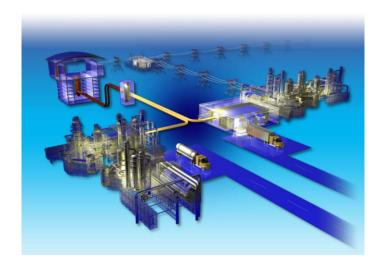
AGC-1 IRRADIATION INDUCED PROPERTY CHANGES ANALYSIS

REPORT: DYNAMIC ELASTIC MODULUS

Tim Burchell

Oak Ridge National Laboratory

March 2016



Prepared for
Office of Nuclear Energy Science and Technology
U.S. Department of Energy
Under a Memorandum Purchase Order No. 00153522 with
Idaho National Laboratory

Prepared by
OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37831
managed by
UT-BATTELLE, LLC
for the
U.S. DEPARTMENT OF ENERGY
under DOE Contract No. DE-AC05-000R22725



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

AGC-1 IRRADIATION INDUCED PROPERTY CHANGES ANALYSIS REPORT: DYNAMIC ELASTIC MODULUS

Tim Burchell
Oak Ridge National Laboratory

March 2016

Contents

Н	gur	es	• • • • • • •		V
T	able	es			vii
Α	bbr	evia	tions	and Acronyms	ix
S	umr	mary	<i>'</i>		1
1		Intro	oduct	tion	2
2		Ехре	erime	ent	2
	2.1	L	Dyn	amic Elastic Modulus (by fundamental frequency)	3
	2.2	2	Dyn	amic Modulus (ultrasonic velocity/time of flight)	3
	2.3	3	Irra	diation Conditions	3
	2.4	1	Erro	r Analysis and Significance Testing	3
		2.4.:	1	Error Analysis	3
		2.4.2	2	Statistical Significance Testing	5
3		Resu	ılts		6
	3.1	L	Dyn	amic Elastic Modulus (by fundamental frequency)	6
		3.1.	1	Grade Anisotropy Effects and Percent Increase of Saturated Dynamic Elastic Modulus	13
	3.2	2	Dyn	amic Elastic Modulus (by ultrasonic velocity/time of flight)	16
4		Disc	ussio	n	23
	4.1	L	Irra	diated Dynamic Elastic Modulus (by fundamental frequency)	23
		4.1.	1	Dynamic Elastic Modulus Statistical Testing (by fundamental frequency method)	27
	4.2	2	Irra	diated Dynamic Elastic Modulus (by ultrasonic velocity/time of flight)	33
		4.2.	1	Dynamic Elastic Modulus Statistical Testing (by ultrasonic velocity/time of flight)	37
5		Qua	lity A	ssurance	41
6		Con	clusio	ons	41
7		Ackr	nowle	edgements	41
8		Арр	endix	·	42
	8.1	L	Dyn	amic Elastic Modulus (by fundamental frequency)	42
	8.2	2	Dyn	amic Elastic Modulus (by ultrasonic velocity/time of flight)	48
9		Dist	ribut	ion	54
1	n	R	aforo	nces	5/1

Figures

Figure 1. Grade A, NBG-17 variation of dynamic elastic modulus with neutron dose	7
Figure 2. Grade A, NBG-17 variation of dynamic elastic modulus with creep strain	7
Figure 3. Grade B, NBG-18 variation of dynamic elastic modulus with neutron dose	8
Figure 4. Grade B, NBG-18 variation of dynamic elastic modulus with creep strain	8
Figure 5. Grade C, H-451 variation of dynamic elastic modulus with neutron dose	9
Figure 6. Grade C, H-451 variation of dynamic elastic modulus with creep strain	9
Figure 7. Grade D, PCEA variation of dynamic elastic modulus with neutron dose	10
Figure 8. Grade D, PCEA variation of dynamic elastic modulus with creep strain	10
Figure 9. Grade E, IG-110 variation of dynamic elastic modulus with neutron dose	11
Figure 10. Grade E, IG-110 variation of dynamic elastic modulus with creep strain	11
Figure 11. Grade F, IG-430 variation of dynamic elastic modulus with neutron dose	12
Figure 12. Grade F, IG-430 variation of dynamic elastic modulus with creep strain	12
Figure 13. Percent increase in dynamic elastic modulus from irradiation	13
Figure 14. Grade A, NBG-17 variation of dynamic elastic modulus (by ultrasonic velocity/tof) with	
neutron dose	17
Figure 15. Grade A, NBG-17 variation of dynamic elastic modulus (by ultrasonic velocity/tof) with c	reep
strain.	17
Figure 16. Grade B, NBG-18 variation of dynamic elastic modulus (by ultrasonic velocity/tof) with	
neutron dose	18
Figure 17. Grade B, NBG-18 variation of dynamic elastic modulus (by ultrasonic velocity/tof) with c	reep
strain.	18
Figure 18. Grade C, H-451 variation of dynamic elastic modulus (by ultrasonic velocity/tof) with ne	utron
dose	19
Figure 19. Grade C, H-451variation of dynamic elastic modulus (by ultrasonic velocity/tof) with cre	ер
strain.	19
Figure 20. Grade D, PCEA variation of dynamic elastic modulus (by ultrasonic velocity/tof) with neu-	ıtron
dose	20
Figure 21. Grade D, PCEA variation of dynamic elastic modulus (by ultrasonic velocity/tof) with cre	ер
strain	20
Figure 22. Grade E, IG-110 variation of dynamic elastic modulus (by ultrasonic velocity/tof) with ne	utron
dose	21
Figure 23. Grade E, IG-110 variation of dynamic elastic modulus (by ultrasonic velocity/tof) with creating the control of the	еер
strain.	21
Figure 24. Grade F, IG-430 variation of dynamic elastic modulus (by ultrasonic velocity/tof) with ne	utron
dose	22
Figure 25. Grade F, IG-430 variation of dynamic elastic modulus (by ultrasonic velocity/tof) with creations are supplied to the contract of th	еер
strain.	22
Figure 26. Grade A, NBG-17 (AL) and (AW) fractional changes in dynamic elastic modulus for control	ol and
creep (stressed) specimens	24

ORNL/SR-2015/378

Figure 27. Grade B, NBG-18 (BL) and (BW) fractional changes in dynamic elastic modulus for control and	ĺ
creep (stressed) specimens)	4
Figure 28. Grade C, H-451 fractional changes in dynamic elastic modulus for control and creep (stressed)
specimens	5
Figure 29. Grade D, PCEA (WG) and (AG) fractional changes in dynamic elastic modulus for control and	
creep (stressed) specimens	5
Figure 30. Grade E, IG-110 fractional changes in dynamic elastic modulus for control and creep (stressed specimens	-
Figure 31. Grade F, IG-430 fractional changes in dynamic elastic modulus for control and creep (stressed specimens	-
Figure 32. Grade A, NBG-17 (AL) and (AW) fractional changes in dynamic elastic modulus (ultrasonic	
velocity/tof) for control and creep (stressed) specimens	4
Figure 33. Grade B, NBG-18 (AL) and (AW) fractional changes in dynamic elastic modulus (ultrasonic	
velocity/tof) for control and creep (stressed) specimens	4
Figure 34. Grade C, H-451 fractional changes in dynamic elastic modulus (ultrasonic velocity/tof) for	
control and creep (stressed) specimens	5
Figure 35. Grade D, PCEA (AG) and (WG) fractional changes in dynamic elastic modulus (ultrasonic	
velocity/tof) for control and creep (stressed) specimens	5
Figure 36. Grade E, IG-110 fractional changes in dynamic elastic modulus (ultrasonic velocity/tof) for	
control and creep (stressed) specimens3	6
Figure 37. Grade F, IG-430 fractional changes in dynamic elastic modulus (ultrasonic velocity/tof) for	
control and creep (stressed) specimens3	6

Tables

Table 1. 3σ expressed as a percentage of mean for unirradiated dynamic elastic modulus (fundamental	1
frequency)	4
Table 2. 3σ expressed as a percentage of mean for unirradiated elastic modulus (ultrasonic velocity/tin of flight)	
Table 3. Significance level and their associated "P" values	
Table 4. Percent increase in mean dynamic elastic modulus by fundamental frequency of vibration	
(saturated value) from irradiation	15
Table 5. Statistical significance test questions	27
Table 6. Unirradiated dynamic elastic modulus means (ff method) and the dynamic elastic modulus (ff	
method) of the three highest dose creep and control specimens for each grade of graphite in AGC-1	28
Table 7. High dose "t" test outcomes	29
Table 8. Significance testing results for dynamic elastic modulus (ff method) at high dose	30
Table 9 Unirradiated dynamic elastic modulus means (ff method) and the dynamic elastic modulus (ff	
method) of the three lowest dose creep and control specimens for each grade of graphite in AGC-1	31
Table 10. Low dose "t" test outcomes	32
Table 11. Significance testing results for dynamic elastic modulus at low dose	32
Table 12. Unirradiated dynamic elastic modulus means (ultrasonic/tof method) and the dynamic elastic	С
modulus (ultrasonic/tof method) of the three highest dose creep and control specimens for each grade	5
of graphite in AGC-1	38
Table 13. Dose "t" test outcomes	39
Table 14. Significance testing results for dynamic elastic modulus (velocity/tof) at high dose	40
Table 15. Grade A, NBG-17 graphite mean dynamic elastic modulus (fundamental frequency method) f	or
creep (stressed) and control samples	42
Table 16. Grade B, NBG-18 graphite mean dynamic elastic modulus (fundamental frequency method) f	
creep (stressed) and control samples	43
Table 17. Grade C, H-451 graphite mean dynamic elastic modulus (fundamental frequency method) for	r
creep (stressed) and control samples	43
Table 18. Grade D, PCEA graphite mean dynamic elastic modulus (fundamental frequency method) for	
creep (stressed) and control samples	
Table 19. Grade E, IG-110 graphite mean dynamic elastic modulus (fundamental frequency method) fo	
creep (stressed) and control samples	46
Table 20. Grade F, IG-430 graphite mean dynamic elastic modulus (fundamental frequency method) fo	r
creep (stressed) and control samples	47
Table 21. Grade A, NBG-17 graphite mean Dynamic elastic modulus (ultrasonic velocity/tof) for creep	
(stressed) and control samples	48
Table 22. Grade B, NBG-18 graphite mean dynamic elastic modulus (ultrasonic velocity/tof) for creep	
(stressed) and control samples	49
Table 23. Grade C, H-451 graphite mean dynamic elastic modulus (ultrasonic velocity/tof) for creep	
(stressed) and control samples	50

