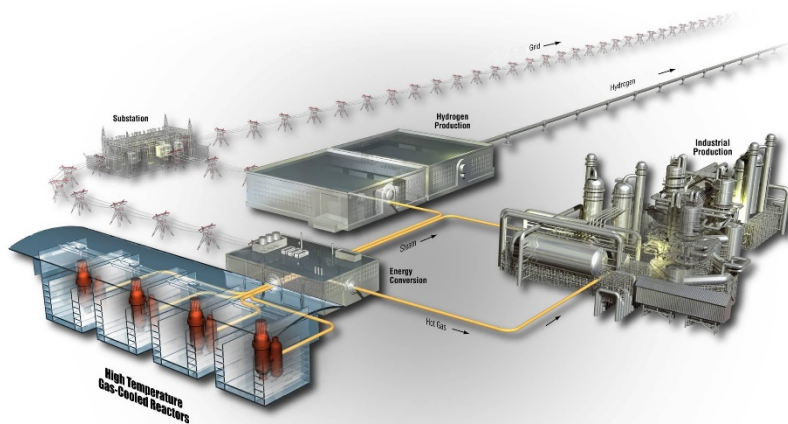


AGC-3 Irradiation Creep Strain Data Analysis

William E. Windes
David T. Rohrbaugh
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July 2019

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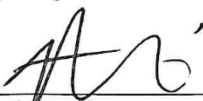
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SUMMARY

This report documents the analysis of the creep-strain data from the third Advanced Graphite Creep (AGC-3) irradiation capsule. This is the third in a series of six irradiation test trains planned as part of the AGC experiment to fully characterize the neutron irradiation effects and radiation creep behavior of current (2019) nuclear graphite grades. The AGC-3 capsule was irradiated in the Idaho National Laboratory Advanced Test Reactor at a nominal temperature of 800°C, beginning with irradiation Cycle 152B on November 28, 2012 and ending with Cycle 155B on April 12, 2014, with a total received dose range of 0.9–3.7 dpa.

AGC-3 was designed to provide irradiation conditions similar to AGC-1 and AGC-2 (similar graphite grades tested, specimen dimensions, mechanical loading conditions), but at a different nominal irradiation temperature of 800°C. AGC-3 was irradiated for a shorter duration than that planned for AGC-4 to provide material property values at lower dose levels. After irradiation, material property and dimensional strain measurements were conducted on all AGC-3 specimens (from 11 nuclear graphite grades) using the same equipment and approved standards as were conducted before irradiation. The specimen loading configuration for all graphite grades within AGC-3 followed a similar pattern as earlier AGC capsules to provide easy future comparison of all irradiated material property data.

Significant modifications were made to the AGC-3 capsule design to improve the uniformity of the specimen temperature. These changes significantly improved the axial temperature variations as encountered within AGC-1. While the AGC-1 irradiation temperature ranged from 409 to 761°C (average 585°C \pm 176°C), the AGC-3 temperature range was significantly reduced to 748–918°C (average 833°C \pm 85°C).

The dimensional change analysis demonstrated that all five major graphite grades had similar irradiation response over the full irradiation temperature range. The isotropic irradiation dimensional behavior for all grades was analyzed by comparing the length and diameter changes for all major grade specimens along with the volumetric behavior of creep (stressed) and control (unstressed) specimens. All grades had isotropic or near-isotropic irradiation response and no grade indicated Turnaround dose was reached at the temperature and dose range of the AGC-3 experiment.

Since both the irradiation dimensional change and creep strain are linear with dose, it was possible to calculate creep coefficients from linear regressions drawn through the dimensional change data. This analytic methodology was used in developing an application to automatically compile and calculate the irradiation-induced creep and creep coefficient for any of the major graphite grades. This QA-validated tool, referred to as the “Graphite Analysis Tool” (GAT), has

provided an efficient and accurate method to calculate the creep coefficient as a function of the AGC irradiation variables.

The creep response and calculated coefficients for all AGC-3 major grades were reasonable and within expected levels. The AGC-3 creep strain behavior was shown to compare well with historical data as well as data from AGC-1 and AGC-2 experiments.

CONTENTS

SUMMARY	v
ABBREVIATIONS	x
1. INTRODUCTION	1
2. ADVANCED GRAPHITE CREEP EXPERIMENT	2
2.1 Design Parameters of AGC Experiment	2
2.2 AGC Graphite Grades and Specimen Dimensions	4
2.3 General AGC Test Train Design.....	5
2.4 Establishing Specimen Dose and Applied Load	6
2.5 Physical Positions of Creep Specimens in the Stacks	8
2.6 Future changes to AGC Experiment	9
3. AGC-3 TEST TRAIN CAPSULE	9
3.1 AGC-3 Graphite Grades.....	10
3.2 AGC-3 Specimen Stack Positions.....	11
4. AGC-3 AS-RUN IRRADIATION CONDITIONS	20
4.1 AGC-3 Irradiation	21
4.2 AGC-3 Mechanical Loading Issues	21
4.3 AGC-3 Temperature	22
5. CREEP STRAIN DATA	22
6. CREEP STRAIN ANALYSIS METHODOLOGY	26
6.1 Methodology Steps	26
7. DIMENSIONAL CHANGE ANALYSIS	27
7.1 Dimensional Change Analysis by Graphite Grade	28
7.2 Volume Change Analysis by Graphite Grade.....	30
8. CREEP ANALYSIS.....	31
8.1 Creep Analysis by Graphite Grade	31
9. CREEP AND CREEP COEFFICIENT ANALYSIS BY GRADE	35
10. CONCLUSIONS	37
11. REFERENCES	39

FIGURES

Figure 1. Irradiation dose and temperature parameters for the AGC experiment (HTV = high temperature vessel, MSR = molten-salt reactor, PB = pebble bed).....	3
Figure 2. The AGC-3 creep capsule.....	6
Figure 3. Elevation sketch of the AGC capsule.....	7
Figure 4. A typical dose profile for creep graphite specimens utilizing similar applied stress levels in matched stacks.....	8
Figure 5. Total maximum volume decrease (%) due to irradiation creep for five major grades of graphite. The dimensional change dependency on irradiation dose and irradiation temperature are not presented.....	23
Figure 6. Average length decrease (%) at maximum dose received for five major grades of graphite. The dimensional change dependency on irradiation dose and irradiation temperature are not presented.....	23
Figure 7. Total maximum diameter decrease (%) due to irradiation creep for five major grades of graphite. Note the diameter decrease in stressed (creep) samples was lower than unstressed (control) samples due to “barreling” effects mitigating the shrinkage (note: barreling produced a variety of out-of-round shapes, including hourglass, pear, and the simple barrel shape). The dimensional change dependency on irradiation dose and irradiation temperature are not presented.	24
Figure 8. A schematic representation of the three stages of irradiation-induced creep.	25
Figure 9. Dimensional change of all AGC-3 major grades of graphite as a function of dose. Data includes creep, control, and button piggyback measurements.....	28
Figure 10. Comparison of dimensional change in longitudinal and diametral directions for vibrational molded graphite grades NBG-17 and NBG-18 unstressed control specimens. Hollow points are taken from the billet in the WG direction, and the red data points indicate specimens taken in the AG direction.	29
Figure 12. Dimensional change in longitudinal and diametral directions for unstressed control specimens of extruded graphite grade PCEA. Hollow data points indicate specimens taken from the billet in the WG direction, and red-filled points indicate AG specimens.....	29
Figure 13. Volume changes of vibrational molded graphite grades NBG-17 and NBG-18 for creep and control specimens. Red data points indicate specimens taken from the billet in the AG direction all others are WG.	30
Figure 14. Volume change of isostatically molded graphite grades IG-110 and 2114 for both creep and control specimens. Red data points indicate specimens taken from the billet in the AG direction all others are WG.	30
Figure 15. Volume change of extruded graphite grade PCEA for creep and control specimens. Red-filled points indicate specimens taken from the billet in the AG direction all others are WG.....	31
Figure 16. Longitudinal creep for all major grades of graphite in AGC-3. Both 13.8 and 20.7 MPa stress levels are included. AG specimens (represented by red dots) are included in this analysis as there is no discernable difference in the WG and AG response for AGC-3 irradiation conditions.....	32

Figure 17. Longitudinal creep and creep coefficient K for NBG-17. K is calculated for two stress levels.	32
Figure 18. Longitudinal creep and creep coefficient K for NBG-18. K is calculated for two stress levels.	33
Figure 19. Longitudinal creep and creep coefficient K for PCEA. K is calculated for two stress levels.	33
Figure 20. Longitudinal creep and creep coefficient K for IG-110. K is calculated for two stress levels.	34
Figure 21. Longitudinal creep and creep coefficient K for 2114. K is calculated for two stress levels.	34
Figure 23. Comparison of AGC creep coefficients (\diamond) to historical values (\bullet) from previous studies.	37

TABLES

Table 1. Major, minor, alternate, and experimental graphite grades within the AGC-3 capsule.	10
Table 2. Total number of irradiated-creep specimens in the AGC-3 test series capsule.	11
Table 3. AGC-3 loading order for Stack 1.	12
Table 4. AGC-3 loading order for Stack 2.	13
Table 5. AGC-3 loading order for Stack 3.	14
Table 6. AGC-3 loading order for Stack 4.	15
Table 7. AGC-3 loading order for Stack 5.	16
Table 8. AGC-3 loading order for Stack 6.	17
Table 9a. AGC-3 loading order for Stack 7 (center channel).	18
Table 9b. AGC-3 loading order for Stack 7 (center channel).	19
Table 9c. AGC-3 loading order for Stack 7 (center channel).	20
Table 10. Stress values for each specimen stack.	22
Table 11. Calculated longitudinal creep coefficients (K) for AGC-1, AGC-2, and AGC-3 graphite grades. The K value for AGC-3 was calculated for 20.7 MPa.	35

ABBREVIATIONS

AG	against grain
AGC	Advanced Graphite Creep
ART	Advanced Reactor Technology
ATR	Advanced Test Reactor
DOE	Department of Energy
dpa	displacements per atom
GAT	Graphite Analysis Tool
HDG	High Dose Graphite capsule
HOPG	highly ordered pyrolytic graphite
HTR	high temperature reactor
INL	Idaho National Laboratory
K	creep coefficient
LDVT	Linear Variable Differential Transformer
PIE	post-irradiation examination
QA	quality assurance
WG	with grain

AGC-3 Irradiation Creep Strain Data Analysis

1. INTRODUCTION

The Advanced Reactor Technologies Graphite Research and Development Program is conducting an extensive graphite irradiation experiment to provide data for licensing of a high temperature reactor (HTR) design. In past applications, graphite has been used effectively as a structural and moderator material in both research and commercial high temperature gas-cooled reactor designs.^{1,2} Nuclear graphite H-451, used previously in the United States for nuclear reactor graphite components, is no longer available. New nuclear graphite grades have been developed and are considered suitable candidates for new HTR reactor designs. To support the design and licensing of HTR core components within a commercial reactor, a complete irradiated properties database must be developed for these current grades of graphite. Quantitative data on in-service material performance are required for the physical, mechanical, and thermal properties of each graphite grade, with a specific emphasis on data accounting for the life-limiting effects of irradiation creep on key physical properties of the HTR candidate graphite grades. Further details on the research and development activities and associated rationale required to qualify nuclear-grade graphite for use within the HTR are documented in the graphite technology research and development plan.³

The phenomenon of irradiation-induced creep within graphite has been shown to be critical to predicting the total useful lifetime of graphite components. Irradiation-induced creep occurs under the simultaneous application of neutron irradiation, irradiation temperature, and applied stresses within the graphite components. Significant internal stresses within the graphite components can result from a second phenomenon due to dose and temperature gradients—irradiation-induced dimensional change—where the graphite physically changes (i.e., first shrinking and then expanding with increasing neutron dose). This disparity in material volume change can induce significant internal stresses within graphite components. Irradiation-induced creep can relax these large internal stresses, thus reducing the risk of crack formation and component failure. Graphite grades exhibiting higher levels of irradiation creep will relieve more internal stress providing the components longer useful lifetimes within the core. Determining the irradiation creep rates of different graphite grades (i.e., differences in grain size, coke source, and fabrication process) is critical for predicting the useful lifetime of graphite components and is a major component of the Advanced Graphite Creep (AGC) experiment.

The AGC experiment is currently ongoing to determine the in-service behavior of these new graphite grades for HTR. This test series will examine the properties and behaviors of nuclear-grade graphite over a large spectrum of temperatures, irradiation fluence, and applied stress levels that are expected to induce irradiation creep strains within an HTR graphite component. Irradiation data are provided through the AGC test series, which comprises six planned capsules irradiated in a large flux trap in the Advanced Test Reactor (ATR) at Idaho National Laboratory (INL). The AGC irradiation conditions are designed to be similar to the anticipated environment within a high temperature core design. Each irradiation capsule is composed of more than 400 graphite specimens that are characterized before and after irradiation to determine the irradiation-induced changes in material properties and the rate of life-limiting irradiation creep for each graphite grade.

This report provides an AGC post-irradiation examination (PIE) analysis of the irradiation-induced creep strain within the third AGC capsule (AGC-3). The AGC-3 ($T_{\text{irr}} = 800^{\circ}\text{C}$) calculated creep coefficients were compared with AGC-1 and AGC-2 ($T_{\text{irr}} = 600^{\circ}\text{C}$) values along with historical data from literature to ascertain any significant trends in the data. In this way, the consistency and soundness of the data are initially tested and validated from previous creep analyses.

The data and information produced in this document and the referenced documents within were generated under the approved quality assurance (QA) programs for the respective organizations, including INL and Oak Ridge National Laboratory, in compliance with the appropriate NQA-1 requirements. It is anticipated that all data will be robust enough to stand up to a review by the Nuclear Regulatory Commission as support for a graphite reactor design selection.

2. ADVANCED GRAPHITE CREEP EXPERIMENT

The AGC test series is designed to establish the data necessary to determine the safe operating envelope of graphite core components for an HTR by measuring the irradiated material property changes and the behavior of several new nuclear graphite grades over a large range of temperatures, neutron fluence, and mechanical compressive loads. The experiment consists of three interrelated stages: pre-irradiation characterization of the graphite specimens, the irradiation test series (designated as six separate irradiation test train capsules), and PIE and analysis of the graphite specimens after irradiation. Separate reports for each distinct stage are prepared after each individual activity is completed.

The pre-irradiation examination report details the total number of graphite grades and individual specimens, the specimen loading configuration designed to expose all specimens to the entire range of irradiation conditions, and the pre-irradiation material property testing data and results. The as-run irradiation report details the irradiation history of each capsule while in the reactor, noting any changes from the technical and functional specifications for each specific test series capsule and identifying the possible improvements to the next test series capsule design. The disassembly report details specimen recovery from the irradiation capsule, noting any damage to the specimens and providing an inventory of recovered specimens for PIE testing. The PIE data package report details the property measurements for specimen dimensional changes (for stressed as well as un-stressed specimens) and irradiated material properties upon exposure to neutron irradiation. Finally, the PIE analysis report analyzes the irradiation measurements reported within the data package report, utilizing the irradiation conditions recorded within the as-run irradiation report. The PIE analysis reports contain determinations of irradiation-induced creep rates for the major grades of graphite and assessments of any changes to the material properties in all graphite grades. These reports also attempt to interpret the irradiation behavior of graphite to assist in determining a credible safe operating envelope for graphite core components in an HTR design and licensing application.

This report is an AGC PIE analysis report on irradiation-induced creep strain within the third AGC capsule (AGC-3). Data from the “AGC-3 Specimen Post-Irradiation Data Package Report”⁴ was used to analyze dimensional-strain data in order to calculate corresponding creep strain and creep coefficients for each graphite grade. An analysis of the irradiation material property (i.e., electrical resistivity, thermal diffusivity, coefficient of thermal expansion, stiffness, and strength) changes will be provided within a future PIE analysis report.

2.1 Design Parameters of AGC Experiment

The AGC test series is designed to measure irradiation induced changes in key thermal, physical, and mechanical material properties over the anticipated range of HTR operating conditions. By comparing the material properties of each specimen before and after irradiation, the experiment generates quantitative material property change data and irradiation creep data that will be used to predict the in-service behavior and operating performance of the current nuclear graphite grades for HTR designs. Specific emphasis is placed on data that pertain to the life-limiting effects of irradiation creep on graphite components and the effects creep may have on key irradiated material properties of several candidate graphite grades for use in an HTR design.

The critical component of the experiment is the irradiation test series, which irradiates the graphite specimens after pre-irradiation examination characterization has been completed. The AGC test series is composed of six planned irradiation test trains that are irradiated in ATR in a large flux trap, as described in the Graphite Technology Development Plan.³ The test series exposes test specimens of select nuclear graphite grades to temperatures and the initial range of irradiation dose that are expected within an HTR design. Specifically, graphite specimens will be exposed to a fast neutron dose ranging from 1 to 7 dpa and temperatures of 600, 800, and 1,100°C, as shown in Figure 1. The first and second AGC capsules, AGC-1 and AGC-2, were designed to be irradiated within the ATR's south flux trap.⁵ All other AGC capsules will be irradiated within ATR's east flux trap including the current AGC-3 irradiation capsule. Generally, irradiations the east flux trap require approximately 200 effective full-power days to provide a nominal fast neutron dose range (in graphite) of approximately 0.5–3.5 dpa. For capsules requiring a large dose range of approximately 3.5–7.0 dpa, the irradiation capsule (containing the graphite specimens) is irradiated for twice as long inside the ATR, approximately 400 effective full-power days.