ORNL/SR-2015/378

Table 24. Grade D, PCEA graphite mean dynamic elastic modulus (ultrasonic velocity/tof) for creep	
(stressed) and control samples	.51
Table 25. Grade E, IG-110 graphite mean dynamic elastic modulus (ultrasonic velocity/tof) for creep	
(stressed) and control samples	.52
Table 26. Grade F, IG-430 graphite mean dynamic elastic modulus (ultrasonic velocity/tof) for creep	
(stressed) and control samples	.53

Abbreviations and Acronyms

AG against grain

AGC Advanced graphite creep

ASTM American Society for Testing and Materials

ATR Advanced Test Reactor

CTE Coefficient of thermal expansion

 E_{dyn} dynamic elastic modulus

dpa displacements per atom

ff fundamental frequency

HTR High Temperature Reactor

INL Idaho National Laboratory

ORNL Oak Ridge National Laboratory

tof time of flight

WG with grain

Summary

Final analyses of irradiated materials property data from the Advanced Graphite Creep (AGC)-1 capsule are provided in this report. Specifically, the analyses of data for the compressive irradiation induced creep strain effects on the dynamic elastic modulus (measured by the fundamental frequency and ultrasonic velocity test methods). Statistical "t" testing was used to establish the statistical significance of any differences observed between the dynamic elastic modulus of the creep (irradiated with compressive stress) and the control (irradiated without stress) specimens. Results indicate compressive creep strain does have a small effect on irradiated dynamic elastic modulus, reducing the modulus at the higher dose as volume turnaround is approached.

1 Introduction

This report, along with a companion report on electrical resistivity and the coefficient of thermal expansion¹, represents the final detailed data assessments from the Advanced Graphite Creep (AGC)-1 capsule, ^{2,3} the first and prototype irradiation creep capsule of the AGC experiment. Pre-irradiation data⁴ and post-irradiation data⁵ have previously been reported. Analysis of the dimensional change, volume change, and creep strain data is given in ORNL/TM-2014/255⁶. This report analyzes the property change data for AGC-1. Specifically, dynamic elastic modulus is measured by the (1) fundamental frequency (ff) of vibration method and (2) ultrasonic velocity/time of flight method, as a function of irradiation induced creep strain at a nominal temperature of 641°C and to a peak dose of 7 displacements per atom (dpa). The dynamic elastic modulus data and analysis from this report will be supplied to the Nuclear Data Management and Analysis System and the Generation (Gen) IV Materials Handbook. Although this report only covers the dynamic elastic modulus, shear velocity was also measured, which allows calculation of the shear modulus G, and Poison's Ratio v. However, these properties will not be analyzed until discrepancies between the Oak Ridge National Laboratory (ORNL) and Idaho National Laboratory (INL) testing methodology have been resolved. The dynamic elastic modulus data are reported as a function of neutron dose, dpa, and percent compressive creep strain. By convention, a compressive stress causes a negative strain. The percent strain data are highlighted in yellow in Sections 8.1 and 8.2.

2 Experiment

The AGC experiment will provide irradiated material property data for current graphite types available for use within a high temperature reactor (HTR) design. The AGC experiment measures the irradiated material property changes and behavior of these new nuclear grade graphites over the operating temperature range, neutron fluences, and mechanical loads anticipated for an HTR core. The experiment generates quantitative material property change data (and steady-state irradiation creep data), which will be used in conjunction with the as-fabricated material property measurement (i.e., Baseline) program to predict the in-service behavior and operating performance of these new nuclear graphite grades for HTR designs.

Changes to key thermal, physical, and mechanical material properties are determined by comparing the material properties of each specimen before irradiation and after irradiation. The approach is to perform extensive pre-irradiation characterization testing on each specimen within the irradiation capsules before exposing them to a range of neutron doses. After irradiation, the same characterization tests are performed on each irradiated specimen to ascertain the quantitative changes to the material properties of the graphite. To ensure consistency, American Society for Testing and Materials (ASTM) test standards are used for both pre-irradiation and post-irradiation testing.

2.1 Dynamic Elastic Modulus (by fundamental frequency)

Approved ASTM testing standards^{9,10} were used to determine the elastic modulus from the fundamental frequency of vibration⁴ as described in the AGC-1 pre-irradiation data report.

2.2 Dynamic Modulus (ultrasonic velocity/time of flight)

ASTM C769-09 test standard was used to determine the dynamic elastic modulus from the ultrasonic velocity or time of flight (tof) as previously described⁴ in the AGC-1 pre-irradiation data report.

2.3 Irradiation Conditions

The AGC-1 creep capsule was irradiated in the Advance Test Reactor (ATR) at INL over the dose range of approximately 3–7 dpa. The as-run mean temperature of all specimens in the capsule was 641°C; however, the temperatures varied along the capsule length, and the nominal capsule temperature ran hotter with each subsequent cycle. Thus, the specimen temperatures varied from 470–716°C depending upon their position within the AGC-1 irradiation capsule (i.e., specimens located at the ends of the capsule experienced 470°C, while specimens in the center region experienced a maximum of 716°C). During each irradiation cycle, the creep specimens located in the upper half of the AGC-1 capsule were subjected to a compressive load. Three different stress levels were applied to the upper portions of the six perimeter stacks (13.8 MPa [2 ksi], 17.2 MPa [2,500 psi], and 20.7 MPa [3 ksi]). The applied load was monitored and recorded at 5-minute intervals for each stack. All loads remained relatively constant throughout the reactor cycles (during reactor outages the load was removed until the beginning of the next reactor cycle). Full details of the specimens irradiation history was previously reported in the AGC-1 creep analysis report⁶.

2.4 Error Analysis and Significance Testing

2.4.1 Error Analysis

An effort has been made to quantify the experimental errors and display them as error bars on the property versus dose plots and the property versus creep strain plots. An estimate of the uncertainty, arising from both experimental errors and material variability, was taken to be $\pm 3\sigma$ of the distribution of the unirradiated property values (from pre-irradiation examination of the specimens⁴). This $\pm 3\sigma$ quantity was expressed as a percentage of the unirradiated mean and applied (as a percentage) to both the unirradiated and irradiated specimen populations discussed here. The error values applied for each grade and orientation are shown in Table 1 and Table 2 for elastic modulus by ff and ultrasonic velocity/tof, respectively.

Table 1. 3σ expressed as a percentage of mean for unirradiated dynamic elastic modulus (fundamental frequency).

Unirradiated Values (All Specimens Zero Dose)													
	Elastic Modulus E (GPa) by ff												
Grade	Number of Samples (n)	Mean	σ	3σ	Error fraction, %, (3σ/mean)								
NBG-17-(AW)	30	11.19	0.093	0.278	2.48								
NBG-17-(AL)	8	11.58	0.170	0.510	4.40								
NBG-18-(BW)	30	12.17	0.072	0.216	1.77								
NBG-18-(BL)	8	12.46	0.230	0.689	5.53								
H-451	22	8.84	0.378	1.135	12.84								
PCEA (WG)	33	10.98	0.149	0.446	4.06								
PCEA (AG)	5	8.98	0.042	0.127	1.42								
IG-110	22	8.88	0.218	0.655	7.37								
IG-430	34	9.48	0.261	0.784	8.27								

Table 2. 3σ expressed as a percentage of mean for unirradiated elastic modulus (ultrasonic velocity/time of flight).

Unirradiated Values (All Specimens Zero Dose)													
	Elastic Modulus E (GPa) by ultrasonic tof												
	(corrected for υ)												
	No. of												
Grade	Samples, n	Mean	St. Dev	3 x SD	(3SD/mean)								
NBG-17-(AW)	30	9.94	0.1	0.3	3.02								
NBG-17-(AL)	8	10.2	0.139	0.417	4.09								
NBG-18-(BW)	30	10.66	0.102	0.306	2.87								
NBG-18-(BL)	8	10.8	0.2	0.6	5.56								
H-451	22	7.97	0.1854	0.5562	6.98								
PCEA (WG)	33	9.41	0.1518	0.4554	4.84								
PCEA (AG)	5	8.61	0.664	1.992	23.14								
IG-110	22	8.04	0.108	0.324	4.03								
IG-430	34	9.65	0.1761	0.5283	5.47								

Based on previous ATR experience, the irradiation dose error was assumed to be ±5%. However, arriving at an estimated error for the creep strain was more challenging. If the specimen pairs were exactly matched in both irradiation temperature and dose, the creep error would only arise from the differences in dimensional measurements. Since specimen dimensions are measured with high precision and accuracy, the errors would be very small. However, AGC-1 was a prototype capsule, and the temperature control was not optimal. There was a significant temperature gradient along the length of the capsule, and the capsule mean temperature increased with each successive ATR cycle. Consequently, there is considerable uncertainty regarding the exact specimen temperatures. Similarly, there is some uncertainty associated with the specimen irradiation dose (±5%) and a larger concern as to whether the matched pair of creep and control samples received identical irradiation doses. Finally, the applied load uncertainties are very small for AGC-1 and are assumed to be enveloped by the

temperature and dose uncertainties. Given the uncertainties associated with the irradiation conditions of the matched pairs in AGC-1, it is unlikely to know the exact specimen creep strain to better than ±10%. Consequently, the creep strain error bars on the property versus creep strain plots (Figure 2, Figure 4, Figure 6, Figure 8, Figure 10, Figure 12, Figure 15, Figure 17, Figure 19, Figure 21, Figure 23, and Figure 25) were set to ±10%.

2.4.2 Statistical Significance Testing

Later in this report, significance testing is extensively used to establish differences between selected properties of the irradiated (control) specimen and the irradiated and stressed (creep) specimens. Moreover, the irradiation doses of each specimen property data group are compared to one another to first establish similar irradiation doses. The statistical significance was tested by calculating the "P" statistic (which is a function of the mean, standard deviation, and population size [n]) and comparing it to the probabilities defined in the student's "t" distribution. The "P" statistic was calculated and significance testing carried out using GraphPad ¹² software. The GraphPad software "t" test was validated by the author prior to use here.

The levels of significance used, their exact terminology and the associated "P" values are given in Table 3.

Table 3. Significance level and their associated "P" values.

"P" Value	Terminology								
Less than 0.0001	Extremely Significant								
0.0001 to 0.001	Extremely Significant								
0.001 to 0.01	Very Significant								
0.01 to 0.05	Significant								
Greater than or equal to 0.05	Not significant								

The statistical significance of any observed material property difference between creep and control specimens are determined entirely on the reported "P" values, as determined from Table 3.

3 Results

3.1 Dynamic Elastic Modulus (by fundamental frequency)

Mean unirradiated specimen dynamic elastic modulus values measured by the ff test method⁴ (Table 1) are plotted at zero dose or strain along with the irradiated control and crept specimen data. The post-irradiation data⁵ for creep and control specimens from AGC-1 are given in Figure 1 to Figure 12, with tabular data presented in Section 8.1. Elastic modulus data are reported for both with grain (WG) and against grain (AG) orientations for the extruded grade PCEA and in the WG orientation only for the extruded grade H-451. In the case of the vibrationally molded grades (NBG-17 and NBG-18), data are reported for specimens cut with their long axis perpendicular to the forming direction (WG), identified as AL or BL (longitudinal specimens), or with the specimen long axis parallel to the forming direction (AG), identified as AW or BW.

As expected, examination of Figure 1 to Figure 12 indicates the elastic modulus increases substantially after irradiation. This increase appears to have saturated over the 3–7 dpa AGC-1 dose range, indicating the modulus has achieved a maximum, relatively constant value as expected over this moderate dose range. Of note, the modulus appears to be constant up to the maximum dose level (7 dpa), indicating that AGC-1 has been irradiated in the steady-state creep regime and turnaround has not been reached for these specimens.

In addition, the elastic modulus values are similar for specimens subjected to compressive stress and those that have not been stressed (i.e., control and creep specimens appear to have similar measured behavior). Statistical significance testing was used to quantitatively evaluate the potential effect that irradiation or creep strain may have had on the elastic modulus (by ff). As reported in Section 4, the reduction of the dynamic modulus (creep) relative to the control specimens, although small, was found to be statistically significant.

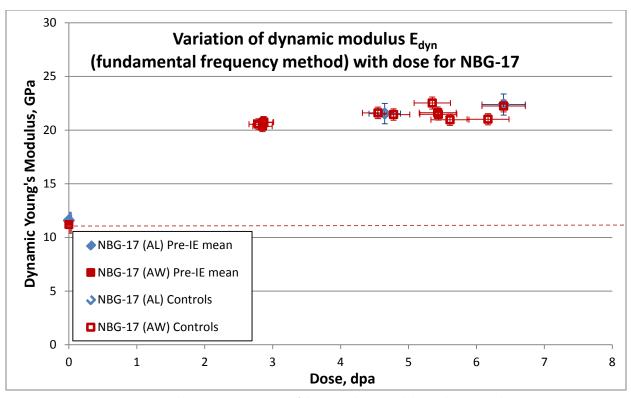


Figure 1. Grade A, NBG-17 variation of dynamic elastic modulus with neutron dose.

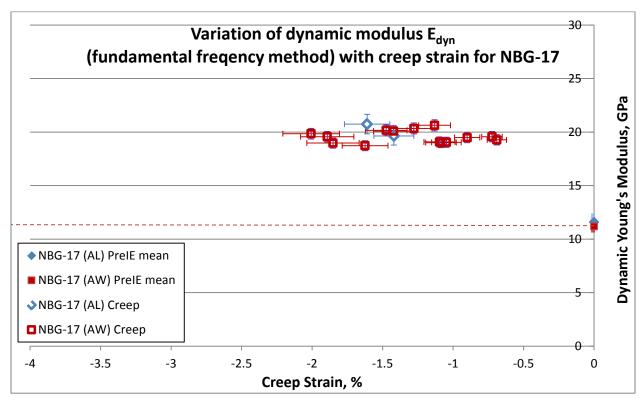


Figure 2. Grade A, NBG-17 variation of dynamic elastic modulus with creep strain.

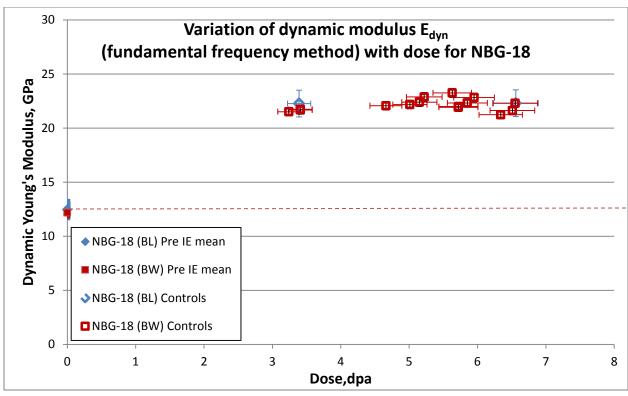


Figure 3. Grade B, NBG-18 variation of dynamic elastic modulus with neutron dose.

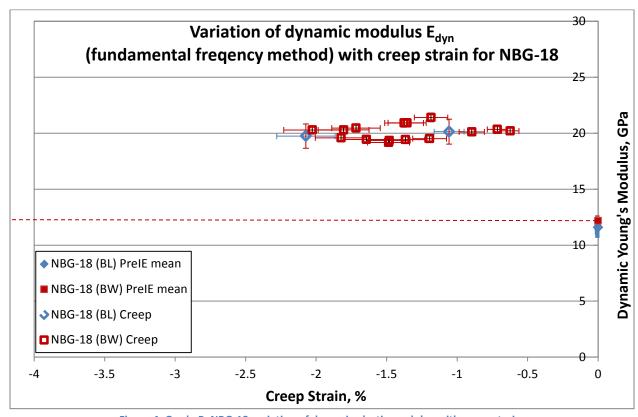


Figure 4. Grade B, NBG-18 variation of dynamic elastic modulus with creep strain.

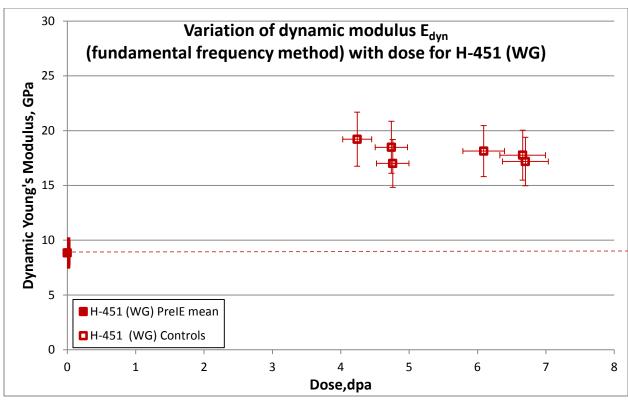


Figure 5. Grade C, H-451 variation of dynamic elastic modulus with neutron dose.