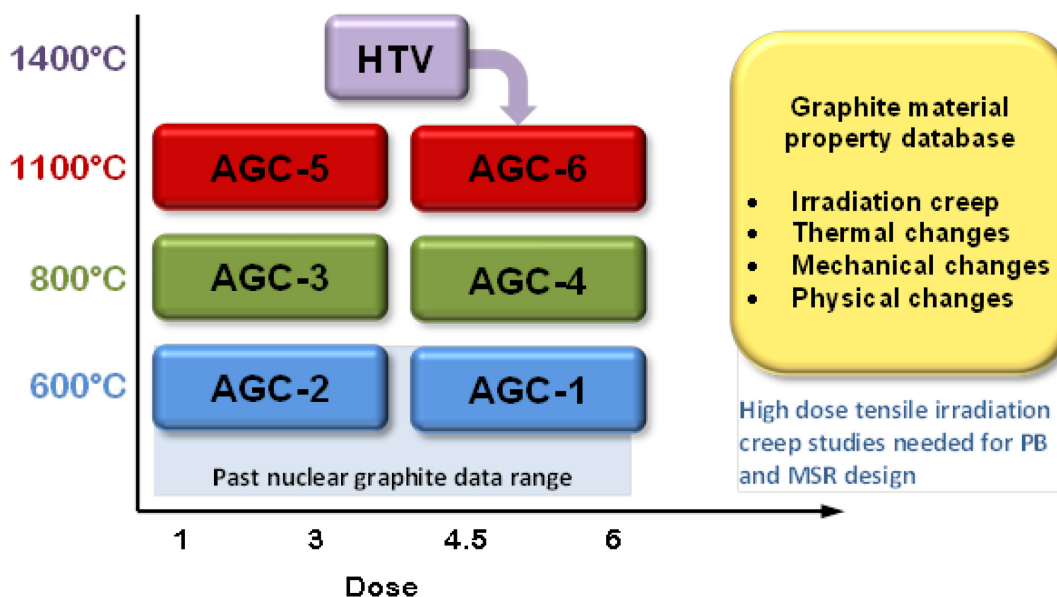


Figure 1. Irradiation dose and temperature parameters for the AGC experiment (HTV = high temperature vessel, MSR = molten-salt reactor, PB = pebble bed).

In addition to determining irradiation-induced changes to the material properties of selected nuclear graphite grades, the AGC experiment dedicates a significant amount of scope to determining rates of irradiation-induced creep for different nuclear graphite grades. The traditional method for measuring irradiation-induced creep is to apply a significant mechanical load (inducing a mechanical stress within the graphite) to half the specimens during irradiation while leaving the remaining half of the specimens unloaded (unstressed). Mechanically loaded (stressed) specimens are traditionally designated as the creep specimens, and the unloaded (unstressed) specimens are designated as the control specimens. The resulting difference in dimensional change between the stressed and unstressed specimens (assuming that temperature and dose levels are the same) provides the amount of irradiation-induced strain for each “matched pair” of graphite specimens. From this strain level, a creep rate for each graphite grade can be calculated as a function of dose if both specimens were irradiated at the same constant temperature and dose level. Thus, each capsule is designed to be irradiated at a constant temperature, allowing only the dose and applied mechanical load to vary within the test train of each test-series capsule. With all graphite specimens at a constant temperature, only the applied stress level and dose will affect the calculated creep rate of each graphite grade within a test series capsule.

The AGC experiment is designed to measure constant creep strain behavior (secondary creep) of the various grades. The experiment assumes that the induced creep strain for all specimens is within the secondary creep regime and, therefore, behaves linearly with respect to received neutron dose.³ This assumption is valid provided the specimens do not go beyond their turnaround dose, where creep strain can no longer be expected to be linear with irradiation dose (the onset of tertiary creep). Once the specimens begin to reach turnaround, the creep strain response becomes nonlinear with received dose.

While the effects from applied mechanical stresses and neutron dose can be determined within each irradiation capsule, the temperature dependency of any irradiation-induced material property changes within the graphite grades is achieved by comparing the measured values of the specimens between irradiation capsules. Because each test train is irradiated at a constant temperature (600, 800 or 1,100°C), the temperature-induced/enhanced material property changes must be determined by comparing specimens in different capsules exposed to similar doses and applied mechanical load levels. All AGC capsules are designed to have the same specimen stacking patterns. Thus, if specimens of identical graphite grades are located in similar positions within each capsule, a similar dose and load level will be imposed on a consistent grade of graphite. Maintaining consistent specimen positions for each grade within the six different capsules will allow the determination of temperature-induced changes for irradiation creep and material properties across the AGC experiment.

2.2 AGC Graphite Grades and Specimen Dimensions

The AGC experiment is designed to ascertain the irradiation behavior of currently available nuclear graphite grades within the anticipated operating parameters of an HTR design. By exposing a variety of nuclear graphite grades representing the range of fabrication parameters (e.g., grain size, fabrication processes, and raw source material) to the expected operating conditions for a moderate HTR design (600 to 1100°C and 0.5 to 7 dpa dose), a comprehensive understanding of the irradiation response and behavior of graphite components in general can be achieved. This will limit the need for additional research in the future if the current graphite grades are altered (i.e., new raw material sources are used) or new grades are used in future reactors.

The AGC experiment uses a variety of current graphite grades to envelope the major fabrication parameters believed to be responsible for the irradiation behavior of nuclear graphite.¹ This range of fabrication parameters is represented by AGC major grades, which were deemed to be production-ready grades that could be used in current or future HTR designs. Major graphite grades are one type of sample within the AGC irradiation capsule. In addition, four other sample types are designated within the AGC experiment. The five AGC sample types are categorized as follows:⁶

1. Major grades (irradiation creep and control specimens). These graphite grades are current reactor candidates for the core structures of an HTR design. Historical or reference grades are considered major grades as well. Due to their fabrication maturity and consistency, HTR core components are most likely to be formed from these major grades and are thus expected to receive reasonably large neutron doses in their lifetime. Only major grade specimens were used to determine the critical irradiation-induced creep strain rate.
2. Minor grades (6 mm tall, button-shaped piggyback specimens). These grades are HTR-relevant grades that are not yet production ready or are most likely to be used in low-neutron-dose regions of the core (e.g., the permanent structure of the prismatic-block HTR design).
3. Alternate grades (piggyback specimens). Grades that current HTR vendors have identified as being of interest as alternate graphite grades for certain components within the reactor.
4. Experimental grades (piggyback specimens). These graphite grades are included in AGC to assess the viability of new graphite grades, the manufacturing processes and raw materials of which are such

that they may offer superior irradiation stability. Additionally, other carbonaceous materials, such as fuel compact matrix materials, carbon-carbon composites, silicon-carbide composites, or other experimental materials that could offer superior performance within the extreme environment of an HTR core are included.

5. Single crystal graphite (piggyback specimens). Samples of highly ordered pyrolytic graphite (HOPG) are included in all AGC capsules to assess the fundamental irradiation response of single crystal graphite. These specimens offer specific dimensional change behavior of graphite, which is particularly significant to the behavior of polycrystalline (polygranular) graphite grades.

To provide all necessary material property tests in the AGC experiment, each test-series capsule contains two primary specimens: (1) “creep” specimens, providing irradiation creep-rate value as well as mechanical properties; and (2) “piggyback” specimens, providing thermal material property changes to the graphite. Creep specimens are fabricated only from major grade graphite types.² Piggyback specimens are fabricated from major, minor, and experimental grade graphite types. The piggyback specimens are not mechanically loaded and are subjected only to neutron irradiation at high operating temperatures to assess the effects of a reactor environment on the specific graphite grade.

All specimens are 12.4 mm in diameter, with the larger creep specimens having a nominal length of 25.4 mm, and the piggyback specimens having a nominal length of 6.3 mm. Small graphite containers that are 12.4 mm in diameter by 6 mm long contain the thin wafer HOPG specimens, while 12.7-mm-long containers the SGL Carbon, Inc. designated MLRF specimens (experimental silicon-carbide coated graphite specimens from SGL Carbon being irradiated in AGC-3 capsule only). The large creep specimens provide a means to acquire dimensional and volume change, elastic modulus, thermal expansion, and electrical resistivity measurements. However, these creep specimens are not suitable for thermal diffusivity measurements because of their length. The small piggyback specimens permitted only dimensional measurements, density, and thermal diffusivity testing to be performed. Together, both types of specimens provide the changes in material properties for stressed and unstressed graphite grades.^{3,4}

2.3 General AGC Test Train Design

All AGC test trains and irradiation capsules have the same general physical configuration to provide consistent dose and applied mechanical stresses on specimens of similar graphite grades. While there are key machining and structural differences between capsules to change the irradiation temperature for the different capsules, the majority of the AGC design is identical for all capsules. A schematic of the AGC-3 test train and location of graphite specimens within the test train is shown in Figure 2.

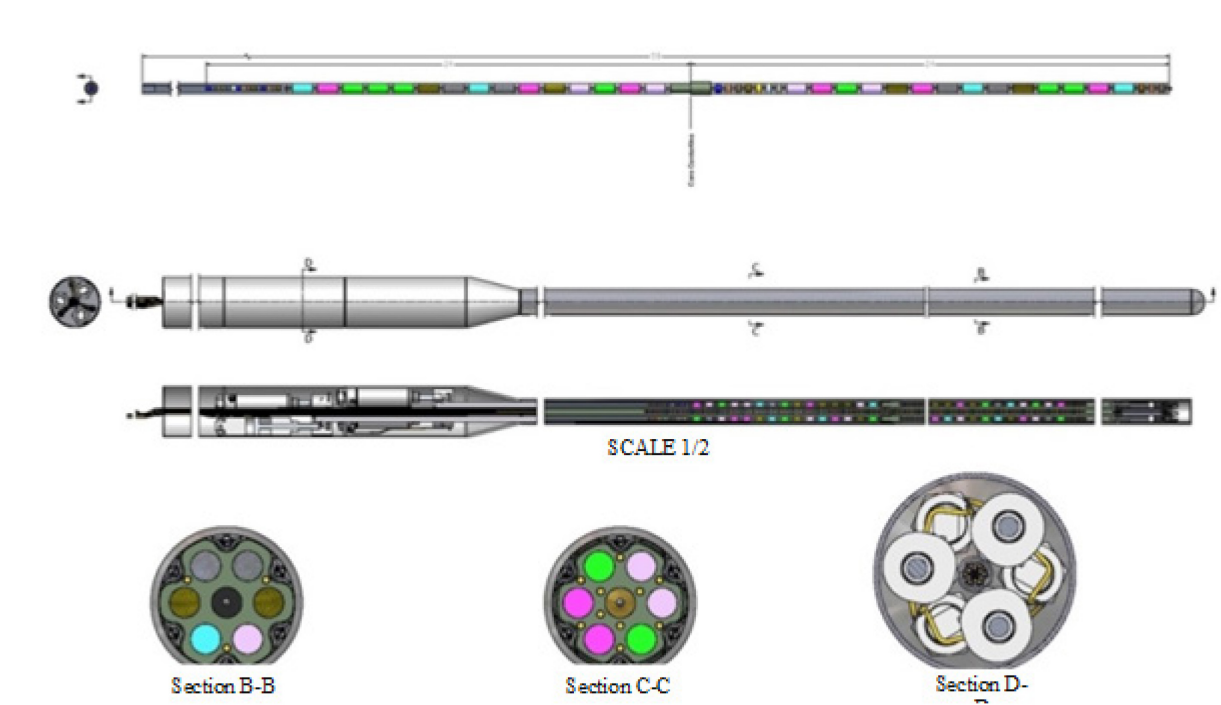


Figure 2. The AGC-3 creep capsule.

All irradiation capsules have six channels located on the outer perimeter of the graphite specimen holder body and a center channel.^{6,7,8} All channels are 12.9 mm in diameter and are designed to hold all types of AGC graphite specimens. The upper (top) half the outer channels has mechanical loads applied to the specimens. However, the lower (bottom) half for these channels has no mechanical load applied to the specimens. Due to the neutron flux profile in ATR, matched pairs with similar neutron fluence and temperatures are achieved by preordering the specimen axial locations. Specimens in the upper half of the channels were stressed by the applied mechanical load while their matched pair received a similar dose in an unstressed state. Three stress levels—13.8, 17.2, and 20.7 MPa nominal—are applied in all AGC capsules to provide a known stress upon the graphite specimens during irradiation. These induced stress levels are high enough to produce irradiation-induced creep strain with the graphite specimens.

Temperature values within all AGC capsules are calculated based upon thermocouple readings at select positions within the capsule. Specimen temperature is calculated with a finite-element model that has been calibrated to predict the known thermocouple readings in the capsule. Dose levels are calculated using Monte Carlo N-Particle Transport Code models and operating conditions in the ATR core and are corroborated from flux wire data.⁹

2.4 Establishing Specimen Dose and Applied Load

To achieve the desired irradiation dose levels and applied mechanical loads to the specific specimens, an exact specimen loading order is critical. Because irradiation creep is usually determined by the difference in dimensional change occurring within matched-pair specimens that have an applied load and those that do not, it is crucial to understand where each and every specimen is located within an AGC capsule.

Specimens within the upper half of the capsule have a mechanical load applied to them via a pneumatic ram system. Specimens within the lower half remain unloaded and thus have no applied stress. A careful specimen loading order within the irradiation capsule is required to ensure similar dose levels

and temperature distribution for specimens in the upper and lower stacks.⁶ Other considerations include the size of each creep specimen, the need for periodically placed flux wires, and the space requirements in the top of the stacks for the pneumatic push rods. The core flux midplane, in relation to the capsule arrangement, was established so that the reactor neutron flux field could be correlated to the physical elevations and positions in the capsule to yield accurate irradiation dose levels, Figure 3.¹⁰

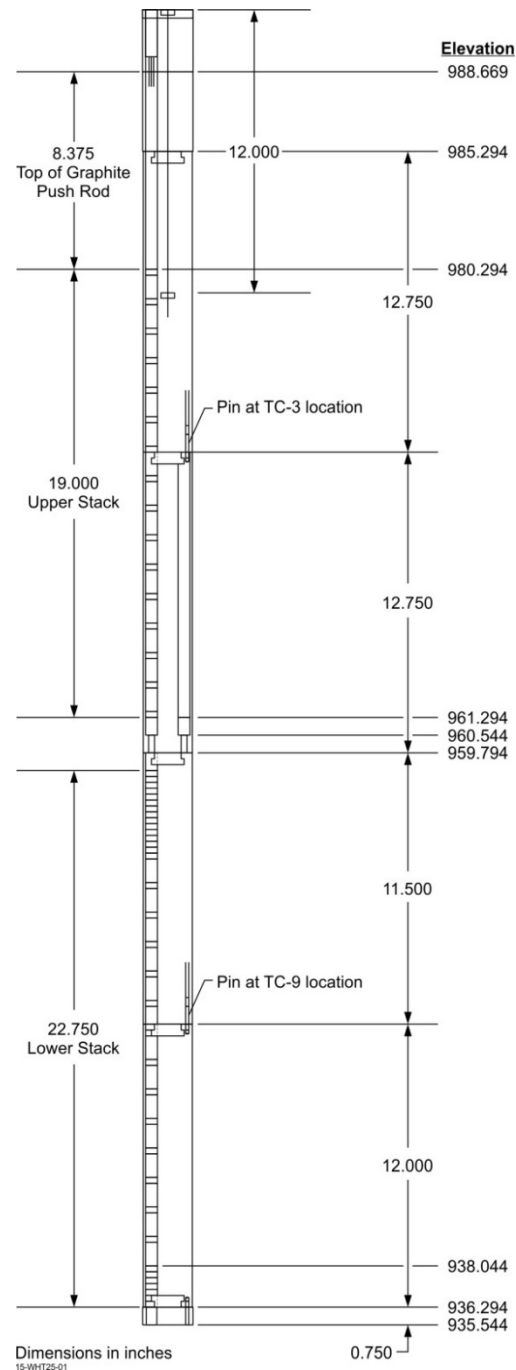


Figure 3. Elevation sketch of the AGC capsule.

Irradiation dose values, as a function of distance from the reactor core centerline, are calculated from the total calculated fluence using standard conversion factors for carbon in a fast neutron irradiation field ($E > 0.1$ MeV).⁹ A neutron flux gradient across the capsule radius requires the capsule to be rotated 180 degrees at the irradiation midpoint. This rotation results in a uniform neutron-fluence profile for all stacks, regardless of their position within the capsule.

As described in previous reports,¹¹ the ATR neutron flux profile is not completely symmetrical along the vertical axis. Thus, to produce a balanced dose for specimens above and below the core centerline, an offset position from the mid-plane is required. An offset distance of 31.75 mm (1.25 in.) from the core mid-plane for the bottom creep specimens produces the closest dose matches between upper and lower stack specimens.

2.5 Physical Positions of Creep Specimens in the Stacks

Once the specimen-position offset is established for the bottom half of the specimens, the number of total creep specimens for each grade of graphite is determined. It should be noted that the specimen stacking order for subsequent AGC irradiation capsules was changed from that initially established for the AGC-1 test train. In the initial AGC capsule design, AGC-1 utilized 6.35-mm-long NBG-25 graphite spacers between all creep specimens to separate them from each other. It was determined that this was not necessary, and the 6.35-mm-long NBG-25 graphite spacers were eliminated for all subsequent AGC test trains. The decision to eliminate spacers increased the total number of creep specimens in the AGC-3 capsule to 222 total specimens.

As mentioned above, the six outer stacks in the capsule allow the specimens in two of the channel stacks to be loaded at 13.8 MPa, while the other two pairs of channels are loaded at 17.2 and 20.7 MPa, respectively. Because two stacks are at similar applied stress levels, the specimen-loading order can be shifted between the two stacks, allowing the same grade of graphite to be mechanically loaded over a broader neutron-dose range, as illustrated in Figure 4. Assuming that both stacks will have the same applied stress level, receive similar dose levels per position, and have a constant temperature allows this shifting of the specimens and, consequently, a more-uniform, smoother dose profile for each graphite grade.

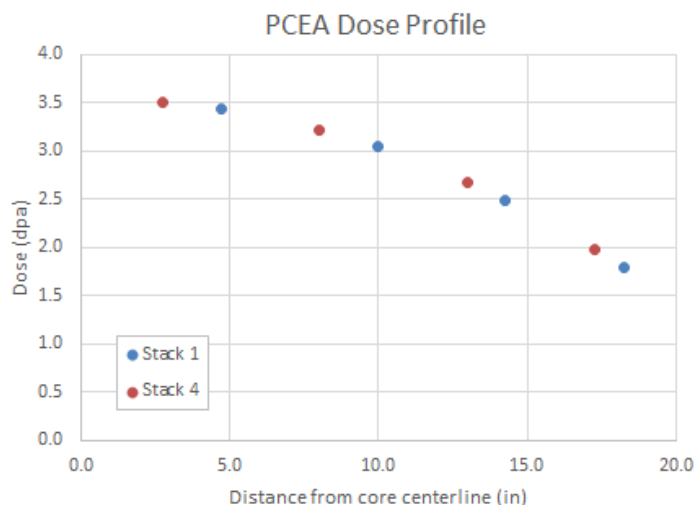


Figure 4. A typical dose profile for creep graphite specimens utilizing similar applied stress levels in matched stacks.

A final consideration when establishing the specimen-loading positions is the grain orientation of the specimens. All AGC capsules attempt to account for the grain orientation relative to irradiation behavior.¹² For extruded graphite grades, the against-grain (AG) and with-grain (WG) directions are obviously perpendicular and parallel to the extrusion direction, respectively. Isomolded grades have little to no grain direction, and there is no consideration for their orientation. However, in the case of the vibration-molded graphites (i.e., NBG-17 and -18), there are actually two WG directions and one AG direction as a consequence of the fabrication process. The total number of WG and AG specimens is dependent on the particular AGC capsule pair (i.e., AGC-1 and AGC-2 had the same number of specimens with similar orientation).

Once these considerations are accounted for, the dose-level profiles are determined for each graphite grade within each channel stack. It should be noted that due to the elimination of the NBG-25 spacers from the AGC-1 design, the dose-level profiles for each graphite grade have been altered for the succeeding AGC capsules.^{11,13,14} However, the changes are modest, allowing nearly direct comparison between AGC-1 and the subsequent AGC capsules.

2.6 Future changes to AGC Experiment

It should be noted that in 2018, The Department of Energy (DOE) Advanced Reactor Technology (ART) approved of a major design change to the AGC Experiment. It was determined that the AGC Experiment should extend the dose range from 0–7 dpa to 0–15 dpa. This new neutron dose increase will extend the current nuclear grades past Turnaround dose levels and into the non-linear tertiary-creep regime. To achieve this higher maximum dose level, it was decided to repurpose the last two irradiation capsules, AGC-5 and AGC-6, which were to be irradiated at 1100°C. Under the new direction (2018), AGC-5 and AGC-6 will be used to irradiate previously exposed specimens from AGC-2, AGC-3, and AGC-4 once they have undergone PIE. AGC-5 will be renamed as HDG-1 (High Dose Graphite) and will re-irradiate AGC-2 specimens at irradiation temperatures of 600°C. AGC-6 will be renamed as HDG-2 and will re-irradiate selected specimens from AGC-3 and AGC-4 at irradiation temperatures of 800°C. No graphite specimens will be irradiated at irradiation temperatures of 1100°C. Once irradiation is complete in HDG-1 and HDG-2, all specimens will undergo a second PIE testing to determine the effects of this higher dose level on material properties.

This major change will not affect AGC-3 or AGC-4 irradiations and PIE. Thus, no changes to the capsule design, specimen testing, or PIE analysis on the initial 0–7 dpa, 800°C irradiations will occur. Any changes in support for the HDG irradiations will be detailed in future HDG-1 and HDG-2 reports.

3. AGC-3 TEST TRAIN CAPSULE

The AGC-3 capsule was irradiated in the ATR at a nominal temperature of 800°C, beginning with irradiation Cycle 152B on November 28, 2012, and ending with Cycle 155B on April 12, 2014.¹⁵ The average estimated irradiation temperature for all samples in the AGC-3 test train was 820°C, with a standard deviation of 30°C and a total range of 748–918°C. While the temperature range was much better than the prototype AGC-1 irradiation capsule, the AGC-3 irradiation temperature range exceeded the design ($800 \pm 50^\circ\text{C}$).¹⁶

The AGC-3 irradiation capsule is the companion capsule for AGC-4. Both of these capsules are to be irradiated at 800°C, but have different dose ranges of 1–3.5 and 3.5–6 dpa, respectively. The average radiation dose of all specimens in AGC-3 was 2.8 dpa, with a standard deviation of 0.8 dpa and a total range of 1.1–3.6 dpa. This includes the piggyback specimens in the extreme top and bottom positions of the irradiation capsule.

The overall design of AGC-3 was kept as close to the AGC-2 capsule as possible. There were, however, some significant modifications made to the AGC-3 capsule. First, a major design change to the

capsule was made to allow finer control of the internal capsule temperature. A new advanced temperature-control system allowed greater control of the temperature conducting gas mixture (the He-Ar gas ratio) within *five distinct* axial regions of the capsule, rather than just one gas composition over the entire capsule length.¹⁷ By altering the gas composition within these five different regions, a much finer control of the temperature throughout the length of the capsule was achieved.

Other design changes included the complete removal of major grades IG-430 and H-451 from AGC-3 capsule. Also, all of the minor grade piggyback specimens were removed and replaced with either major grade or experimental-grade piggyback specimens. All AGC-3 design documents and drawings pertinent to the AGC-3 graphite specimens have been reported in the previous AGC-3 graphite pre-irradiation data analysis report, INL/EXT-13-30297.¹⁸

3.1 AGC-3 Graphite Grades

The major grades of the nuclear graphite tested in AGC-3 are similar to those tested in AGC-2. The difference is that H-451 and IG-430 were removed from the AGC-3 capsule and replaced with 2114. The major grades for AGC-3 are now NBG-17, NBG-18, PCEA, IG-110, and 2114. Minor, alternate, and experimental grades of graphite are presented below:

1. Major grades: NBG-17, NBG-18, PCEA, IG-110, and 2114
2. Minor grades: None
3. Alternate grades: PCIB and GrafTech
4. Experimental grades: SGL, SGL SiC, and MLRF
5. Single crystal graphite: HOPG.

A more complete description of the graphite specimens included in AGC-3 is given in Table 1.⁴ Specimen ID letters were given to each graphite grade in place of their name designations to shorten the specimen identification number for each specimen. This allowed the specimen to have a short ID number which was necessary for the laser engraving of each specimen. For grades NBG-17, NBG-18, IG-110, and PCEA (i.e., codes A, B, D, and E) both WG and AG specimen orientations are included in the capsule.

Table 1. Major, minor, alternate, and experimental graphite grades within the AGC-3 capsule.

Graphite Grade	Forming Method	Intended Purpose	Specimen ID Letter
NBG-17	Vibrational molded	AREVA Next Generation Nuclear Plant design	A
NBG-18	Vibrational molded	Pebble-bed modular reactor (not currently being pursued)	B
PCEA	Extruded	AREVA Next Generation Nuclear Plant design	D
IG-110	Isostatically pressed	HTR-pebble-bed module (China)	E
2114	Isostatically pressed	Candidate graphite	T
HOPG	Vapor deposited	Fundamental studies	CAN101-CAN117
PCIB	Isostatically pressed	Alternative candidate grades	P
GrafTech	Isostatically pressed	Alternate/experiment candidate grades TS and PCIB-MG	200,324,325,328
SGL	Vibrational molded	SiC coating experiments	S1-S20

Graphite Grade	Forming Method	Intended Purpose	Specimen ID Letter
SGL-SiC	Vibrational molded	SiC coating experiments	A-H
MLRF	Vibrational molded	SiC coating experiments	CAN121-CAN129

3.2 AGC-3 Specimen Stack Positions

The final loading configuration for the outer channel/stacks was determined for each graphite grade to optimize the number of specimens for each grade, to create a smooth dose profile for the creep and piggyback specimens, and to ensure the proper position of creep specimens to create a symmetric dose profile above and below the core centerline.

A further decision was made to increase the creep specimen number population for the newer graphite grades because little-to-no irradiation data are available on these grades. Specifically, more specimens of graphite grades NBG-18 and PCEA were chosen to be irradiated instead of the IG-110 and NBG-17 graphite grades.⁴ NBG-18 and PCEA were determined to have 16 specimens per applied stress level for a total of 48 specimens within AGC-3. NBG-17 was represented by only 12 specimens per applied stress level, for a total of 36 specimens, and IG-110 was represented by 14 specimens, totaling 42 specimens within AGC-3. Table 2 lists the total number of specimens irradiated per major graphite grade.

Table 2. Total number of irradiated-creep specimens in the AGC-3 test series capsule.

Graphite Grade	Number of Creep Specimens
PCEA	48
NBG-18	48
IG-110	42
NBG-17	36
2114	48
Total Creep	222

In AGC-3, approximately 75% of the 25.4-mm-long PCEA and IG-110 specimens were oriented in the WG direction and 25% of the specimens were AG. For the vibration-molded graphite grades (NBG-17 and NBG-18), which possess two WG directions and one AG direction, the 25.4-mm-long WG and AG specimens were evenly distributed, i.e. 50/50 ratio.

The orientation of the specimen is designated by the second position in the specimen identification number. Specimen identification numbers possessing a “W” in the second position (e.g., CW101) are specimens in the WG orientation. Specimen identification numbers possessing an “A” in the second position (e.g., DA402) are specimens machined from an AG orientation. As discussed previously, vibrationally molded grades are designated with an “L” or “P” for the two WG orientations (e.g., BP402 for an NBG-18 grade specimen).

The final loading configuration for AGC-3, including creep positions, piggyback order, lower stack offset, and flux wire spacers, was mapped for each graphite specimen as it was loaded into the AGC-3 irradiation capsule during assembly (shown in Table 3 through Table 6 below).^{4,19}

Table 3. AGC-3 loading order for Stack 1.

Loading Order	Specimen ID	Graphite Grade	Nominal Specimen Elevation (in)	End of Test Specimen Elevation (in)	Specimen Temperature (°C)	Specimen Dose (DPA)	Stack Load (lbf)
23	AP3001	NBG-17	19.500	19.096	789	1.6	378
22	1J	Flux Monitor	18.875	18.479	775	1.7	378
21	DW3001	PCEA	18.250	17.858	764	1.8	378
20	BW3001	NBG-18	17.250	16.868	752	2.0	378
19	EW3001	IG-110	16.250	15.876	749	2.2	378
18	TW3001	2114	15.250	14.889	753	2.3	378
17	DW3004	PCEA	14.250	13.899	762	2.5	378
16	Y1	Flux Monitor	13.625	13.290	769	2.6	378
15	BP3002	NBG-18	13.000	12.669	777	2.7	378
14	TW3004	2114	12.000	11.677	789	2.8	378
13	EW3004	IG-110	11.000	10.690	800	2.9	378
12	DW3103	PCEA	10.000	9.707	809	3.1	378
11	BW3101	NBG-18	9.000	8.727	816	3.2	378
10	TW3705	2114	8.000	7.742	821	3.2	378
9	DA	Flux Monitor	7.375	7.132	822	3.3	378
8	AP3301	NBG-17	6.750	6.511	823	3.3	378
7	EW3103	IG-110	5.750	5.533	822	3.4	378
6	DW3203	PCEA	4.750	4.553	821	3.4	378
5	BL3001	NBG-18	3.750	3.579	819	3.5	378
4	TW3010	2114	2.750	2.598	817	3.5	378
3	1L	Flux Monitor	2.125	1.991	816	3.5	378
2	AW3103	NBG-17	1.500	1.370	815	3.5	378
29	TW3605	2114	-1.750	-1.811	816	3.6	0
28	200-9	GrafTech-200	-2.375	-2.432	817	3.6	0
27	AW3104	NBG-17	-3.000	-3.051	818	3.5	0
26	1U	Flux Monitor	-3.625	-3.669	819	3.5	0
25	TW3103	2114	-4.250	-4.290	821	3.5	0
24	BL3004	NBG-18	-5.250	-5.285	823	3.5	0
23	DW3302	PCEA	-6.250	-6.277	825	3.4	0
22	EW3202	IG-110	-7.250	-7.267	826	3.4	0
21	AP3004	NBG-17	-8.250	-8.258	826	3.3	0
20	AL3804	NBG-17	-8.875	-8.877	826	3.3	0
19	TW3107	2114	-9.500	-9.498	826	3.2	0
18	BW3501	NBG-18	-10.500	-10.493	824	3.2	0
17	DW3401	PCEA	-11.500	-11.485	822	3.1	0
16	EW3301	IG-110	-12.500	-12.477	820	3.0	0
15	TW3201	2114	-13.500	-13.473	818	2.8	0
14	BP3005	NBG-18	-14.500	-14.469	816	2.7	0
13	H7	Flux Monitor	-15.125	-15.091	815	2.6	0
12	DW3404	PCEA	-15.750	-15.712	815	2.5	0
11	TW3402	2114	-16.750	-16.707	814	2.4	0
10	EW3304	IG-110	-17.750	-17.703	813	2.2	0
9	BW3301	NBG-18	-18.750	-18.699	813	2.0	0
8	DW3504	PCEA	-19.750	-19.697	814	1.9	0
7	F7	Flux Monitor	-20.375	-20.319	815	1.7	0
6	AP3202	NBG-17	-21.000	-20.942	817	1.6	0
5	TW3608	2114	-22.000	-21.940	824	1.4	0
4	325-12	GrafTech-325	-22.625	-22.564	832	1.2	0
3	200-12	GrafTech-200	-22.875	-22.813	836	1.2	0
2	AL3805	NBG-17	-23.125	-23.062	841	1.1	0

Table 4. AGC-3 loading order for Stack 2.

Loading Order	Specimen ID	Graphite Grade	Nominal Specimen Elevation (in)	End of Test Specimen Elevation (in)	Specimen Temperature (°C)	Specimen Dose (DPA)	Stack Load (lbf)
23	AP3002	NBG-17	19.500	19.073	788	1.6	380
22	EA-SP1	IG-110	18.875	18.457	774	1.7	380
21	DW3002	PCEA	18.250	17.836	762	1.8	380
20	BW3002	NBG-18	17.250	16.846	751	2.0	380
19	EW3802	IG-110	16.250	15.855	748	2.2	380
18	TW3002	2114	15.250	14.868	753	2.4	380
17	DW3101	PCEA	14.250	13.878	762	2.5	380
16	2B	Flux Monitor	13.625	13.270	769	2.6	380
15	BP3003	NBG-18	13.000	12.649	776	2.7	380
14	TW3005	2114	12.000	11.662	789	2.8	380
13	EW3101	IG-110	11.000	10.675	800	3.0	380
12	DW3201	PCEA	10.000	9.693	809	3.1	380
11	BW3402	NBG-18	9.000	8.714	816	3.2	380
10	TW3008	2114	8.000	7.731	820	3.3	380
9	EA-SP2	IG-110	7.375	7.124	822	3.3	380
8	AP3302	NBG-17	6.750	6.503	822	3.4	380
7	EW3104	IG-110	5.750	5.525	822	3.4	380
6	DW3204	PCEA	4.750	4.548	820	3.5	380
5	BL3002	NBG-18	3.750	3.576	818	3.5	380
4	TW3101	2114	2.750	2.597	816	3.5	380
3	FA	Flux Monitor	2.125	1.991	814	3.6	380
2	AW3201	NBG-17	1.500	1.370	813	3.6	380
29	TW3606	2114	-1.750	-1.812	813	3.6	0
28	200-10	GrafTech-200	-2.375	-2.434	815	3.6	0
27	AW3202	NBG-17	-3.000	-3.052	816	3.6	0
26	EA3609	IG-110	-3.625	-3.670	817	3.6	0
25	TW3105	2114	-4.250	-4.290	819	3.5	0
24	BL3101	NBG-18	-5.250	-5.283	821	3.5	0
23	DW3303	PCEA	-6.250	-6.275	823	3.5	0
22	EW3203	IG-110	-7.250	-7.266	824	3.4	0
21	AP3101	NBG-17	-8.250	-8.257	825	3.4	0
20	EA3610	IG-110	-8.875	-8.875	825	3.3	0
19	TW3108	2114	-9.500	-9.496	824	3.3	0
18	BW3202	NBG-18	-10.500	-10.492	823	3.2	0
17	DW3402	PCEA	-11.500	-11.485	821	3.1	0
16	EW3302	IG-110	-12.500	-12.478	819	3.0	0
15	TW3202	2114	-13.500	-13.473	816	2.9	0
14	BP3101	NBG-18	-14.500	-14.469	814	2.7	0
13	7O	Flux Monitor	-15.125	-15.092	813	2.7	0
12	DW3501	PCEA	-15.750	-15.713	811	2.6	0
11	TW3403	2114	-16.750	-16.708	810	2.4	0
10	EW3401	IG-110	-17.750	-17.704	810	2.2	0
9	BW3503	NBG-18	-18.750	-18.702	810	2.1	0
8	DW3601	PCEA	-19.750	-19.700	811	1.9	0
7	EA3611	IG-110	-20.375	-20.321	813	1.7	0
6	AP3203	NBG-17	-21.000	-20.943	816	1.6	0
5	TW3609	2114	-22.000	-21.941	824	1.4	0
4	325-13	GrafTech-325	-22.625	-22.564	832	1.3	0
3	200-13	GrafTech-200	-22.875	-22.814	836	1.2	0
2	AL3806	NBG-17	-23.125	-23.062	842	1.1	0

Table 5. AGC-3 loading order for Stack 3.