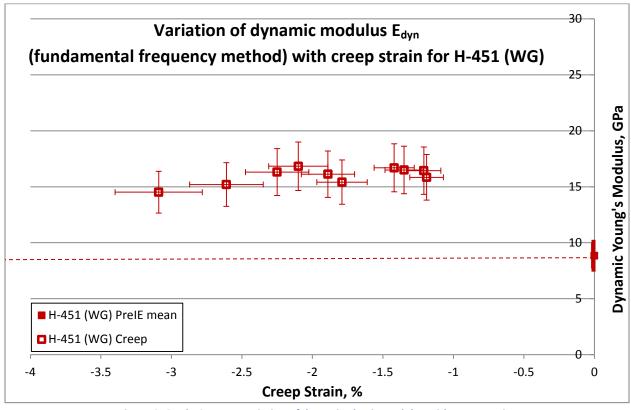


Figure 6. Grade C, H-451 variation of dynamic elastic modulus with creep strain.

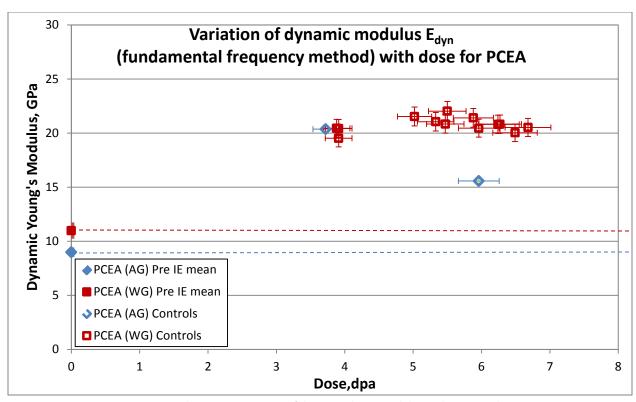


Figure 7. Grade D, PCEA variation of dynamic elastic modulus with neutron dose.

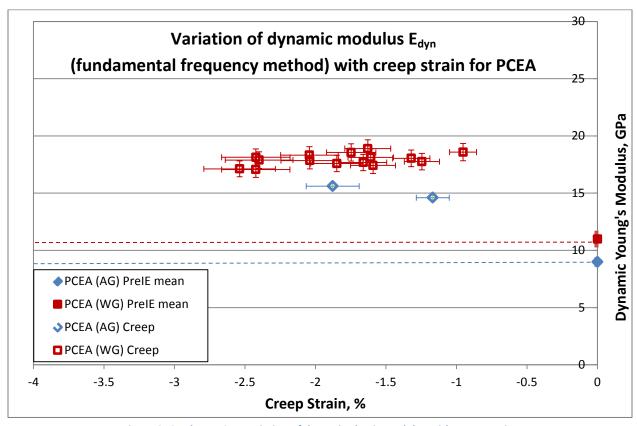


Figure 8. Grade D, PCEA variation of dynamic elastic modulus with creep strain.

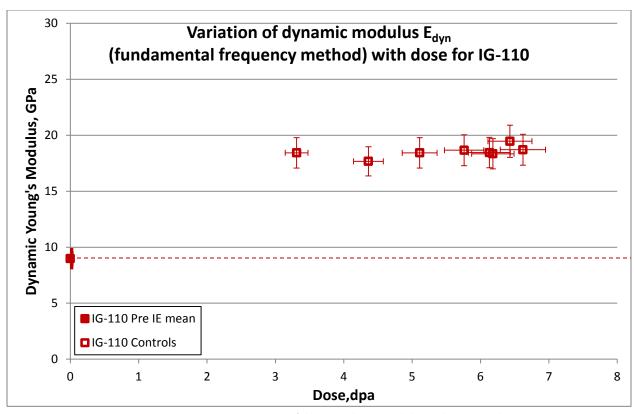


Figure 9. Grade E, IG-110 variation of dynamic elastic modulus with neutron dose.

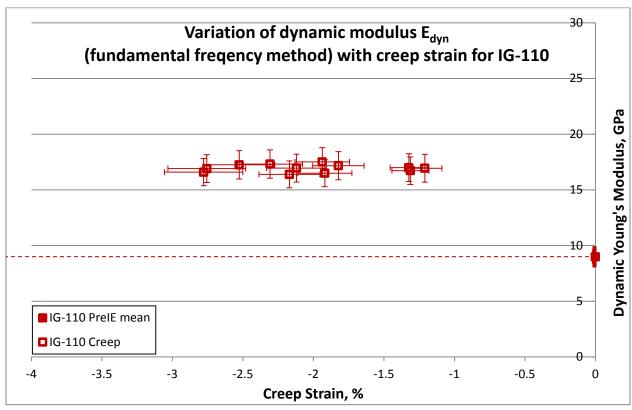


Figure 10. Grade E, IG-110 variation of dynamic elastic modulus with creep strain.

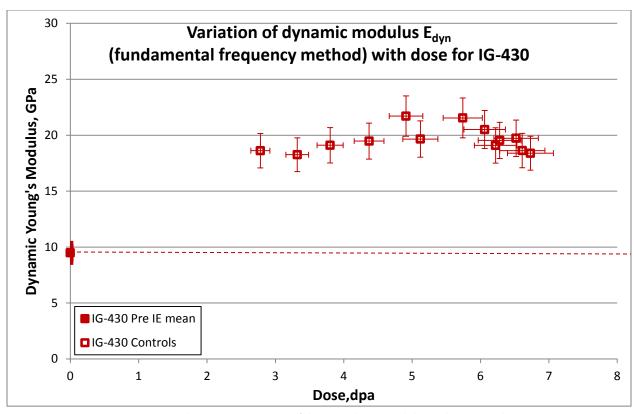


Figure 11. Grade F, IG-430 variation of dynamic elastic modulus with neutron dose.

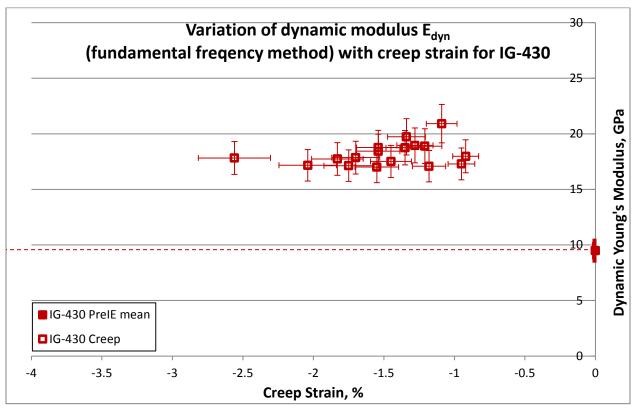


Figure 12. Grade F, IG-430 variation of dynamic elastic modulus with creep strain.

3.1.1 Grade Anisotropy Effects and Percent Increase of Saturated Dynamic Elastic Modulus

Assuming the modulus saturates at an elevated level from defect pinning and does not change further due to pore structure changes in the graphite over the AGC-1 dose range (this is in doubt, as there is probably some pore modification occurring in certain grades at these fluences), the increase in the mean elastic modulus attributed to irradiation only can be calculated. The assumption of a constant saturated elastic modulus is reasonable because the pore size derived change in elastic modulus is small compared to the initial increase due to the irradiation induced lattice defect population.

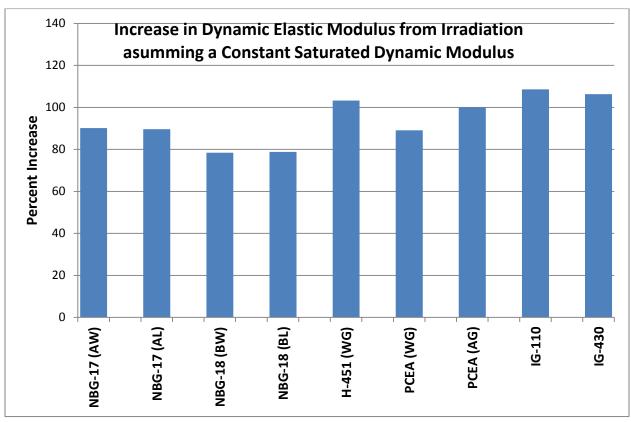


Figure 13. Percent increase in dynamic elastic modulus from irradiation.

The percent increases in dynamic elastic modulus presented in Figure 13 were calculated using the irradiated means in Table 4 and the unirradiated means in Table 1. The largest increases are seen with the fine-grained isostatically-pressed isotropic grades IG-110 and IG-430. The vibration molded grades (NBG-17 and NBG-18) were also very isotropic. The extruded grade PCEA exhibited some anisotropy, the AG direction exhibiting a lower dynamic elastic modulus (ff) than the WG direction (Table 1). This difference should be apparent in an extruded grade because of the preferred alignment of the filler coke along the extrusion axis. The atomic bonding between the carbon atoms within the crystallographic basal planes is the extremely stiff (high modulus covalent bonds), whereas the bonding between the graphite crystal basal planes is of the Van der Waals type and orders of magnitude less stiff. The elastic modulus will therefore be greater (for the PCEA [WG]) since there is a preferred alignment of the

graphite basal planes (i.e., where the coke particles are aligned along the extrusion axis forming a WG and AG direction).

Table 4. Percent increase in mean dynamic elastic modulus by fundamental frequency of vibration (saturated value) from irradiation.