Loading Order	Specimen ID	Graphite Grade	Nominal Specimen Elevation (in)	End of Test Specimen Elevation (in)	Specimen Temperature (°C)	Specimen Dose (DPA)	Stack Load (lbf)
23	AP3003	NBG-17	19.500	18.997	787	1.6	566
22	EW-SP1	IG-110	18.875	18.383	772	1.7	566
21	DW3003	PCEA	18.250	17.763	761	1.8	566
20	BW3401	NBG-18	17.250	16.778	750	2.0	566
19	EW3003	IG-110	16.250	15.789	748	2.2	566
18	TW3003	2114	15.250	14.805	753	2.4	566
17	DW3102	PCEA	14.250	13.820	762	2.5	566
16	AF	Flux Monitor	13.625	13.216	770	2.6	566
15	BP3004	NBG-18	13.000	12.597	778	2.7	566
14	TW3006	2114	12.000	11.619	791	2.9	566
13	EW3801	IG-110	11.000	10.637	802	3.0	566
12	DW3202	PCEA	10.000	9.659	812	3.1	566
11	BW3103	NBG-18	9.000	8.687	819	3.2	566
10	TW3009	2114	8.000	7.708	822	3.3	566
9	EW-SP2	IG-110	7.375	7.105	824	3.3	566
8	AP3303	NBG-17	6.750	6.485	824	3.4	566
7	EW3201	IG-110	5.750	5.511	823	3.4	566
6	DW3301	PCEA	4.750	4.538	821	3.5	566
5	BL3003	NBG-18	3.750	3.571	818	3.5	566
4	TW3102	2114	2.750	2.596	815	3.5	566
3	8I	Flux Monitor	2.125	1.993	813	3.6	566
2	AW3203	NBG-17	1.500	1.374	812	3.6	566
29	TW3607	2114	-1.750	-1.814	812	3.6	0
28	200-11	GrafTech-200	-2.375	-2.436	813	3.6	0
27	AW3204	NBG-17	-3.000	-3.054	815	3.6	0
26	EW4607	IG-110	-3.625	-3.672	816	3.6	0
25	TW3106	2114	-4.250	-4.292	818	3.5	0
24	BL3102	NBG-18	-5.250	-5.286	820	3.5	0
23	DW3304	PCEA	-6.250	-6.278	822	3.5	0
22	EW3204	IG-110	-7.250	-7.269	824	3.4	0
21	AP3102	NBG-17	-8.250	-8.260	824	3.4	0
20	EW4608	IG-110	-8.875	-8.879	824	3.3	0
19	TW3109	2114	-9.500	-9.499	823	3.3	0
18	BW3502	NBG-18	-10.500	-10.494	822	3.2	0
17	DW3403	PCEA	-11.500	-11.487	819	3.1	0
16	EW3303	IG-110	-12.500	-12.480	817	3.0	0
15	TW3704	2114	-13.500	-13.474	814	2.9	0
14	BP3102	NBG-18	-14.500	-14.471	812	2.7	0
13	XA	Flux Monitor	-15.125	-15.094	810	2.7	0
12	DW3502	PCEA	-15.750	-15.716	809	2.6	0
11	TW3404	2114	-16.750	-16.711	808	2.4	0
10	EW3402	IG-110	-17.750	-17.707	808	2.2	0
9	BW3303	NBG-18	-18.750	-18.703	809	2.1	0
8	DW3602	PCEA	-19.750	-19.699	810	1.9	0
7	EW4609	IG-110	-20.375	-20.320	812	1.7	0
6	AP3204	NBG-17	-21.000	-20.942	815	1.6	0
5	TW3610	2114	-22.000	-21.940	823	1.4	0
4	325-14	GrafTech-325	-22.625	-22.564	832	1.3	0
3	200-14	GrafTech-200	-22.875	-22.814	836	1.2	0
2	AL3807	NBG-17	-23.125	-23.062	842	1.1	0

Table 6. AGC-3 loading order for Stack 4.

Loading Order	Specimen ID	Graphite Grade	Nominal Specimen Elevation (in)	End of Test Specimen Elevation (in)	Specimen Temperature (°C)	Specimen Dose (DPA)	Stack Load (lbf)
23	EW3601	IG-110	19.500	19.076	789	1.6	378
22	7A	Flux Monitor	18.875	18.459	775	1.7	378
21	TW3703	2114	18.250	17.838	763	1.8	378
20	DA3001	PCEA	17.250	16.846	751	2.0	378
19	BP3103	NBG-18	16.250	15.856	748	2.2	378
18	AL3202	NBG-17	15.250	14.866	753	2.3	378
17	EW3604	IG-110	14.250	13.878	763	2.5	378
16	1H	Flux Monitor	13.625	13.267	771	2.6	378
15	DW3801	PCEA	13.000	12.646	779	2.7	378
14	BL3103	NBG-18	12.000	11.663	793	2.8	378
13	TW3405	2114	11.000	10.677	805	2.9	378
12	AW3001	NBG-17	10.000	9.692	814	3.0	378
11	EA3001	IG-110	9.000	8.710	821	3.1	378
10	DA3203	PCEA	8.000	7.730	825	3.2	378
9	YA	Flux Monitor	7.375	7.127	826	3.3	378
8	BP3202	NBG-18	6.750	6.506	826	3.3	378
7	TW3408	2114	5.750	5.524	825	3.4	378
6	AP3304	NBG-17	4.750	4.540	822	3.4	378
5	EA3004	IG-110	3.750	3.561	819	3.5	378
4	DW3603	PCEA	2.750	2.583	816	3.5	378
3	1C	Flux Monitor	2.125	1.985	814	3.5	378
2	BP3301	NBG-18	1.500	1.364	813	3.5	378
35	AW3810	NBG-17	-1.375	-1.448	813	3.6	0
34	EW4601	IG-110	-1.625	-1.694	814	3.6	0
33	TW3821	2114	-1.875	-1.941	814	3.6	0
32	325-9	GrafTech-325	-2.125	-2.189	814	3.6	0
31	P3-07	PCIB	-2.375	-2.438	815	3.5	0
30	BP3304	NBG-18	-3.000	-3.059	816	3.5	0
29	R7	Flux Monitor	-3.625	-3.680	818	3.5	0
28	DW3702	PCEA	-4.250	-4.300	820	3.5	0
27	EA3103	IG-110	-5.250	-5.291	822	3.5	0
26	AP3403	NBG-17	-6.250	-6.282	824	3.4	0
25	TW3501	2114	-7.250	-7.275	824	3.4	0
24	BP3603	NBG-18	-8.250	-8.270	824	3.3	0
23	TW3904	2114	-8.875	-8.891	824	3.3	0
22	DA3302	PCEA	-9.500	-9.511	823	3.2	0
21	EA3202	IG-110	-10.500	-10.503	820	3.2	0
20	AW3004	NBG-17	-11.500	-11.495	817	3.1	0
19	TW3504	2114	-12.500	-12.489	815	3.0	0
18	BL3201	NBG-18	-13.500	-13.485	812	2.8	0
17	DW3803	PCEA	-14.500	-14.479	809	2.7	0
16	10	Flux Monitor	-15.125	-15.100	808	2.6	0
15	EW3403	IG-110	-15.750	-15.722	808	2.5	0
14	AL3301	NBG-17	-16.750	-16.716	807	2.4	0
13	BP3801	NBG-18	-17.750	-17.712	807	2.2	0
12	DA3403	PCEA	-18.750	-18.709	808	2.0	0
11	TW3507	2114	-19.750	-19.706	810	1.8	0
10	JA	Flux Monitor	-20.375	-20.329	812	1.7	0
9	EW3502	IG-110	-21.000	-20.952	815	1.6	0
8	BL3601	NBG-18	-21.625	-21.574	819	1.5	0
7	AL3801	NBG-17	-21.875	-21.822	822	1.4	0
6	EW4509	IG-110	-22.125	-22.069	824	1.3	0
5	AL3904	NBG-17	-22.375	-22.316	828	1.3	0
4	DW4511	PCEA	-22.625	-22.565	831	1.2	0
3	DA3510	PCEA	-22.875	-22.814	836	1.2	0
2	TW3827	2114	-23.125	-23.062	841	1.1	0

Table 7. AGC-3 loading order for Stack 5.

Loading Order	Specimen ID	Graphite Grade	Nominal Specimen Elevation (in)	End of Test Specimen Elevation (in)	Specimen Temperature (°C)	Specimen Dose (DPA)	Stack Load (lbf)
23	EW3602	IG-110	19.500	19.060	789	1.6	382
22	DA-SP1	PCEA	18.875	18.444	775	1.7	382
21	TW3702	2114	18.250	17.824	763	1.8	382
20	DA3003	PCEA	17.250	16.832	752	2.0	382
19	BP3104	NBG-18	16.250	15.843	749	2.2	382
18	AL3203	NBG-17	15.250	14.852	754	2.4	382
17	EW3701	IG-110	14.250	13.865	764	2.5	382
16	J7	Flux Monitor	13.625	13.254	771	2.6	382
15	DW3804	PCEA	13.000	12.633	780	2.7	382
14	BL3104	NBG-18	12.000	11.652	793	2.9	382
13	TW3406	2114	11.000	10.666	805	3.0	382
12	AW3002	NBG-17	10.000	9.680	815	3.1	382
11	EA3002	IG-110	9.000	8.700	822	3.2	382
10	DA3204	PCEA	8.000	7.720	826	3.3	382
9	DA-SP2	PCEA	7.375	7.116	828	3.3	382
8	BP3204	NBG-18	6.750	6.496	828	3.4	382
7	TW3409	2114	5.750	5.515	828	3.4	382
6	AP3401	NBG-17	4.750	4.532	826	3.5	382
5	EA3101	IG-110	3.750	3.556	824	3.5	382
4	DW3604	PCEA	2.750	2.580	821	3.6	382
3	O7	Flux Monitor	2.125	1.985	820	3.6	382
2	BP3302	NBG-18	1.500	1.364	819	3.6	382
35	AW3811	NBG-17	-1.375	-1.458	820	3.6	0
34	EW4602	IG-110	-1.625	-1.704	820	3.6	0
33	TW3822	2114	-1.875	-1.951	821	3.6	0
32	325-10	GrafTech-325	-2.125	-2.199	821	3.6	0
31	P3-08	PCIB	-2.375	-2.447	822	3.6	0
30	BP3601	NBG-18	-3.000	-3.068	823	3.6	0
29	DA3603	PCEA	-3.625	-3.688	825	3.6	0
28	DW3703	PCEA	-4.250	-4.306	826	3.6	0
27	EA3104	IG-110	-5.250	-5.296	828	3.5	0
26	AP3404	NBG-17	-6.250	-6.287	830	3.5	0
25	TW3502	2114	-7.250	-7.281	831	3.4	0
24	BP3604	NBG-18	-8.250	-8.275	830	3.4	0
23	DA3604	PCEA	-8.875	-8.895	829	3.3	0
22	DA3303	PCEA	-9.500	-9.513	828	3.3	0
21	EA3203	IG-110	-10.500	-10.504	826	3.2	0
20	AW3101	NBG-17	-11.500	-11.495	823	3.1	0
19	TW3505	2114	-12.500	-12.489	819	3.0	0
18	BL3202	NBG-18	-13.500	-13.485	816	2.9	0
17	DW3901	PCEA	-14.500	-14.479	813	2.7	0
16	Y2	Flux Monitor	-15.125	-15.100	812	2.7	0
15	EW3704	IG-110	-15.750	-15.721	811	2.6	0
14	AL3101	NBG-17	-16.750	-16.715	810	2.4	0
13	BP3702	NBG-18	-17.750	-17.710	810	2.2	0
12	DA3404	PCEA	-18.750	-18.706	810	2.1	0
11	TW3508	2114	-19.750	-19.704	812	1.9	0
10	DA3605	PCEA	-20.375	-20.327	813	1.7	0
9	EW3503	IG-110	-21.000	-20.950	816	1.6	0
8	BL3602	NBG-18	-21.625	-21.573	820	1.5	0
7	AL3802	NBG-17	-21.875	-21.821	822	1.4	0
6	EW4510	IG-110	-22.125	-22.068	825	1.4	0
5	AW3902	NBG-17	-22.375	-22.316	828	1.3	0
4	DW4512	PCEA	-22.625	-22.564	832	1.3	0
3	DA3511	PCEA	-22.875	-22.813	836	1.2	0
2	TW3828	2114	-23.125	-23.062	842	1.1	0

Table 8. AGC-3 loading order for Stack 6.

Loading Order	Specimen ID	Graphite Grade	Nominal Specimen Elevation (in)	End of Test Specimen Elevation (in)	Specimen Temperature (°C)	Specimen Dose (DPA)	Stack Load (lbf)
23	EW3603	IG-110	19.500	18.972	788	1.6	574
22	DA-SP3	PCEA	18.875	18.358	774	1.7	574
21	TW3602	2114	18.250	17.740	763	1.8	574
20	DA3004	PCEA	17.250	16.752	752	2.0	574
19	BP3201	NBG-18	16.250	15.767	750	2.2	574
18	AL3204	NBG-17	15.250	14.780	754	2.4	574
17	EW3702	IG-110	14.250	13.798	764	2.5	574
16	AU	Flux Monitor	13.625	13.192	771	2.6	574
15	DW3902	PCEA	13.000	12.573	779	2.7	574
14	BL3204	NBG-18	12.000	11.597	792	2.9	574
13	TW3407	2114	11.000	10.616	804	3.0	574
12	AW3003	NBG-17	10.000	9.636	814	3.1	574
11	EA3003	IG-110	9.000	8.661	821	3.2	574
10	DA3301	PCEA	8.000	7.686	825	3.3	574
9	DA-SP4	PCEA	7.375	7.090	827	3.3	574
8	BP3205	NBG-18	6.750	6.471	828	3.4	574
7	TW3410	2114	5.750	5.495	827	3.5	574
6	AP3402	NBG-17	4.750	4.517	826	3.5	574
5	EA3301	IG-110	3.750	3.547	824	3.6	574
4	DW3701	PCEA	2.750	2.578	822	3.6	574
3	ID	Flux Monitor	2.125	1.992	821	3.6	574
2	BP3303	NBG-18	1.500	1.372	820	3.6	574
35	AW3812	NBG-17	-1.375	-1.470	821	3.6	0
34	EW4603	IG-110	-1.625	-1.716	821	3.6	0
33	TW3823	2114	-1.875	-1.962	822	3.6	0
32	325-11	GrafTech-325	-2.125	-2.211	822	3.6	0
31	P3-09	PCIB	-2.375	-2.459	823	3.6	0
30	BP3602	NBG-18	-3.000	-3.080	824	3.6	0
29	DA3608	PCEA	-3.625	-3.699	825	3.6	0
28	DW3704	PCEA	-4.250	-4.317	827	3.6	0
27	EA3201	IG-110	-5.250	-5.306	829	3.6	0
26	AP3103	NBG-17	-6.250	-6.296	831	3.5	0
25	TW3503	2114	-7.250	-7.288	832	3.5	0
24	BP3605	NBG-18	-8.250	-8.282	832	3.4	0
23	DA3607	PCEA	-8.875	-8.901	831	3.3	0
22	DA3401	PCEA	-9.500	-9.520	831	3.3	0
21	EA3204	IG-110	-10.500	-10.511	829	3.2	0
20	AW3102	NBG-17	-11.500	-11.502	826	3.1	0
19	TW3506	2114	-12.500	-12.495	824	3.0	0
18	BL3203	NBG-18	-13.500	-13.490	821	2.9	0
17	DW3903	PCEA	-14.500	-14.482	819	2.8	0
16	AH	Flux Monitor	-15.125	-15.102	818	2.7	0
15	EW3501	IG-110	-15.750	-15.724	817	2.6	0
14	AL3103	NBG-17	-16.750	-16.717	815	2.4	0
13	BP3703	NBG-18	-17.750	-17.712	814	2.2	0
12	DA3104	PCEA	-18.750	-18.708	814	2.1	0
11	TW3509	2114	-19.750	-19.704	814	1.9	0
10	DA3606	PCEA	-20.375	-20.328	815	1.7	0
9	EW3504	IG-110	-21.000	-20.951	817	1.6	0
8	BL3603	NBG-18	-21.625	-21.574	821	1.5	0
7	AL3803	NBG-17	-21.875	-21.823	823	1.4	0
6	EW4511	IG-110	-22.125	-22.070	825	1.4	0
5	AW3903	NBG-17	-22.375	-22.317	828	1.3	0
4	DW4601	PCEA	-22.625	-22.565	832	1.3	0
3	DA3512	PCEA	-22.875	-22.814	836	1.2	0
2	TW3829	2114	-23.125	-23.062	841	1.1	0

Table 9a. AGC-3 loading order for Stack 7 (center channel).

Loading Order	Specimen ID	Graphite Grade	Nominal Specimen Elevation (in)	End of Test Specimen Elevation (in)	Specimen Temperature (°C)	Specimen Dose (DPA)
161	BP3401	NBG-18	18.375	18.337	937	1.8
160	AP3501	NBG-17	18.125	18.089	899	1.8
159	EA3504	IG-110	17.875	17.842	868	1.9
158	TW3801	2114	17.625	17.594	843	1.9
157	DA3501	PCEA	17.375	17.346	822	2.0
156	CAN101	HOPG	17.125	17.097	805	2.0
155	CAN129	MLRF	16.750	16.724	788	2.1
154	324-1	GrafTech-324	16.375	16.352	777	2.1
153	328-1	GrafTech-328	16.125	16.103	773	2.2
152	BW4002	NBG-18	15.875	15.856	771	2.2
151	AW3801	NBG-17	15.625	15.608	771	2.3
150	EW4501	IG-110	15.375	15.362	773	2.3
149	TW3802	2114	15.125	15.114	775	2.4
148	DW4502	PCEA	14.875	14.866	779	2.4
147	CAN102	HOPG	14.625	14.618	783	2.4
146	A	SGL-SiC	14.375	14.368	788	2.5
145	S1	SGL	14.125	14.119	793	2.5
144	325-1	GrafTech-325	13.875	13.871	799	2.6
143	200-1	GrafTech-200	13.625	13.621	804	2.6
142	BP3502	NBG-18	13.375	13.373	810	2.6
141	AP3502	NBG-17	13.125	13.125	816	2.7
140	EA3505	IG-110	12.875	12.878	821	2.7
139	TW3803	2114	12.625	12.630	827	2.7
138	DA3502	PCEA	12.375	12.382	832	2.8
137	CAN103	HOPG	12.125	12.133	837	2.8
136	CAN121	MLRF	11.750	11.760	843	2.9
135	324-2	GrafTech-324	11.375	11.387	849	2.9
134	328-2	GrafTech-328	11.125	11.139	853	2.9
133	BW4003	NBG-18	10.875	10.891	856	3.0
132	AW3802	NBG-17	10.625	10.643	859	3.0
131	EW4502	IG-110	10.375	10.397	861	3.0
130	TW3804	2114	10.125	10.150	864	3.0
129	DW4503	PCEA	9.875	9.903	866	3.1
128	CAN104	HOPG	9.625	9.655	867	3.1
127	B	SGL-SiC	9.375	9.405	869	3.1
126	S2	SGL	9.125	9.155	870	3.1
125	325-2	GrafTech-325	8.875	8.907	870	3.2
124	200-2	GrafTech-200	8.625	8.658	871	3.2
123	BP3403	NBG-18	8.375	8.411	872	3.2
122	AP3503	NBG-17	8.125	8.164	872	3.2
121	EA3506	IG-110	7.875	7.917	872	3.3
120	TW3805	2114	7.625	7.670	872	3.3
119	DA3503	PCEA	7.375	7.423	872	3.3
118	CAN105	HOPG	7.125	7.175	871	3.3
117	CAN122	MLRF	6.750	6.803	871	3.3
116	324-3	GrafTech-324	6.375	6.429	870	3.4
115	328-3	GrafTech-328	6.125	6.181	870	3.4
114	BW4004	NBG-18	5.875	5.933	869	3.4
113	AW3803	NBG-17	5.625	5.685	869	3.4
112	EW4503	IG-110	5.375	5.440	869	3.4
111	TW3806	2114	5.125	5.193	868	3.5
110	DW4504	PCEA	4.875	4.946	868	3.5
109	CAN106	HOPG	4.625	4.699	867	3.5
108	C	SGL-SiC	4.375	4.449	867	3.5
107	S3	SGL	4.125	4.200	867	3.5
106	325-3	GrafTech-325	3.875	3.952	866	3.5
105	200-3	GrafTech-200	3.625	3.704	866	3.5
104	BP3404	NBG-18	3.375	3.456	866	3.5
103	AP3504	NBG-17	3.125	3.209	866	3.5
102	EA3507	IG-110	2.875	2.963	866	3.5

Table 9b. AGC-3 loading order for Stack 7 (center channel).

Loading Order	Specimen ID	Graphite Grade	Nominal Specimen Elevation (in)	End of Test Specimen Elevation (in)	Specimen Temperature (°C)	Specimen Dose (DPA)
101	TW3807	2114	2.625	2.717	866	3.6
100	DA3504	PCEA	2.375	2.469	866	3.6
99	CAN107	HOPG	2.125	2.222	866	3.6
98	CAN123	MLRF	1.750	1.849	866	3.6
97	324-4	GrafTech-324	1.375	1.476	867	3.6
96	328-4	GrafTech-328	1.125	1.228	867	3.6
95	BW4005	NBG-18	0.875	0.980	867	3.6
94	AW3804	NBG-17	0.625	0.732	868	3.6
93	EW4504	IG-110	0.375	0.487	868	3.6
92	TW3808	2114	0.125	0.241	868	3.6
91	DW4505	PCEA	-0.125	-0.006	869	3.6
90	CAN108	HOPG	-0.375	-0.253	869	3.6
89	D	SGL-SiC	-0.625	-0.503	869	3.6
88	S4	SGL	-0.875	-0.751	870	3.6
87	325-4	GrafTech-325	-1.125	-0.999	870	3.6
86	200-4	GrafTech-200	-1.375	-1.247	871	3.6
85	BP3405	NBG-18	-1.625	-1.494	871	3.6
84	AP3505	NBG-17	-1.875	-1.740	871	3.6
83	EA3508	IG-110	-2.125	-1.986	872	3.6
82	TW3809	2114	-2.375	-2.233	872	3.6
81	DA3505	PCEA	-2.625	-2.480	872	3.6
80	CAN109	HOPG	-2.875	-2.728	872	3.6
79	CAN124	MLRF	-3.250	-3.100	872	3.6
78	324-5	GrafTech-324	-3.625	-3.473	873	3.6
77	328-5	GrafTech-328	-3.875	-3.722	873	3.6
76	BW4006	NBG-18	-4.125	-3.970	873	3.6
75	AW3805	NBG-17	-4.375	-4.218	873	3.6
74	EW4505	IG-110	-4.625	-4.464	872	3.5
73	TW3810	2114	-4.875	-4.710	872	3.5
72	DW4506	PCEA	-5.125	-4.957	872	3.5
71	CAN110	HOPG	-5.375	-5.204	872	3.5
70	E	SGL-SiC	-5.625	-5.454	871	3.5
69	S5	SGL	-5.875	-5.703	871	3.5
68	325-5	GrafTech-325	-6.125	-5.951	871	3.5
67	200-5	GrafTech-200	-6.375	-6.199	870	3.5
66	BP3406	NBG-18	-6.625	-6.447	870	3.5
65	AP3506	NBG-17	-6.875	-6.694	869	3.5
64	EA3509	IG-110	-7.125	-6.940	869	3.4
63	TW3811	2114	-7.375	-7.187	868	3.4
62	DA3506	PCEA	-7.625	-7.435	868	3.4
61	CAN111	HOPG	-7.875	-7.682	867	3.4
60	CAN125	MLRF	-8.250	-8.055	866	3.4
59	324-6	GrafTech-324	-8.625	-8.429	865	3.3
58	328-6	GrafTech-328	-8.875	-8.678	864	3.3
57	BW4007	NBG-18	-9.125	-8.926	863	3.3
56	AW3806	NBG-17	-9.375	-9.173	862	3.3
55	EW4506	IG-110	-9.625	-9.419	862	3.3
54	TW3812	2114	-9.875	-9.666	861	3.3
53	DW4507	PCEA	-10.125	-9.913	860	3.2
52	CAN112	HOPG	-10.375	-10.160	859	3.2
51	F	SGL-SiC	-10.625	-10.410	858	3.2
50	S6	SGL	-10.875	-10.659	858	3.2
49	325-6	GrafTech-325	-11.125	-10.907	857	3.1
48	200-6	GrafTech-200	-11.375	-11.155	856	3.1
47	BP3407	NBG-18	-11.625	-11.403	855	3.1
46	AP3507	NBG-17	-11.875	-11.650	854	3.1
45	EA3510	IG-110	-12.125	-11.897	853	3.1
44	TW3813	2114	-12.375	-12.144	853	3.0
43	DA3507	PCEA	-12.625	-12.391	852	3.0
42	CAN113	HOPG	-12.875	-12.639	851	3.0

Table 9c. AGC-3 loading order for Stack 7 (center channel).

Loading Order	Specimen ID	Graphite Grade	Nominal Specimen Elevation (in)	End of Test Specimen Elevation (in)	Specimen Temperature (°C)	Specimen Dose (DPA)
41	CAN126	MLRF	-13.250	-13.012	850	2.9
40	324-7	GrafTech-324	-13.625	-13.385	849	2.9
39	328-7	GrafTech-328	-13.875	-13.633	848	2.9
38	BW4101	NBG-18	-14.125	-13.882	848	2.8
37	AW3808	NBG-17	-14.375	-14.129	847	2.8
36	EW4507	IG-110	-14.625	-14.375	846	2.8
35	TW3814	2114	-14.875	-14.622	846	2.7
34	DW4508	PCEA	-15.125	-14.870	845	2.7
33	CAN114	HOPG	-15.375	-15.118	845	2.7
32	G	SGL-SiC	-15.625	-15.367	844	2.6
31	S7	SGL	-15.875	-15.616	844	2.6
30	325-7	GrafTech-325	-16.125	-15.864	844	2.5
29	200-7	GrafTech-200	-16.375	-16.112	843	2.5
28	BP3408	NBG-18	-16.625	-16.361	843	2.5
27	AP3508	NBG-17	-16.875	-16.609	843	2.4
26	EA3511	IG-110	-17.125	-16.856	843	2.4
25	TW3815	2114	-17.375	-17.103	842	2.3
24	DA3508	PCEA	-17.625	-17.350	842	2.3
23	CAN115	HOPG	-17.875	-17.597	842	2.3
22	CAN127	MLRF	-18.250	-17.970	842	2.2
21	324-8	GrafTech-324	-18.625	-18.343	843	2.1
20	328-8	GrafTech-328	-18.875	-18.592	843	2.1
19	BW4102	NBG-18	-19.125	-18.841	843	2.0
18	AW3809	NBG-17	-19.375	-19.089	843	2.0
17	EW4508	IG-110	-19.625	-19.336	844	1.9
16	TW3816	2114	-19.875	-19.583	844	1.9
15	DW4509	PCEA	-20.125	-19.831	844	1.8
14	CAN116	HOPG	-20.375	-20.080	845	1.8
13	H	SGL-SiC	-20.625	-20.329	845	1.7
12	S8	SGL	-20.875	-20.579	846	1.7
11	325-8	GrafTech-325	-21.125	-20.829	846	1.6
10	200-8	GrafTech-200	-21.375	-21.077	847	1.6
9	BP3409	NBG-18	-21.625	-21.326	847	1.5
8	AP3509	NBG-17	-21.875	-21.575	847	1.5
7	EA3512	IG-110	-22.125	-21.823	847	1.4
6	TW3817	2114	-22.375	-22.071	847	1.4
5	DA3509	PCEA	-22.625	-22.319	847	1.3
4	CAN117	HOPG	-22.875	-22.568	846	1.3
3	CAN128	MLRF	-23.250	-22.941	845	1.2
2	324-9	GrafTech-324	-23.625	-23.314	842	1.1
1	328-9	GrafTech-328	-23.875	-23.562	840	1.0

Following irradiation in the ATR at INL, the AGC-3 capsule was disassembled.²⁰ All specimens recovered from disassembly were visually inspected and physically measured within the INL Carbon Characterization Laboratory before being stored in the irradiated graphite storage vault. It should be noted that NBG-18 specimens BP3302 and BL3103, and 2114 specimen TW3405 were lost (missing) during AGC-3 disassembly activities. After accounting for all recovered specimens from the AGC-3 capsule, PIE and testing were performed for each specimen at the INL Carbon Characterization Laboratory.²¹

4. AGC-3 AS-RUN IRRADIATION CONDITIONS

AGC-3 was designed to provide irradiation conditions similar to the 600°C graphite irradiations (i.e., AGC-1 and AGC-2) with similar graphite grades, the same applied mechanical stresses, and similar He-Ar gas environment, but irradiated at 800°C. AGC-3 is also the lower dose 800°C capsule (0–3.5 dpa), requiring a shorter irradiation time within ATR. AGC-4 is designated as the higher dose 800°C capsule

(3.5–7.0 dpa), requiring an irradiation period in ATR twice as long. An additional objective was to implement capsule design improvements learned from AGC-1 and AGC-2 to reduce the large specimen temperature range experienced in AGC-1.

4.1 AGC-3 Irradiation

AGC-3 was irradiated in the south flux trap of ATR between November 28, 2012, and April 12, 2014.¹⁵ AGC-3 was irradiated over four reactor cycles, Cycles 152B, 154B, 155A, and 155B (the AGC-3 capsule was not in ATR for Cycles 153A/B and 154A), for a total of 4374 MW days or approximately 209.5 effective full power days.

As discussed in Section 2, during irradiation, the graphite specimen length, as well as the graphite specimen holder, will decrease (shrink) under neutron exposure. The received dose in ATR is calculated as a function of the mid-plane elevation within the reactor, where specimens located in the middle of the reactor receive the highest fluence/dose levels and the specimen located at the top and bottom of the reactor receiving the lowest fluence/dose levels. To account for the change in specimen elevation during irradiation (i.e., the entire specimen column moves toward the bottom of the reactor due to graphite irradiation dimensional change) the position of each specimen at the beginning and end of each irradiation cycle was calculated based upon the individual sample dimensional change and the capsule linear variable-differential Transducer (LDVT) readouts.²² Once the position changes were calculated, the final specimen dose levels were determined from the fluence as calculated by the mid-plane elevation for each irradiation cycle. The total AGC-3 specimen dose ranged from 0.9–3.7 dpa, with the specimens at the mid-plane elevation receiving the highest accumulated dose levels.²³

4.2 AGC-3 Mechanical Loading Issues

As discussed in Section 2, to induce irradiation creep strain within the AGC-3 creep specimens, half of the specimens were subjected to compressive mechanical stresses while the remainder of the specimens had no applied stress. Stressed specimens were initially subjected to three different levels: a nominal stress of 13.8 MPa for specimens in Stacks 1 and 4, 17.2 MPa for Stacks 2 and 5, and the highest nominal stress of 20.7 MPa for the 12.7-mm-diameter specimens in Stacks 3 and 6.

However, the AGC-3 capsule encountered a high gas leak rate (5.6 L/min system leak rate versus the average rate of 1.0 L/min for the AGC-1 and AGC-2 experiments) in the compressive load control system, with its significant increase attributed primarily to the portion of the system providing the load on Stack 4. Therefore, a compressive load was not placed on Stack 4 until the source and future consequences of the leak were evaluated. In addition, a compressive load was not placed on Stack 1 (diametrically opposite and load pair companion to Stack 4) to prevent eccentrically loading the graphite specimen holder, which could possibly result in damaging the holder. The compressive loads on Stacks 2 and 5 were reduced from 17.3 to 13.8 MPa on December 5, 2012, to maximize the range of stack loading from the experiment if Stacks 1 and 4 were not able to have compressive loads.

Because compressive loads must remain constant throughout the irradiation, there were two major concerns: (1) the leak rate might increase to the point that the load could not be maintained, and (2) sufficient helium might not be obtainable to overcome the high leak rate due to a national shortage of helium at the time. Data from the inlet and outlet system flow meters indicated that the source of the leak was internal to the test train, most probably within the pneumatic ram for Stack 4. After discussions with the vendor and review of data taken during preassembly testing of the pneumatic rams, it was decided to test the system to determine the stability of the leak rate. The desired loads were imposed on Stacks 1 and 4 on Tuesday, December 11, 2012, and the leak rate was monitored until Monday, December 17, 2012. The leak rate was extremely stable, with essentially no perceptible change over the 6-day period. This result indicated that the leak was most likely located in a metallic component (such as a fitting) and

should remain very stable throughout the irradiation. Sufficient helium was located to support maintaining the load on the stacks for the duration of Cycle 152B. The compression gas was switched from helium to argon at the end of Cycle 152B. With the gas shortage issue resolved, and the determination that the leak rate was stable, it was concluded that loads could successfully be applied to Stacks 1 and 4 for the duration of the AGC-3 experiment.

A compressive load of 13.8 MPa was then applied to Stacks 1 and 4 for the duration of the experiment. Stacks 2 and 5 remained at 13.8 MPa for the duration of the experiment so that the load applied to these stacks would be constant. Therefore, four stacks were loaded to 13.8 MPa for the experiment, and two stacks were loaded to 20.7 MPa (Stacks 3 and 6). Because Stacks 2 and 5 were kept at 13.8 MPa for the duration of the experiment, no AGC-3 specimen was loaded to 17.2 MPa.

Stress level changes resulting from specimen lateral shrinkage or expansion during irradiation were ignored.²⁴ The steady-state applied stress levels over all irradiation cycles is reported in Table 7. The coefficient of variation of applied stress to all stacks (the extent of variability to the mean of all applied stresses) is ~5% across the entire AGC-3 irradiation.

Table 10. Stress values for each specimen stack.

	Stack 1	Stack 2	Stack 3	Stack 4	Stack 5	Stack 6
Average (MPa)	13.3	13.4	19.9	13.3	13.4	20
2 * standard deviation (MPa)	1.1	1.1	1.2	1.3	1.1	1.0
Coefficient of variance (%)	4.0	4.2	3.0	5.0	4.0	2.9

4.3 AGC-3 Temperature

Modifications to the AGC-3 capsule design significantly reduced the large axial temperature variation across the axial length of the AGC-1 capsule.¹⁶ A sophisticated capsule temperature control system was employed to allow all specimens to be irradiated as close to 800°C as possible.¹⁷ The AGC-3 specimen temperatures ranged from 748–918°C,²⁵ which was significantly better than even the AGC-2 specimens experienced (with a range of 437–707°C).²⁶ The optimal range as defined for the AGC-3 capsule irradiation specifications were for the average capsule temperature to be maintained at 800°C ±50°C.⁵ While this range was achieved in the outside Stacks 1–6, where the temperatures varied from 748–843°C, the center stack ranged from 771–918°C due to the much-lower temperatures experienced at the ends of the capsule where the flux is lowest. A detailed uncertainty analysis of the temperature model used to derive the specimen temperatures yields a maximum uncertainty of ±50°C for the individual AGC-3 specimen temperatures.

5. CREEP STRAIN DATA

Dimensional change (strain) data for all major graphite grades are provided in INL’s “AGC-3 Specimen Post-Irradiation Examination Data Package Report.”⁴ The total specimen irradiation dose, average irradiation temperature, and applied mechanical load during irradiation for each specimen are recorded, along with dimensional change measurements. Total volumetric, length, and diametric dimensional AGC-3 specimen changes are summarized in Figure 5 through Figure 7 for the control specimens and two applied mechanical load states. It should be noted that volume, length, and diameter changes are strongly related to irradiation temperature and received dose, as will be discussed further in later sections. These figures, demonstrating dimensional change effects from the applied mechanical load, are meant as a simple comparison of the mean values of the total dimensional and volume variables between grades.