Saturared Dynamic Modulus vaules, GPa																	
NBG-17 (AW) NBG-17 (AL) NBG-			8 (BW) NBG-18 (BL)			H-451 (WG) PCE		PCE/	A (WG) PCEA (AG		(AG)	AG) IG-110		IG-430			
	Dynamic		Dynamic		Dynamic		Dynamic		Dynamic		Dynamic		Dynamic		Dynamic		Dynamic
Specimen	Moduls,	Specimen	Moduls,	Specimen	Moduls,	Specimen	Moduls,	Specimen	Moduls,	Specimen	Moduls,	Specimen	Moduls,	Specimen	Moduls,	Specimen	Moduls,
ID	GPa	ID	GPa	ID	GPa	ID	GPa	ID	GPa	ID	GPa	ID	GPa	ID	GPa	ID	GPa
AW6-01	20.55	AL8-02	21.54	BW8-03	21.51	BL7-02	22.26	CW13-01	19.21	DW10-02	20.44	DA702	20.36	EW9-02	18.43	FW4-01	18.61
AW13-01	20.36	AL6-01	22.38	BW1-02	21.67	BL6-02	22.29	CW14-02	18.48	DW2-03	20.42	DA601	15.56	EW2-03	17.67	FW7-03	18.26
AW5-01	20.71			BW11-02	21.73			CW8-03	17.01	DW5-01	19.52			EW10-01	18.43	FW7-02	19.10
AW2-03	20.75			BW7-01	22.06			CW10-03	18.13	DW6-01	21.53			EW5-03	18.66	FW3-03	19.47
AW7-01	21.60			BW10-02	22.17			CW10-02	17.76	DW9-01	21.05			EW8-01	18.45	FW9-01	21.71
AW4-03	21.45			BW3-01	22.39			CW8-02	17.18	DW4-01	20.83			EW5-02	18.35	FW2-01	19.65
AW5-03	22.52			BW8-02	22.87					DW7-02	22.02			EW10-03	19.47	FW5-03	21.55
AW12-03	21.62			BW5-03	23.25					DW5-03	21.41			EW9-01	18.71	FW10-02	20.51
AW2-02	21.47			BW5-02	21.99					DW2-02	20.45					FW3-02	19.09
AW10-01	20.96			BW12-01	21.92					DW8-03	20.80					FW7-01	19.54
AW2-01	21.02			BW10-01	22.32					DW7-01	20.82					FW10-01	19.73
AW9-03	22.25			BW8-01	22.82					DW2-01	20.03					FW1-03	18.63
				BW1-03	21.23					DW4-03	20.52					FW11-03	18.39
				BW11-03	21.62												
				BW2-03	22.30												
Mean	21.27	Mean	21.96	Mean	22.12	Mean	22.28	Mean	17.96	Mean	20.76	Mean	17.96	Mean	18.52	mean	19.56
SD	0.67	SD	0.423	SD	0.55	SD	0.02	SD	0.83	SD	0.65	SD	3.39	SD	0.50	SD	1.11
% Inc. on		% Inc. on		% Inc. on		% Inc. on		% Inc. on		% Inc. on		% Inc. on		% Inc. on		% Inc. on	
Irradition		Irradition		Irradition		Irradition		Irradition		Irradition		Irradition		Irradition		Irradition	
only	90.10	only	89.62	only	78.38	only	78.79	only	103.21	only	89.04	only	100.03	only	108.59	only	106.30

3.2 Dynamic Elastic Modulus (by ultrasonic velocity/time of flight)

Mean unirradiated specimen dynamic elastic modulus values (measured by the ultrasonic velocity/tof testing method)⁴ corrected for υ are summarized in Table 2. The unirradiated means are plotted along with the irradiated and crept specimen data. The post-irradiation data⁵ for the creep and control specimens from AGC-1 are given in Figure 14 to Figure 25, with tabular data in Section 8.2. As with the previous data plots, elastic modulus data are reported for both WG and AG orientations for the extruded grade PCEA and in the WG orientation only for the extruded grade H-451. In the case of the vibrationally molded grades (NBG-17 and NBG-18), data are reported for specimens cut with their long axis perpendicular to the forming direction (WG), identified as AL or BL (longitudinal specimens) or with the specimen long axis parallel to the forming direction (AG), identified as AW or BW.

Similar to previous results, examination of Figure 14 to Figure 25 indicates the elastic modulus for the AGC-1 specimens increases substantially after irradiation with or without being subjected to a compressive stress (i.e., control and creep specimens have similar initial behavior). Moreover, there appears to be only a small additional effect of creep strain on elastic modulus in the 3–7 dpa dose range of AGC-1. Statistical significance testing was used to quantitatively evaluate the potential effect that irradiation or creep may have had on elastic modulus differences between unirradiated, control, and creep specimens. As reported in the Section 4, the reduction of the dynamic modulus (creep) relative to the control specimens, although small, was found to be statistically significant.

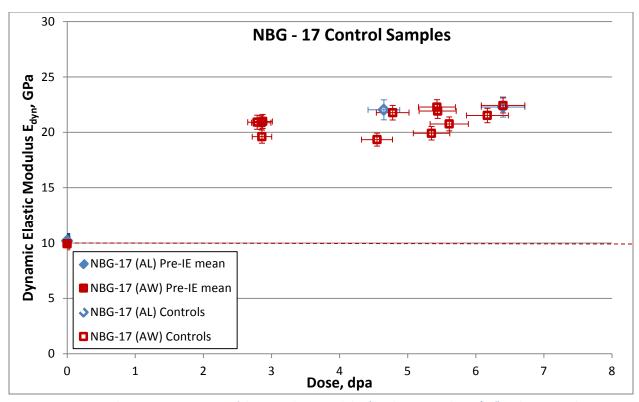


Figure 14. Grade A, NBG-17 variation of dynamic elastic modulus (by ultrasonic velocity/tof) with neutron dose.

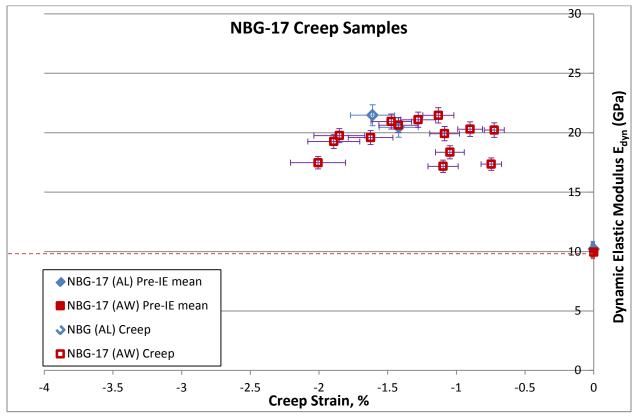


Figure 15. Grade A, NBG-17 variation of dynamic elastic modulus (by ultrasonic velocity/tof) with creep strain.

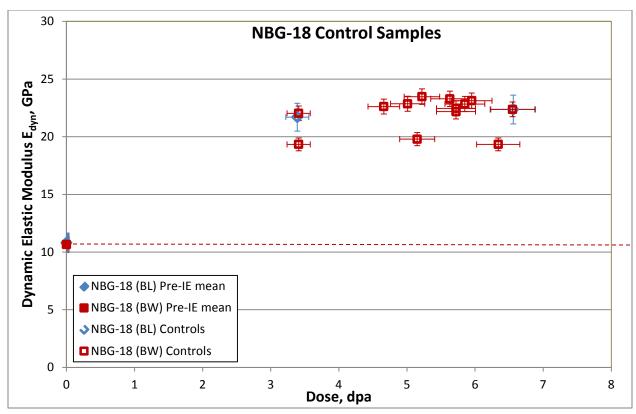


Figure 16. Grade B, NBG-18 variation of dynamic elastic modulus (by ultrasonic velocity/tof) with neutron dose.

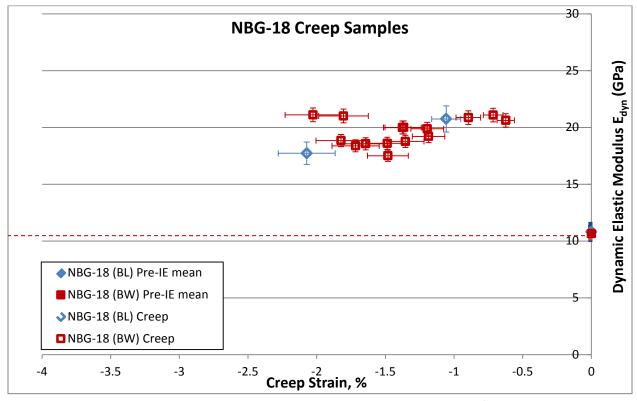


Figure 17. Grade B, NBG-18 variation of dynamic elastic modulus (by ultrasonic velocity/tof) with creep strain.

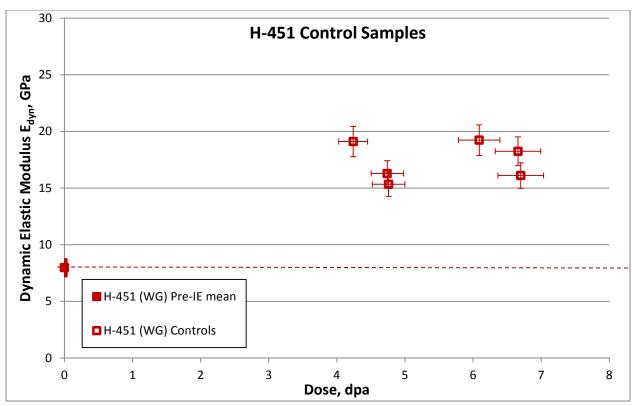


Figure 18. Grade C, H-451 variation of dynamic elastic modulus (by ultrasonic velocity/tof) with neutron dose.