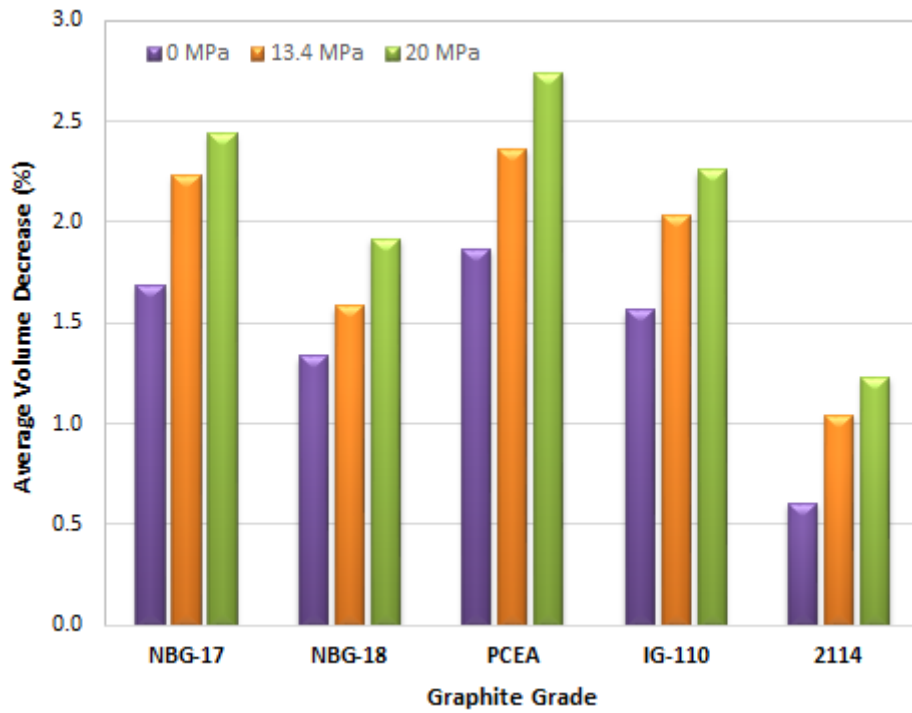


Figure 5. Total maximum volume decrease (%) due to irradiation creep for five major grades of graphite. The dimensional change dependency on irradiation dose and irradiation temperature are not presented.

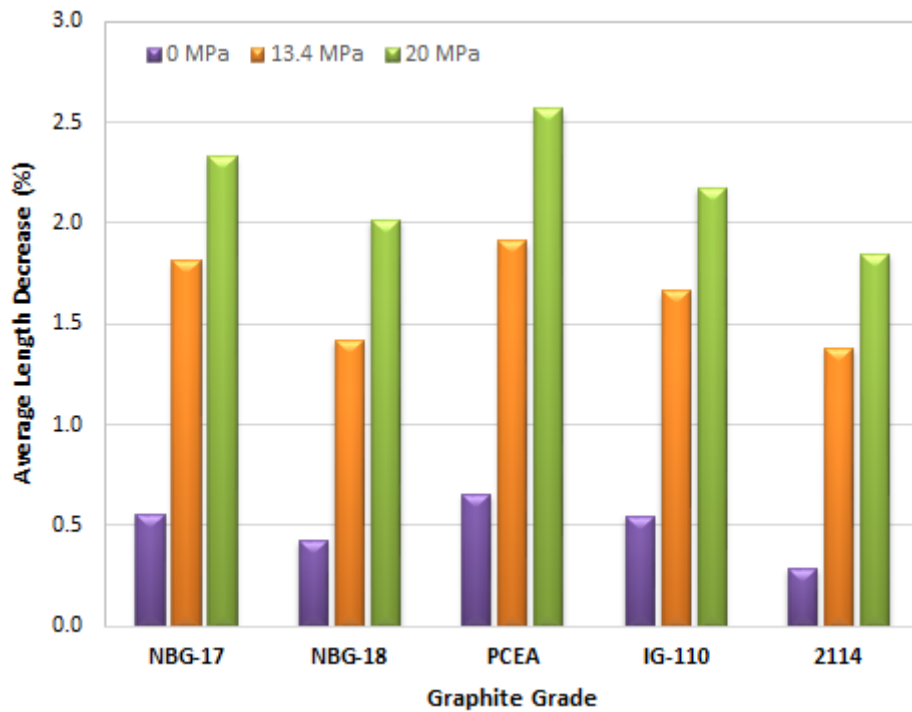


Figure 6. Average length decrease (%) at maximum dose received for five major grades of graphite. The dimensional change dependency on irradiation dose and irradiation temperature are not presented.

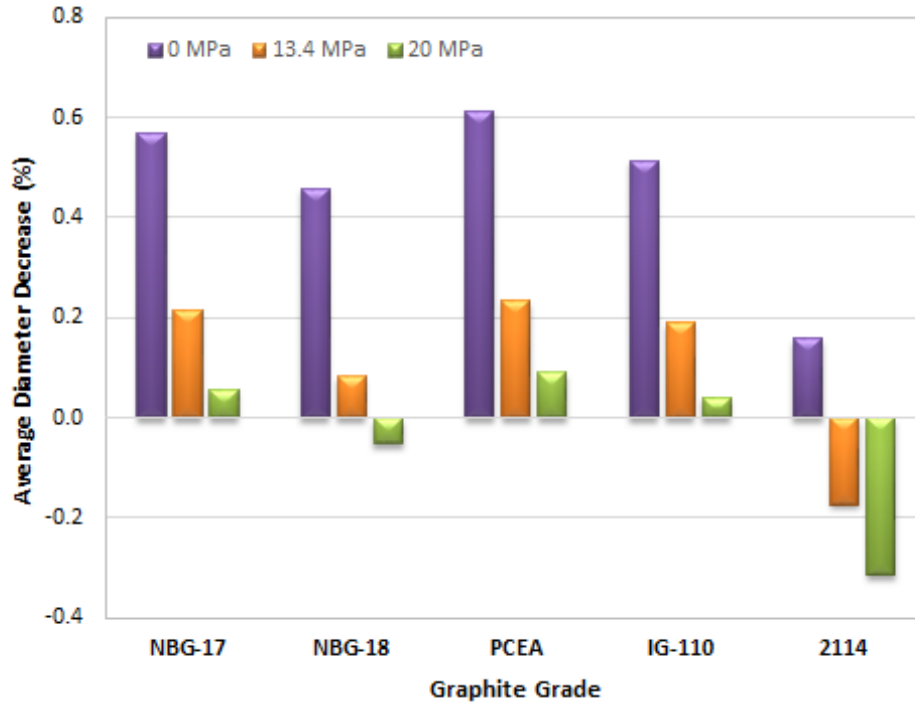


Figure 7. Total maximum diameter decrease (%) due to irradiation creep for five major grades of graphite. Note the diameter decrease in stressed (creep) samples was lower than unstressed (control) samples due to “barreling” effects mitigating the shrinkage (note: barreling produced a variety of out-of-round shapes, including hourglass, pear, and the simple barrel shape). The dimensional change dependency on irradiation dose and irradiation temperature are not presented.

The underlying mechanisms responsible for irradiation-induced creep in graphite are still relatively unknown. Traditionally, irradiation creep of graphite is defined as the macroscopic dimensional change in a specimen occurring under the simultaneous influence of neutron irradiation and mechanical stresses. Stresses sufficiently large to cause irradiation creep are induced in the graphite microstructure by large temperature and/or neutron flux gradients through the large reactor components. Irradiation creep is fluence (i.e., time in reactor) dependent at temperatures well below where thermal creep would normally be observed in a graphite specimen.

Conventionally, irradiation induced creep has been studied experimentally in material test reactors on small graphite specimens kept at a constant mechanical load. The apparent creep strain is determined by comparing the irradiation-induced dimensional changes (strains) in specimens irradiated under identical conditions, but where one specimen (i.e., the creep specimen) is mechanically loaded and the other specimen (the control specimen) is not. The difference between the dimensional change of these matched-pair creep and control specimen yields the creep strain. While all graphite specimens will experience dimensional change, the mechanically stressed specimens will experience larger strain values than the matching unstressed specimens, yielding a value for the irradiation induced creep strain.

Creep experiments of this nature, with both tensile and compressive applied stresses, have been performed in previous studies across a relatively large neutron dose range. Generally, three stages of graphite irradiation creep are recognized (Figure 8):

- *Stage I:* In the first stage of creep, initial strain accumulates rapidly with dose but quickly saturates to a strain level of approximately one elastic strain unit defined as $\sigma_{\text{(applied)}}/E_0$, where $\sigma_{\text{(applied)}}$ is the

applied mechanical stress to the specimen and E_0 is the unirradiated Young's modulus value of the stressed specimen. Stage I creep is referred to as "primary creep" and occurs at very low dose level, <1 dpa.

- *Stage II*: In the second stage, accumulation of creep strain that is linearly proportional to the neutron dose and applied stress. Stage II creep is referred to as "steady-state" or "linear" creep. The slope of a plot of second-stage or secondary creep strain should be a straight line with slope K , the creep coefficient. Typically, this stage is dominant between 1 and 10 dpa for most graphite grades, but the actual dose range over which secondary creep occurs is temperature and graphite grade dependent. Previous studies have demonstrated that Stage II creep (linear accumulation of strain) stops when dimensional volume change Turnaround has been achieved.
- *Stage III*: The third and final stage is a nonlinear acceleration of accumulated creep strain that usual occurs after dimensional volume-change Turnaround. Stage III occurs at higher doses, typically >10 dpa, where the rate of accumulation of irradiation creep strain accelerates with dose.

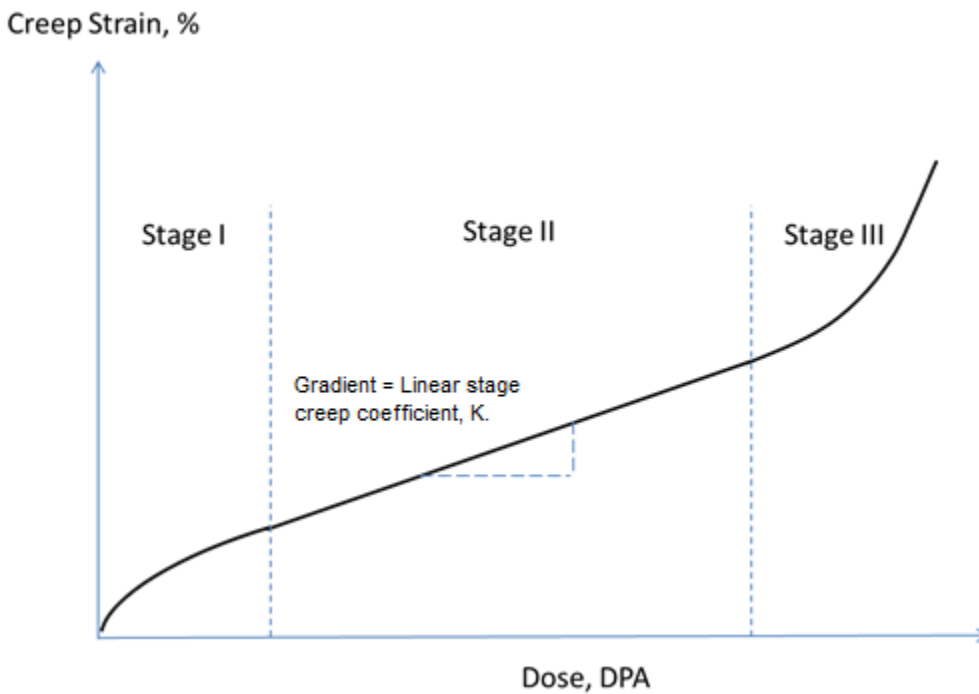


Figure 8. A schematic representation of the three stages of irradiation-induced creep.

Creep during Stages I and II are thought to be dominated by dislocation formation and flow processes (i.e., primarily in-crystal effects only). Whereas Stage III creep (which occurs at higher doses, after the point of volume change turnaround) is thought to be related to graphite structure changes (i.e., both in-crystal and pore volume effects). Additional irradiation-induced creep strain data are required for the development of a more complete understanding of the underlying mechanisms responsible for graphite creep.

The AGC-3 experiment was designed to operate in Stage I and II, where dose and irradiation temperature produce a linear accumulation of creep strain. Previous studies have developed a viscoelastic creep law that applies to this linear stage of creep (Figure 8).^{27,28,29,30} Under the linear viscoelastic creep law, the normalized secondary creep data are shown to be linear, indicating no structure effects at these (lower) doses. This report uses this linear relationship to obtain the total creep strain for the AGC-3 specimens with the following relationship:

$$\varepsilon_{c(TOTAL)} = \varepsilon_{cPRIMARY} + \varepsilon_{cSECONDARY} (\%) \quad (1)$$

$$\varepsilon_{cPRIMARY} = \frac{A\sigma}{E_0} [1 - \text{Exp}(-b\gamma)] \approx \frac{\sigma}{E_0} (\%)$$

$$\varepsilon_{cSECONDARY} = K\sigma\gamma (\%)$$

$$\varepsilon_{c(TOTAL)} = \frac{\sigma}{E_0} + K\sigma\gamma (\%) \quad (2)$$

where

K = secondary creep coefficient in % change/(dpa·MPa) or 10⁻³⁰cm²/(n·Pa)

σ = applied stress (MPa)

γ = neutron dose (dpa)

E₀ = initial (pre-irradiation) Young's modulus of stressed specimen with A and b as fitting constants to the primary creep equation.

Applying this viscoelastic creep law relationship to the AGC-3 data allows the creep coefficient, K, to be calculated from the measured dimensional change data for each graphite grade. The methodology and calculations for calculating the creep coefficient are described in the following section.

6. CREEP STRAIN ANALYSIS METHODOLOGY

The exceptionally large dataset of irradiation strain measurements from the AGC experiment allows for a more sophisticated analysis than has been performed from historical studies which have a limited dataset resulting from the more restricted matched-pairs experiments. Rather than use individual matched pair stressed vs. unstressed comparisons to analyze creep strain data, as was done in AGC-1 and AGC-2, the creep strain of the five major grades of graphite in AGC-3 was analyzed by plotting the length dimensional change for the stressed and unstressed specimens as a function of irradiation dose and graphite grade. Least-square linear regressions are then performed through each group of data. The unstressed regression is subtracted from the stressed to obtain the true creep strain. Applying the linear viscoelastic creep law, discussed above, the creep coefficient, K, is determined.

6.1 Methodology Steps

Unlike AGC-1 and AGC-2, the creep coefficients from the AGC-3 capsule were calculated using a linear regression method instead of using matched specimen pairs. Below is an outline of the process used to calculate the creep coefficients for each graphite grade.

1. Plot the percent change in specimen length versus specimen dose for each of the unloaded specimens.
2. Using least squares, fit a linear regression line through the un-stressed specimen percent length change data while forcing the y-intercept through zero. The description of this fit line is given by the equation:

$$y = m_{UL}\gamma, \text{ where } m_{UL} \text{ is the slope of the fit line through the un-stressed data and } \gamma \text{ is the dose.}$$

3. Plot the percent change in specimen length versus specimen dose for each of the loaded specimens.

4. Using least squares, fit a linear regression line through the stressed specimen percent length change data while forcing the y-intercept through zero. The description of this fit line is given by the equation:

$y = m_L \gamma$, where m_L is the slope of the fit line through the stressed data and γ is the dose.

5. Substitute equation (2) into equation (1) :

$$\varepsilon_{c(TOTAL)} = m_L \gamma - m_{UL} \gamma = \frac{\sigma}{E_0} + K \sigma \gamma$$

6. Solve for the creep coefficient K.

$$K = \frac{m_L - m_{UL}}{\sigma} - \frac{1}{E_0 \gamma}$$

A new LabVIEW-derived application (Graphite Analysis Tool, GAT) was developed to implement the creep strain calculations. This application uses data that were directly exported from the INL's Nuclear Data Management and Analysis System and the methodology outlined above. It generates data plots and creep coefficient calculations for a user-selectable graphite grade. The application also includes data filtering that can be used to limit the specimens considered in the calculation to those within a specified temperature and/or dose range. Once generated, these data can be exported to a Microsoft Excel file format.

Results from the GAT calculations were independently validated by manually performing creep strain calculations through an independent reviewer. The results of this acceptance testing and the application's software management plan are documented using INL Form 562.41, "Software Management Plan and Life Cycle Documentation for Research and Development Activities."³¹

7. DIMENSIONAL CHANGE ANALYSIS

Before creep strain results are calculated, it is useful to investigate the dimensional change behavior of major AGC-3 graphite grades. Dimensional changes (longitudinal, lateral, and volumetric) can provide insights into the isotropic material response, diametral/longitudinal irradiation response, and volumetric conservation of the grades after being irradiated.

Figure 9 shows a compilation of the longitudinal dimensional change as a function of irradiation dose for all the major graphite grades contained in AGC-3. As expected, the dimensional change for the group of stressed specimens is greater than that of the unstressed group. This is expected due to the stressed specimens not only responding to irradiation shrinkage but also mechanically induced creep. Also, both stressed and unstressed specimen dimensional change as a function of irradiation dose appears to be linear.

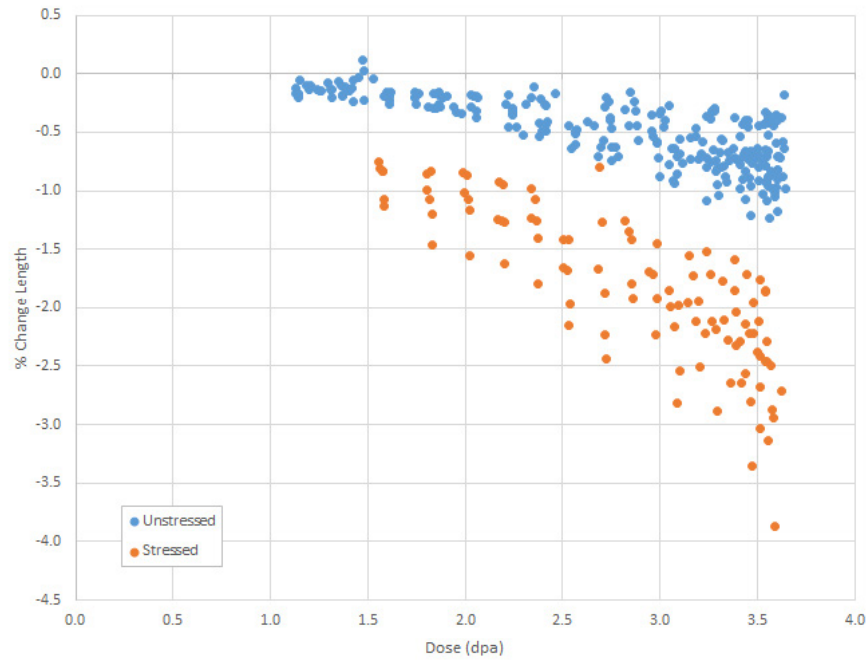


Figure 9. Dimensional change of all AGC-3 major grades of graphite as a function of dose. Data includes creep, control, and button piggyback measurements.