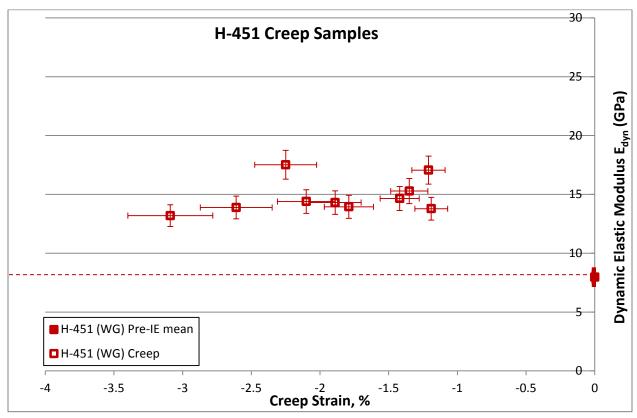


Figure 19. Grade C, H-451variation of dynamic elastic modulus (by ultrasonic velocity/tof) with creep strain.

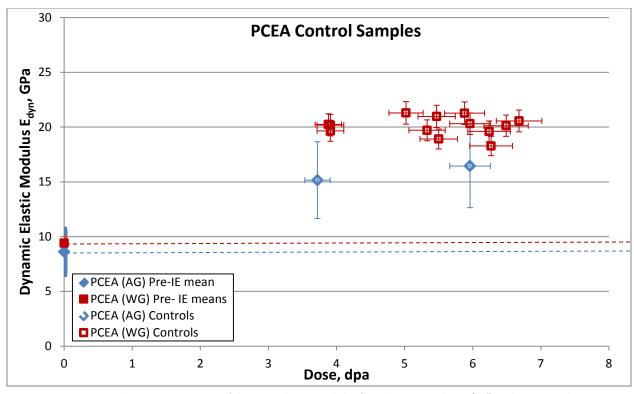


Figure 20. Grade D, PCEA variation of dynamic elastic modulus (by ultrasonic velocity/tof) with neutron dose.

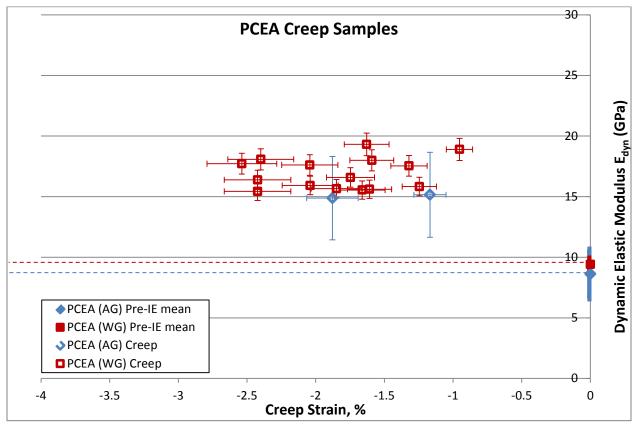


Figure 21. Grade D, PCEA variation of dynamic elastic modulus (by ultrasonic velocity/tof) with creep strain.

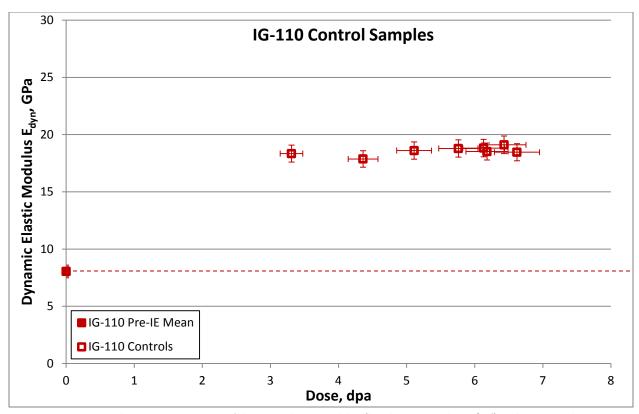


Figure 22. Grade E, IG-110 variation of dynamic elastic modulus (by ultrasonic velocity/tof) with neutron dose.

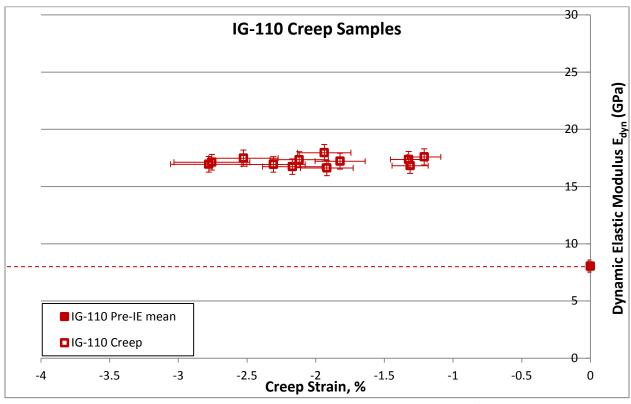


Figure 23. Grade E, IG-110 variation of dynamic elastic modulus (by ultrasonic velocity/tof) with creep strain.

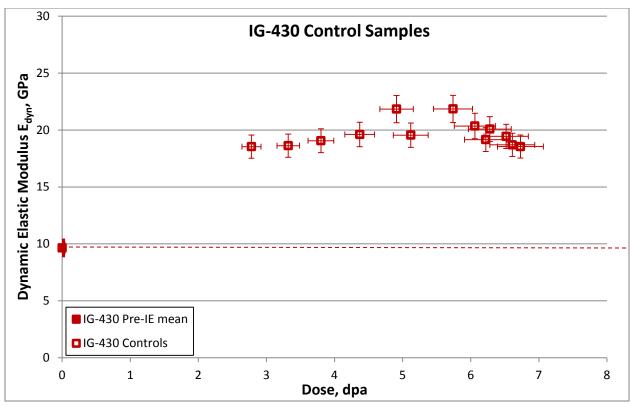


Figure 24. Grade F, IG-430 variation of dynamic elastic modulus (by ultrasonic velocity/tof) with neutron dose.

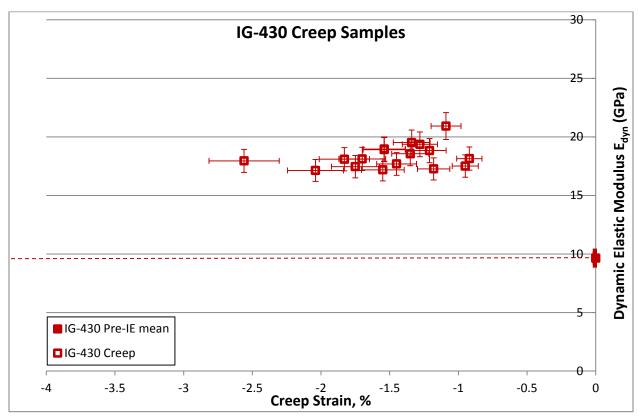


Figure 25. Grade F, IG-430 variation of dynamic elastic modulus (by ultrasonic velocity/tof) with creep strain.

4 Discussion

4.1 Irradiated Dynamic Elastic Modulus (by fundamental frequency)

Irradiated dynamic elastic modulus values appear to saturate at 15–25 GPa, depending on the graphite grade (Table 4 and Figure 13). Increases in dynamic elastic modulus after irradiation are expected to arise from the increased number of irradiation induced damage sites within the crystal structure, which act as dislocation pinning points in the graphite crystallites; thus, making basal plane slip more difficult and resulting in an increase in the stiffness.

The relative changes between irradiated graphite (control specimens) and irradiated and stressed graphite (creep specimens) are more easily seen if fractional changes in dynamic elastic modulus are plotted. The fractional change in modulus is given by

Fractional Change =
$$\frac{E_i - E_0}{E_0}$$

= $\left(\frac{E_i}{E_0} - \frac{E}{E_0}\right)$
= $\left(\left(\frac{E_i}{E_0}\right) - 1\right)$

Equation 1

where E_0 = unirradiated dynamic elastic modulus (GPa) and

E_i = irradiated or irradiated/stressed dynamic elastic modulus (GPa).

The following figures show the fraction changes in dynamic elastic modulus (measured by the ff method) as a function of dose (dpa) for irradiated (control specimens) and irradiated/stressed (creep) specimens of the six grades of graphite examined.

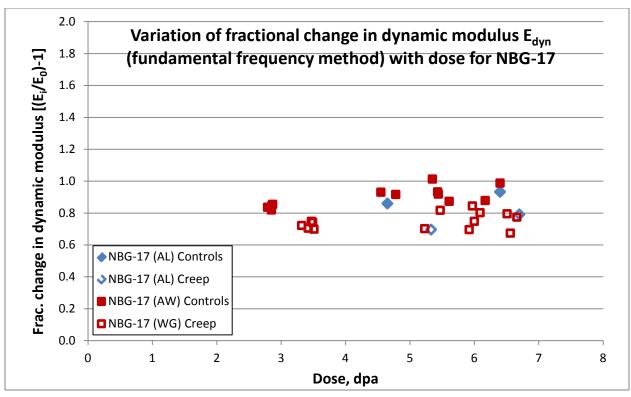


Figure 26. Grade A, NBG-17 (AL) and (AW) fractional changes in dynamic elastic modulus for control and creep (stressed) specimens.