7.1 Dimensional Change Analysis by Graphite Grade

After establishing the validity of the AGC experimental parameters it is instructional to analyze the dimensional irradiation response for all major grades of graphite in AGC-3. The dimensional change behavior can provide insights into the isotropic material response, diametral and/or longitudinal irradiation response, or whether a grade is approaching turnaround, as well as other behaviors.

The dimensional changes over the AGC-3 dose range for all major AGC-3 graphite grades are summarized from pre- and post-irradiation specimen measurements. Plotting dimensional behavior of the unstressed control specimen length and diameter changes illustrates the isotropic irradiation response of the graphite. Because no additional mechanical stresses are involved, it can be assumed that the irradiation-induced stress within the graphite specimens is isostatic, creating similar dimensional change behavior in both lateral and longitudinal directions. The dimensional changes for the major grades in AGC-3 are shown in Figure 10, Figure 11, and Figure 12.

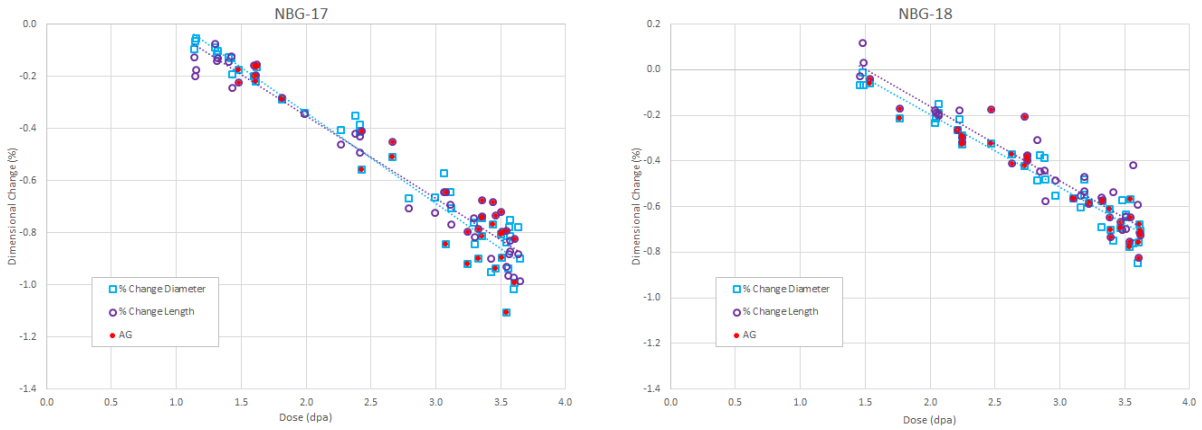


Figure 10. Comparison of dimensional change in longitudinal and diametral directions for vibrational molded graphite grades NBG-17 and NBG-18 unstressed control specimens. Hollow points are taken from the billet in the WG direction, and the red data points indicate specimens taken in the AG direction.

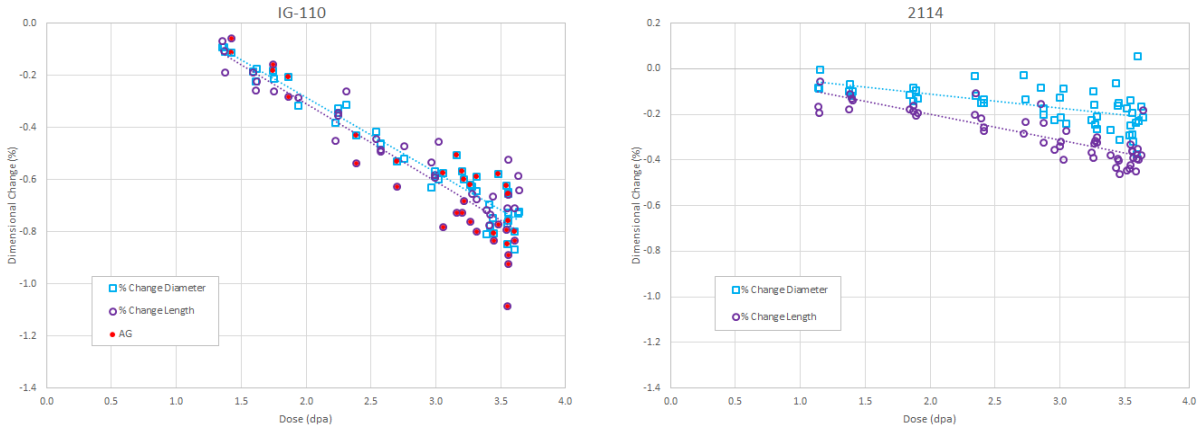


Figure 11. Comparison of dimensional change in longitudinal and diametral directions for isostatically molded graphite grades IG-110 and 2114 unstressed control specimens. Red data points indicate AG specimens. Hollow points are taken from the billet in the WG direction and the red data points indicate specimens taken in the AG direction.

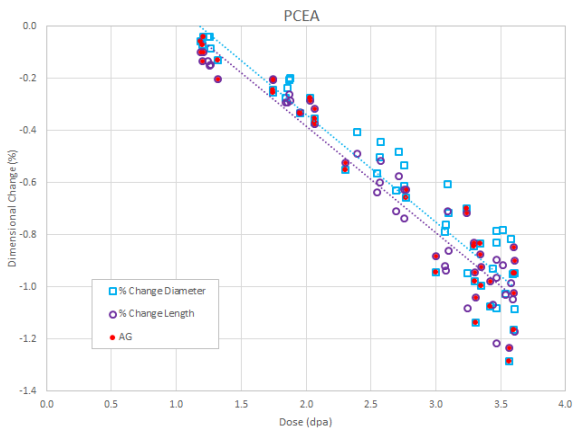


Figure 12. Dimensional change in longitudinal and diametral directions for unstressed control specimens of extruded graphite grade PCEA. Hollow data points indicate specimens taken from the billet in the WG direction, and red-filled points indicate AG specimens.

Nuclear grade isotropic graphite should have nearly identical dimensional change in all directions. The five major grades of graphite in AGC-3 do demonstrate isotropic or near-isotropic response over the AGC-3 dose and temperature range. NBG-17 demonstrates the highest isotropic response (i.e., lowest variation between length and diameter change) while the 2114 had the least isotropic behavior, with diametral change seen to be lower than axial length changes.

It should be noted that AG and WG specimens had a nearly identical response, again demonstrating near-isotropic response for those major graphite grades.

Any change in isotropy from irradiation, as determined through changes in Poisson's ratio, will be investigated in a future AGC-3 irradiated material analysis. These data trends will be compared to the results from Poisson's ratio changes.

7.2 Volume Change Analysis by Graphite Grade

The volume change behavior for each graphite grade is calculated from the specimen pre-irradiation examination and PIE dimensional measurements and summarized in Figure 13, Figure 14, and Figure 15. All grades exhibited linear volumetric change, with the PCEA showing the largest decrease. With exception of 2114, all of the other grades had fairly similar levels of volumetric decrease.

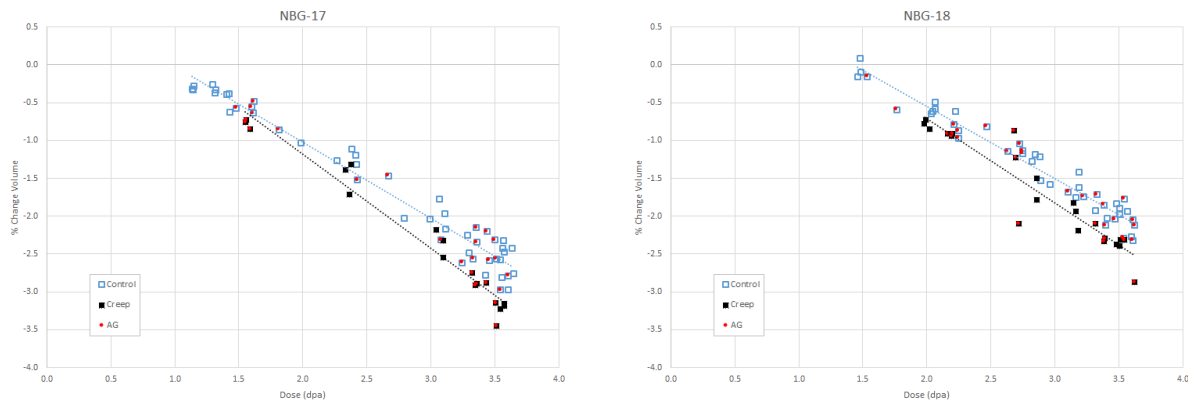


Figure 13. Volume changes of vibrational molded graphite grades NBG-17 and NBG-18 for creep and control specimens. Red data points indicate specimens taken from the billet in the AG direction all others are WG.

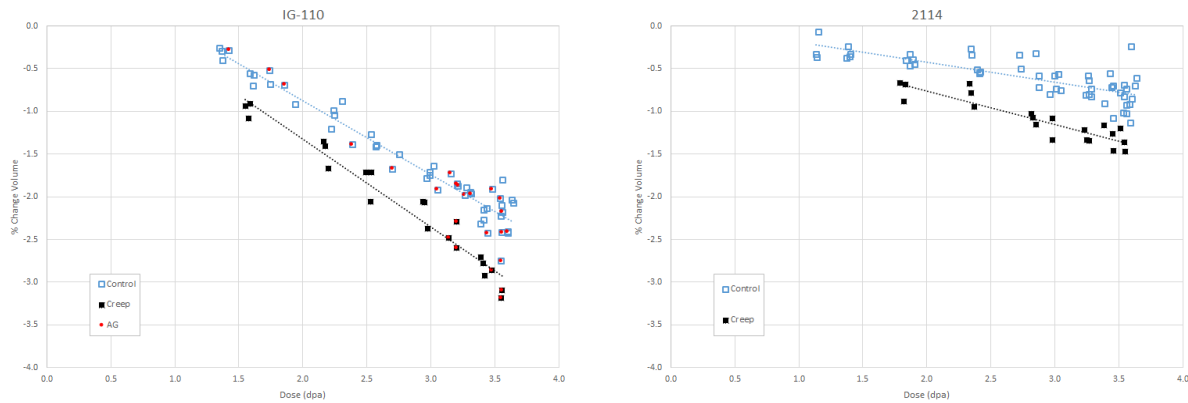


Figure 14. Volume change of isotastically molded graphite grades IG-110 and 2114 for both creep and control specimens. Red data points indicate specimens taken from the billet in the AG direction all others are WG.

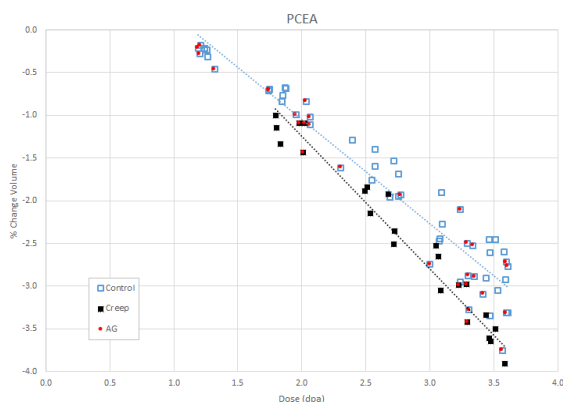


Figure 15. Volume change of extruded graphite grade PCEA for creep and control specimens. Red-filled points indicate specimens taken from the billet in the AG direction all others are WG.

All grades exhibited similar volumetric change behavior. As expected, the volume of all specimens decreased with irradiation and the percent change increased with dose. Furthermore, the decrease in volume of the creep (stressed) specimens was consistently higher than the control (unstressed) specimens. Overall, 2114 had the least amount of volumetric change (about 1.5% at 3.5 dpa for creep specimens). PCEA showed the highest volumetric change of about 4% at 3.5 dpa for creep specimens.

The lower 2114 volumetric change is not easily understood when compared to the other major grades. The grain size (super-fine grain) and fabrication method (isostatic molding) is similar to the Toyo Tanso IG-110 grade and should have a comparable response. The shallow volumetric response may indicate an early Turnaround dose. However, this will need to be proven in the longer dose capsule, AGC-4.

8. CREEP ANALYSIS

Longitudinal creep strain are calculated from the difference between the creep- and control-specimen lengths. AGC-3 creep calculations for all major graphite grades were performed using GAT. For these relatively lower dose levels (<10 dpa), the creep response should be linear and proportional to the applied stress. The creep specimens are subjected to a compressive stress, which will yield a negative slope to the longitudinal creep curve.

8.1 Creep Analysis by Graphite Grade

The combined measured strain response and calculated creep coefficients for all five major graphite grades are shown in Figure 16 through Figure 21 for stress levels of 0, 13.8, and 20.7 MPa. The data include a specimen irradiation temperature range of 750–920°C and are plotted as a function of the full dose range, 1.0–3.7 dpa. Figure 16 illustrates the combined creep strain for all graphite grades in AGC-3.

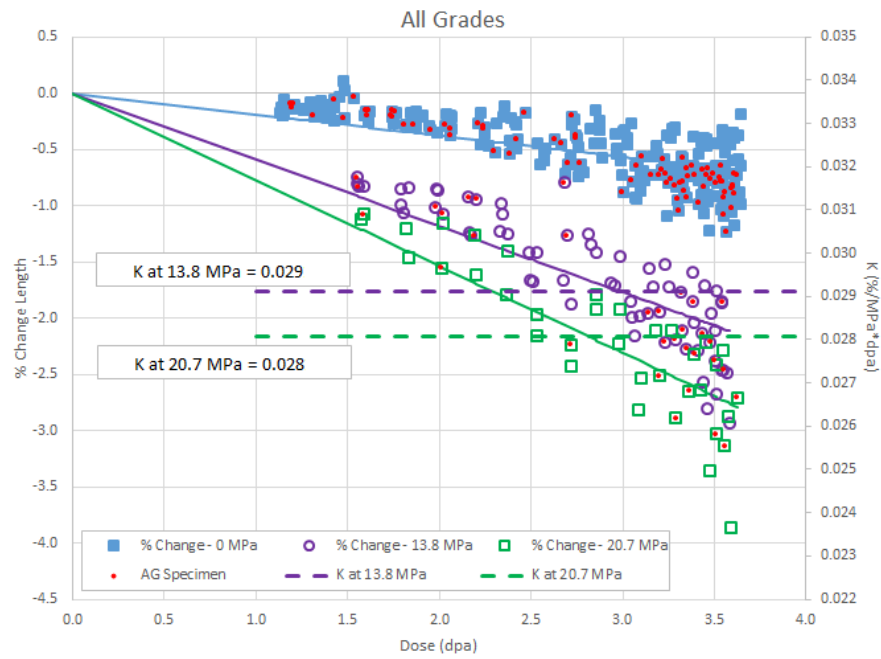


Figure 16. Longitudinal creep for all major grades of graphite in AGC-3. Both 13.8 and 20.7 MPa stress levels are included. AG specimens (represented by red dots) are included in this analysis as there is no discernable difference in the WG and AG response for AGC-3 irradiation conditions.

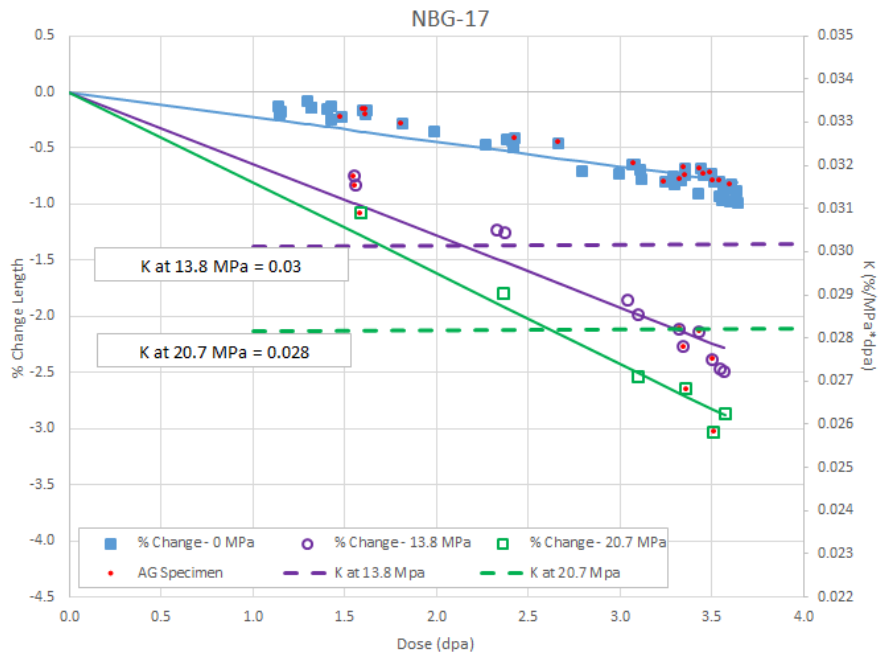


Figure 17. Longitudinal creep and creep coefficient K for NBG-17. K is calculated for two stress levels.

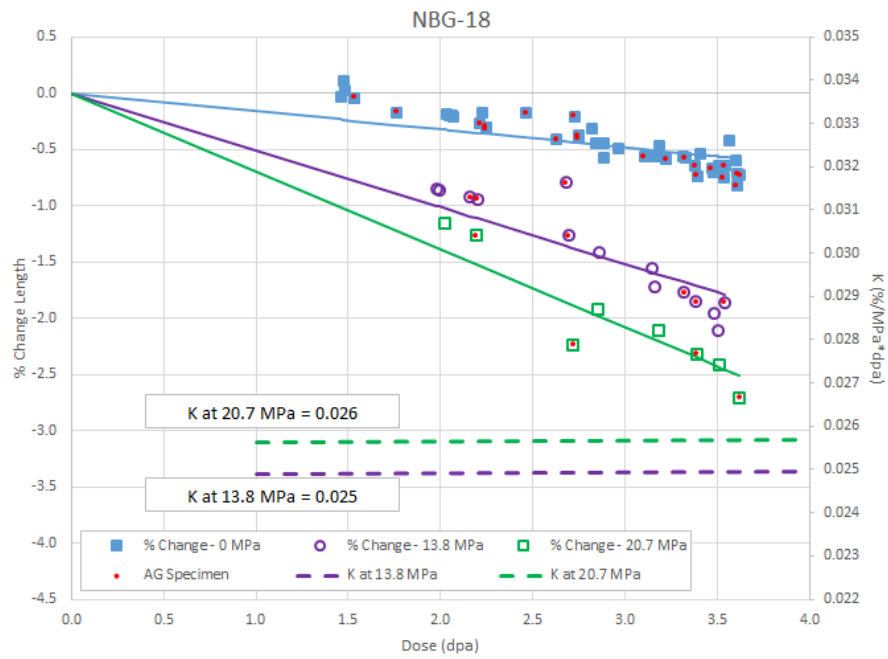


Figure 18. Longitudinal creep and creep coefficient K for NBG-18. K is calculated for two stress levels.

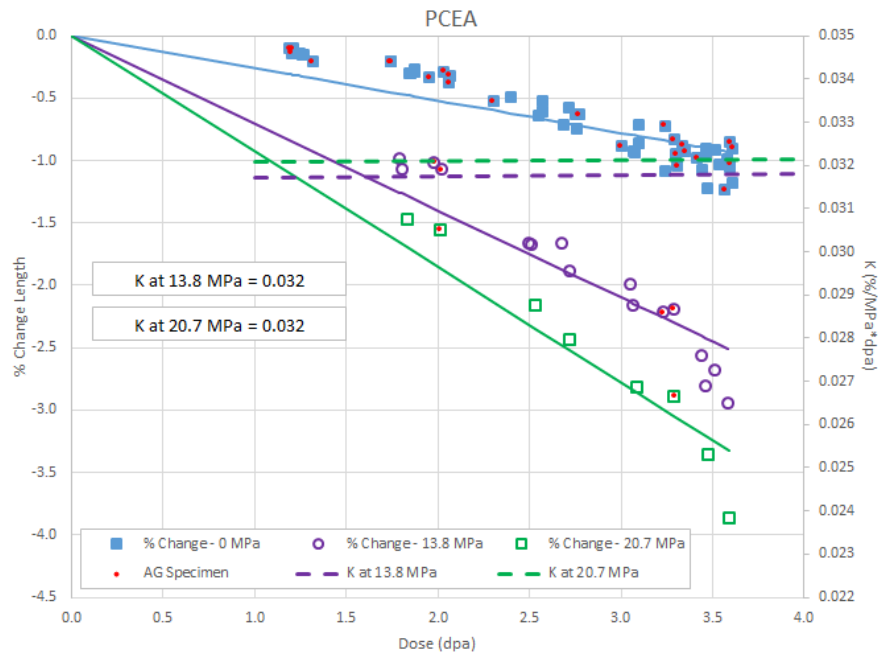


Figure 19. Longitudinal creep and creep coefficient K for PCEA. K is calculated for two stress levels.

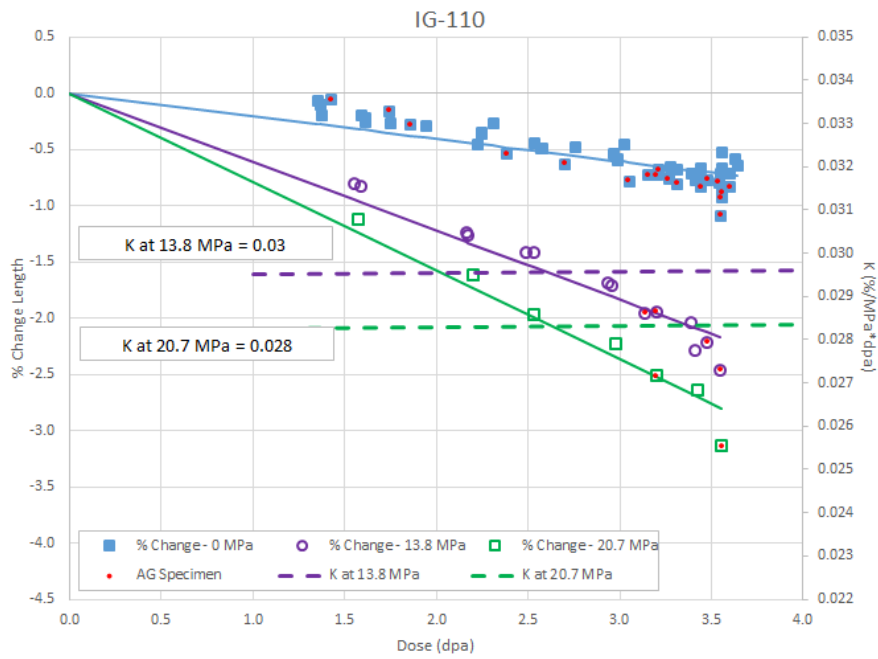


Figure 20. Longitudinal creep and creep coefficient K for IG-110. K is calculated for two stress levels.

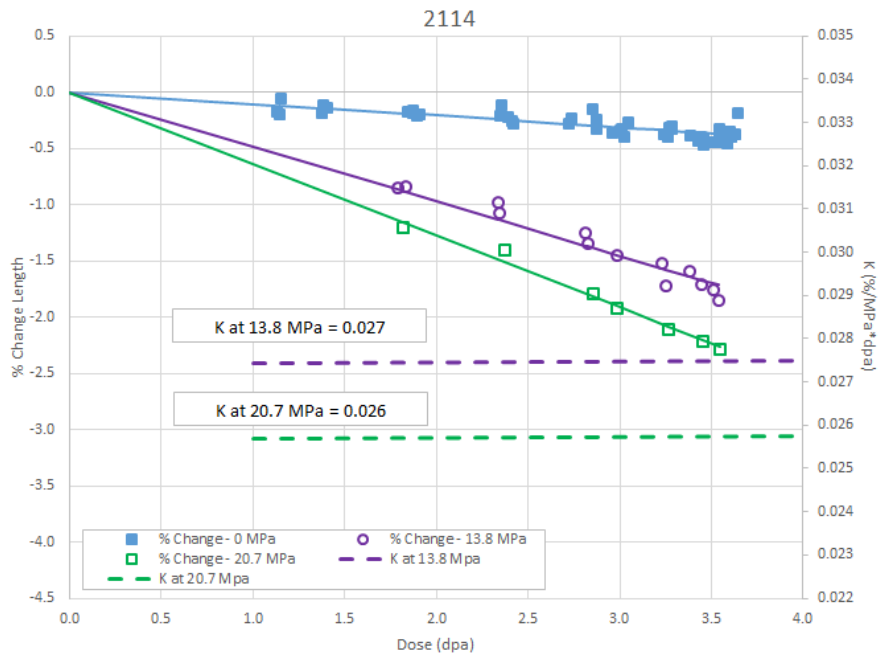


Figure 21. Longitudinal creep and creep coefficient K for 2114. K is calculated for two stress levels.

All grades exhibit linear creep response, and no grade indicates turnaround has been achieved over the temperature and dose ranges of AGC-3 irradiation. In all grades, the creep data show slight separation between the 13.8 MPa specimens and the 20.7 MPa specimens. The K values for PCEA (extruded grade) are the highest, while NBG-18 (vibrational molded) and 2114 (isostatic molded) have the lowest K values.

Grades NBG-17 and IG-110 had a similar creep response even though their fabrication processes are quite different. As seen in the 600°C analysis (AGC-1 and AGC-2), the largest creep strain experienced was for the extruded grades while the vibrational and isostatic molded grades experienced generally the same levels of strain.

9. CREEP AND CREEP COEFFICIENT ANALYSIS BY GRADE

The AGC-3 creep coefficients have been calculated for all major graphite grades and are compared to the calculated values for AGC-1 and AGC-2 (Table 11 and in Figure 23). Note that AGC-1 and AGC-2 creep coefficient values are for 600°C irradiation temperatures and were calculated using matched specimen pairs and normalized to a stress of 20.7 MPa. The AGC-3 creep coefficient values were calculated using the regression method described above for the stress of 20.7 MPa.

Table 11. Calculated longitudinal creep coefficients (K) for AGC-1, AGC-2, and AGC-3 graphite grades. The K value for AGC-3 was calculated for 20.7 MPa.

Capsule	Grade	Avg Temp	K (%/MPa*dpa)
AGC-1	NBG-17	600 °C	0.015
	NBG-18	600 °C	0.014
	PCEA	600 °C	0.018
	H-451	600 °C	0.020
	IG-110	600 °C	0.020
	IG-430	600 °C	0.032
	2114	-	-
AGC-2	NBG-17	625 °C	0.015
	NBG-18	625 °C	0.014
	PCEA	625 °C	0.019
	H-451	625 °C	0.017
	IG-110	625 °C	0.018
	IG-430	625 °C	0.016
	2114	-	-
AGC-3	NBG-17	820 °C	0.028
	NBG-18	820 °C	0.026
	PCEA	820 °C	0.032
	H-451	-	-
	IG-110	820 °C	0.028
	IG-430	-	-
	2114	820 °C	0.026

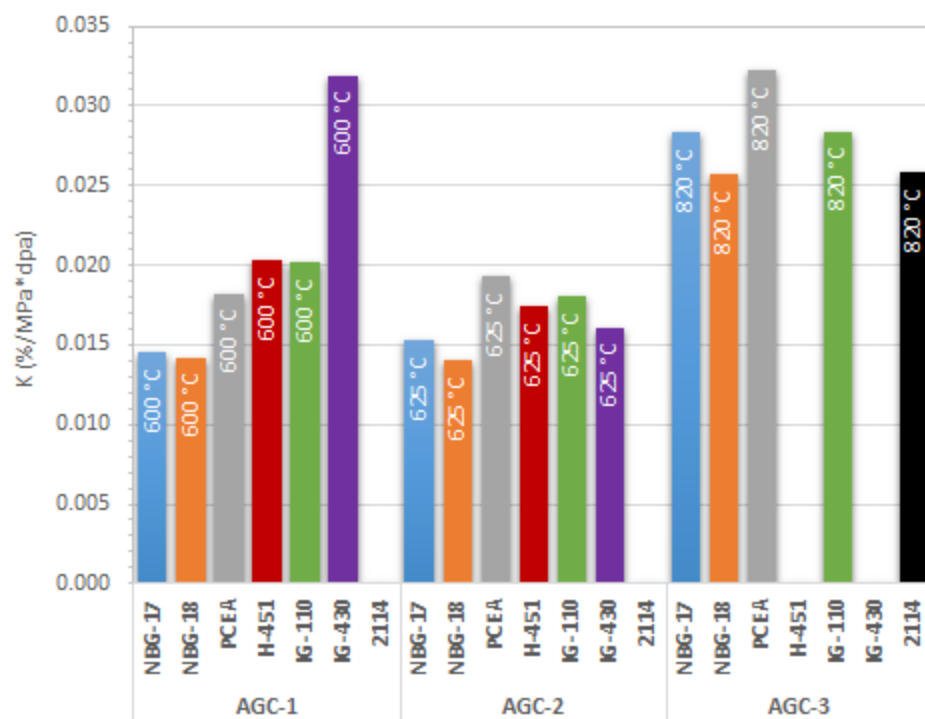


Figure 22. Calculated longitudinal creep coefficients for AGC 1, AGC 2, and AGC-3 graphite grades. The K value for AGC-3 was calculated for 20.7 MPa.

In general, 800°C AGC-3 creep coefficients are higher than the 600°C coefficients demonstrating the temperature dependence of irradiation creep in graphite. The creep coefficients for all three AGC experiments are shown in comparison to previous values calculated from historical creep studies (Figure 23).^{32,33,34,35,36,37,38,39,40} This comparison demonstrates the AGC-3 creep coefficients are well within the historical values determined for the irradiation temperatures and dose levels of the AGC-3 test train.

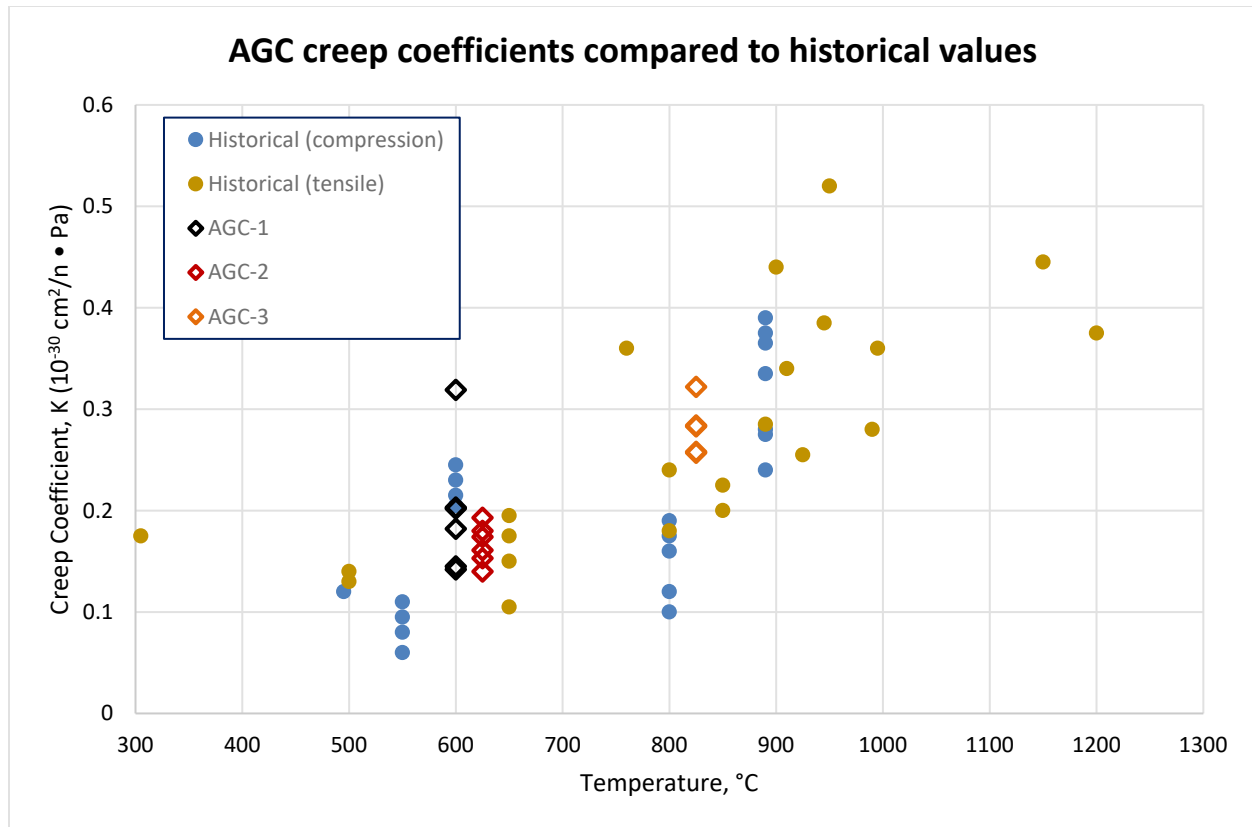


Figure 23. Comparison of AGC creep coefficients (\diamond) to historical values (\bullet) from previous studies.

The creep coefficient uncertainty is related to the applied stress, dose, and modulus values in addition to specimen irradiation temperature. Specific uncertainty calculations for AGC-3 have been initiated, with quantitative calculations of the temperature and applied stress levels being determined on a per specimen basis and semi-quantitative estimates of the received dose level.⁴¹ These uncertainty estimates will be applied to the combined 800°C AGC irradiation analysis (i.e., AGC-3 and AGC-4 analysis report) to ascertain the total uncertainty over the entire dose range at the nominal 800°C temperature experiments.

10. CONCLUSIONS

Dimensional change, creep strain, and creep coefficients have been analyzed for the five major graphite grades in the AGC-3 experiment.

The dimensional change analysis demonstrated that all five major grades had similar response to irradiation dose when considering specimens inside the full irradiation temperature range. The isotropic irradiation behavior for all grades was analyzed by comparing the length and diameter changes for all major grade specimens along with the volumetric behavior of creep (stressed) and control (unstressed) specimens. All grades had isotropic or near-isotropic irradiation response, and none of the grades indicated had signs of turnaround over the temperature and dose ranges of the AGC-3 irradiation.

Since both the irradiation dimensional change and creep strain are linear with dose, it was possible to calculate creep coefficients from linear regressions drawn through the dimensional change data. This analysis methodology was used in developing an application to automatically compile and calculate the irradiation-induced creep and creep coefficient for any of the major graphite grades. The Graphite Analysis Tool, has provided an efficient and accurate method to calculate the creep coefficient as a function of the AGC irradiation variables.

The creep response and calculated coefficients for all AGC-3 major grades were reasonable and within expected levels. The AGC-3 creep strain behavior was shown to compare well with historical data as well as data from AGC-1 and AGC-2 experiments. As with the matched specimen pair method, the K values calculated using the regression method are consistent across the different graphite grades. The maximum K value (PCEA) and the minimum K value (NBG-18) are within 25% of each other.

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