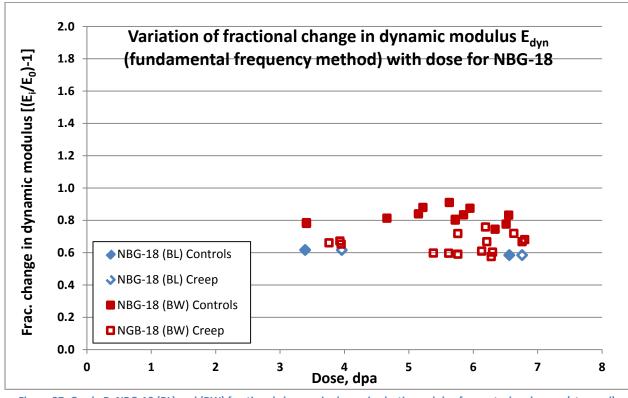


Figure 27. Grade B, NBG-18 (BL) and (BW) fractional changes in dynamic elastic modulus for control and creep (stressed) specimens).

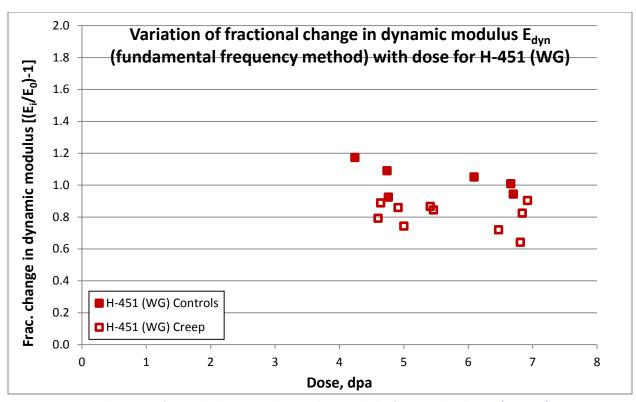


Figure 28. Grade C, H-451 fractional changes in dynamic elastic modulus for control and creep (stressed) specimens.

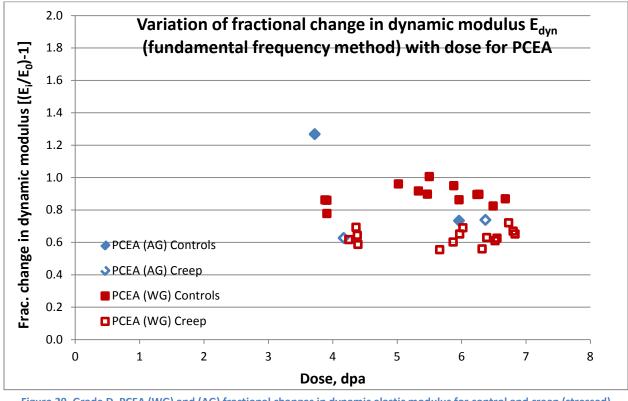


Figure 29. Grade D, PCEA (WG) and (AG) fractional changes in dynamic elastic modulus for control and creep (stressed) specimens.

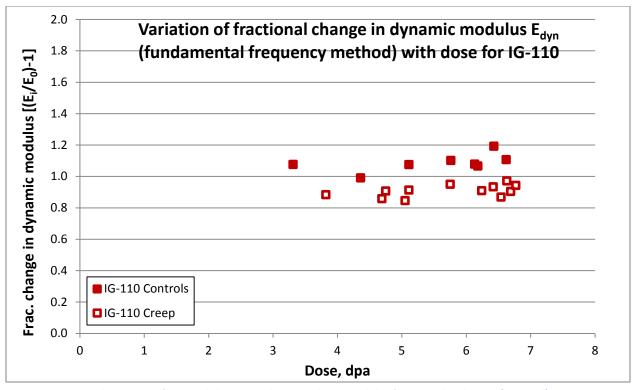


Figure 30. Grade E, IG-110 fractional changes in dynamic elastic modulus for control and creep (stressed) specimens.

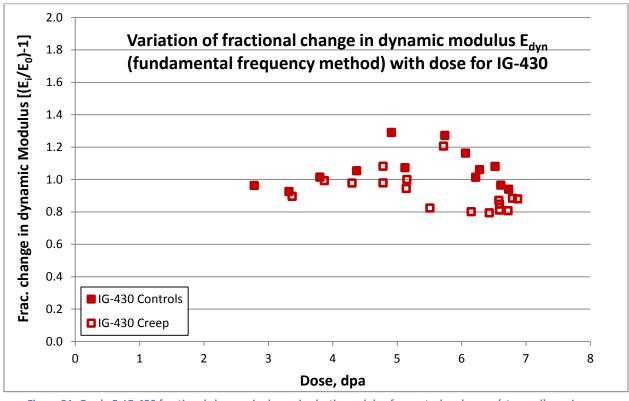


Figure 31. Grade F, IG-430 fractional changes in dynamic elastic modulus for control and creep (stressed) specimens.

The largest dynamic modulus (by ff) fractional changes due to irradiation were observed for the isostatically-pressed grade IG-430 and the smallest was for the extruded grade PCEA (AG) (Section 3.1.1). Examination of the fractional change plots in Figure 26 to Figure 31 indicates the fractional changes for specimens subjected to irradiation creep appear slightly lower than the fractional changes measured in control specimens for all graphite grades and orientations. This small difference in modulus may occur from either crystallite changes due to the additional plastic strain occurring during irradiation creep, or it may be a macroscopic controlled effect resulting from pore and grain rearrangement or large pore closures during irradiation creep. Additional analysis from future AGC irradiation test trains, as well as ongoing fundamental irradiation damage studies, will assist in further improving our understanding the underlying mechanisms responsible for this material property change. Statistical testing is applied to the modulus change (measured by the ff method) for irradiated, creep, and control specimens to determine whether the differences in the test condition outcomes were quantitatively different.

4.1.1 Dynamic Elastic Modulus Statistical Testing (by fundamental frequency method)

To establish whether there was a quantitative difference in the creep and control specimen dynamic modulus, statistical significance testing^{11,12} of the dynamic modulus data was conducted, (E_{dyn} , measured by ff method). To analyze the dynamic modulus data, a series of significance questions (Section 2.4.2) were posed (Table 5).

Table 5. Statistical significance test questions.

Question Number	Question
	Is the irradiated modulus of control specimen greater than unirradiated
Question/Test i	specimen modulus for grade?
	Is the irradiated modulus of creep specimens greater than unirradiated
Question/Test ii	specimen modulus for grade?
Question/Test iii	Is the elastic modulus of the creep and control specimens different?
	Are control specimen modulus doses and creep specimen modulus doses
Question/Test iv	similar?

Table 6. Unirradiated dynamic elastic modulus means (ff method) and the dynamic elastic modulus (ff method) of the three highest dose creep and control specimens for each grade of graphite in AGC-1.

highest dose creep and control specimens for each grade of graphite in AGC-1.								
	Grade		Irrad	liated Modu	ulus Statistics for "t" Testing			
				Controls (3 highest				
			dose	e)	Creep	Creep (3 highest dose)		
		Unirradiated	Madulua	Daga	Madulija	Daga	Creen	
ID	NAME	Modulus, (ff) GPa	Modulus, GPa	Dose, dpa	Modulus, GPa	Dose, dpa	Creep Strain, %	
		` '		•		•		
A	NBG-17 (AW)	n=30	21.02	6.17	20.09	6.51	-1.42	
			20.96	5.61	18.73	6.65	-1.63	
			22.25	6.40	19.85	6.66	-2.00	
	MEAN	11.19	21.41	6.06	19.56	6.61	-1.68	
	S.D.	0.093	0.73	0.41	0.73	0.08	0.29	
В	NBG-18 (WG)	n=30	21.62	6.09	20.93	6.63	-1.35	
			21.23	6.66	20.46	6.8	-1.72	
			22.30	6.70	20.29	6.76	-2.03	
	MEAN	12.17	21.72	6.48	20.56	6.73	-1.70	
	S.D.	0.072	0.54	0.34	0.33	0.09	0.34	
С	H-451(BW)	n=22	18.13	6.09	16.83	6.92	-2.10	
			17.76	6.66	14.52	6.81	-3.09	
			17.18	6.77	16.12	6.84	-1.89	
	MEAN	8.84	17.69	6.51	15.82	6.86	-2.36	
	S.D.	0.378	0.48	0.37	1.19	0.06	0.64	
D	PCEA(WG)	n=33	20.82	6.27	18.89	6.73	-1.63	
			20.03	6.49	18.32	6.8	-2.04	
			20.52	6.68	18.13	6.83	-2.42	
	MEAN	10.98	20.46	6.48	18.45	6.79	-2.03	
	S.D.	0.149	0.40	0.21	0.39	0.05	0.40	
Е	IG-110	n=22	18.35	6.18	16.91	6.69	-2.76	
<u> </u>	10 110	11-22	19.47	6.43	17.25	6.77	-2.53	
			18.71	6.62	17.50	6.63	-1.94	
	MEAN	8.88	18.84	6.41	17.22	6.70	-2.41	
	S.D.	0.218	0.57	0.41	0.30	0.07	0.42	
F	IG-430	n=34	19.73	6.52	17.83	6.87	-2.56	
 	10 400	11-54	18.63	6.61	17.13	6.72	-1.75	
			18.39	6.73	17.13	6.79	-1.75	
	MEAN	9.48	18.92	6.62	17.61	6.79	-1.70 -2.00	
	IVILAIN	9.40	10.92	0.02	17.01	0.79	-2.00	
	S.D.	0.261	0.71	0.11	0.41	0.08	0.48	

Before significance testing of the specimen dynamic elastic modulus (ff) could commence it was necessary to establish whether the chosen creep and control specimens had comparable irradiation doses. An unpaired student's "t" test was employed to test the statistical significance of the differences (Table 5, Question iv).

Table 7. High dose "t" test outcomes.

	UNPAIRED "t" TEST RESULTS FOR DOSE								
Grade	Test	Statistics Parameter, Two-Tailed "P"	Two-Tailed Test Result						
A(WG)	iv	0.0847	Difference is not quite statistically significant						
B(WG)	iv	0.0676	Difference is not quite statistically significant						
C(WG)	iv	0.1293	Difference is not statistically significant						
D(WG)	iv	0.0677	Difference is not quite statistically significant						
E	iv	0.0952	Difference is not quite statistically significant						
F	iv	0.0964	Difference is not quite statistically significant						

In each of the cases tested, the difference in the doses was found to be not statistically significant or not quite statistically significant (Table 7). Since the physical property values to be significance tested are a function of neutron dose, it was imperative to establish that the doses are comparable. Essentially, we may consider the highest doses of the creep and control specimens of each graphite grade to be similar.

After establishing the comparability of the dose levels between the creep and control specimens, the elastic modulus data (Table 6) was significance tested. The results of dynamic elastic modulus statistical significance testing (high dose) are given in Table 8.

Table 8. Significance testing results for dynamic elastic modulus (ff method) at high dose.

J J J J J J J J J J J J J J J J J J J							
	UNPA	IRED "t" TEST	RESULTS FOR DYNAMIC ELASTIC MODULUS				
Grade	Test	Statistics Parameter, Two-Tailed "P"	Two-Tailed Test Result				
A(AW)	i	<0.0001	Difference is extremely statistically significant				
A(AW)	ii	<0.0001	Difference is extremely statistically significant				
A(AW)	iii	0.0361	Difference is statistically significant				
B(BW)	i	<0.0001	Difference is extremely statistically significant				
B(BW)	ii	<0.0001	Difference is extremely statistically significant				
B(BW)	iii	0.0377	Difference is statistically significant				
C(WG)	i	<0.0001	Difference is extremely statistically significant				
C(WG)	ii	<0.0001	Difference is extremely statistically significant				
C(WG)	iii	0.0651	Difference is not statistically significant				
D(WG)	i	<0.0001	Difference is extremely statistically significant				
D(WG)	ii	<0.0001	Difference is extremely statistically significant				
D(WG)	iii	0.0034	Difference is very statistically significant				
Е	i	<0.0001	Difference is extremely statistically significant				
Е	ii	<0.0001	Difference is extremely statistically significant				
Е	iii	0.0121	Difference is statistically significant				
F	i	<0.0001	Difference is extremely statistically significant				
F	ii	<0.0001	Difference is extremely statistically significant				
F	iii	0.0505	Difference is not quite statistically significant				

In all cases, the dynamic elastic modulus of the irradiated (control) or irradiated/stressed (creep) specimen was significantly greater than the unirradiated dynamic elastic modulus (Questions i and ii, Table 5). This clearly indicates that irradiation increases the dynamic elastic modulus (Table 8). In only one of the six cases tested (H-451) was the difference between the two data sets, i.e., the creep and control specimens "not statistically significant" (Table 5, Question iii). In the remaining five cases, the difference was "statistically significant" or in the case of grade, IG-430 the difference was "not quite statistically significant" (Table 8). This suggests that irradiation creep has a small additional effect on the dynamic elastic modulus over the dose range examined in AGC-1 (i.e., a slight reduction in E_{dyn} from creep).

The initial increase in modulus (low dose level) has previously been reported and attributed to dislocation line pinning by irradiation induced defects in the graphite crystallite regions ^{13,14,15,16,17}. Subsequent changes in modulus can also occur due to strain induced structure (porosity, re-orientation, etc.) changes in the bulk graphite ¹³⁻¹⁷.

Because of the potential for pore generation at higher doses, the means of the three modulus data points at the lowest dose were also statistically tested to establish if there was significance in the

modulus changes as a result of the additional effect of creep strain. The modulus and dose of the three lowest dose specimens are given in Table 9, and the modulus and dose of the three highest dose specimens are given in Table 6.

Table 9 Unirradiated dynamic elastic modulus means (ff method) and the dynamic elastic modulus (ff method) of the three lowest dose creep and control specimens for each grade of graphite in AGC-1.

Grade lowest dose creep and control specimens for each grade of graphite in AGC-1. Grade Irradiated Modulus Statistics for "t" Testing								
-	Graue	Irradiated Modulus Statistics for "t" Testing Controls (3 lowest						
			dose			ep (3 lowest dose)		
			uosi	-) 	Cleek	J (3 lowest	l dose)	
		Unirradiated	Modulus,	Dose,	Modulus,	Dose,	Creep	
ID	NAME	Modulus, (ff) GPa	GPa	dpa	GPa	dpa	Strain, %	
			<u> </u>		0.1.0.			
Α	NBG-17 (AW)	n=30	20.55	2.79	19.27	3.32	-0.69	
			20.36	2.85	19.56	3.47	-0.72	
			20.71	2.86	19.08	3.42	-1.10	
	MEAN	11.19	20.54	2.83	19.30	3.40	-0.84	
	S.D.	0.093	0.18	0.04	0.24	0.08	0.23	
В	NBG-18 (WG)	n=30	21.51	3.24	20.21	3.76	-0.62	
	, ,		21.67	3.41	20.34	3.93	-0.72	
			21.73	3.41	20.12	3.95	-0.90	
	MEAN	12.17	21.64	3.35	20.22	3.88	-0.75	
	S.D.	0.072	0.11	0.10	0.11	0.10	0.14	
С	H-451(BW)	n=22	19.21	4.24	15.84	4.60	-1.19	
			18.48	4.74	16.69	4.64	-1.42	
			17.01	4.76	16.44	4.91	-1.21	
	MEAN	8.84	18.23	4.58	16.32	4.72	-1.27	
	S.D.	0.378	1.12	0.29	0.44	0.17	0.13	
D	PCEA(WG)	n=33	20.44	3.88	18.58	4.36	-0.95	
	1 CLA(VVO)	11=55	20.44	3.91	17.75	4.25	-1.25	
			19.52	3.91	18.04	4.38	-1.32	
	MEAN	10.98	20.13	3.90	18.12	4.33	-1.17	
	S.D.	0.149	0.44	0.90	0.42	0.07	0.20	
E	IG-110	n=22	18.43	3.31	16.72	3.82	-1.31	
			17.67	4.36	16.50	4.69	-1.92	
			18.43	5.11	16.94	4.75	-1.21	
	MEAN	8.88	18.18	4.26	16.72	4.42	-1.48	
	S.D.	0.218	0.44	0.90	0.22	0.52	0.38	
F	IG-430	n=34	18.61	2.78	17.98	3.37	-0.92	
			18.26	3.32	18.89	3.87	-1.21	
			19.10	3.80	18.75	4.30	-1.35	
	MEAN	9.48	18.66	3.30	18.54	3.85	-1.16	
	S.D.	0.261	0.42	0.51	0.49	0.47	0.22	

Before significance testing of the specimen dynamic elastic modulus could commence (at low dose), it was necessary to establish whether the chosen creep and control specimens had comparable irradiation doses. An unpaired student's "t" test was employed to test the statistical significance of the differences (Table 5, Question iv).

Table 10 gives the results for the t-testing of the low neutron dose means. In the case of the NBG grades (A and B), the dose mean of the low dose data was statistically significantly different, and thus the modulus data could not be compared. In the case of the remaining four grades (H-451(C), PCEA(D), IG-110(E) and IG-430(F)), the low dose means had differences that were not statistically significant and the dynamic elastic modulus data could be compared. After establishing which doses are comparable, the data in Table 9 was statistically tested. The results of dynamic elastic modulus statistical significance testing (at low dose) are given in Table 11.

Table 10. Low dose "t" test outcomes.

	Table 10. Low dose it test outcomes.								
	UNPAIRED "t" TEST RESULTS FOR DOSE								
Grade	Test	Statistics Parameters, Two-Tailed "P"	Two-Tailed Test Result						
A(AW)	iv	0.0004	Difference is extremely statistically significant						
B(AW)	iv	0.0029	Difference is very statistically significant						
C(WG)	iv	0.5106	Difference is not statistically significant						
D(WG)	iv	0.4557	Difference is not statistically significant						
Е	iv	0.8029	Difference is not statistically significant						
F	iv	0.2415	Difference is not statistically significant						

Table 11. Significance testing results for dynamic elastic modulus at low dose.

L	UNPAIRED "t" TEST RESULTS FOR DYNAMIC ELASTIC MODULUS									
Grade	Test	Statistics Parameters, Two-Tailed "P"	Two-Tailed Test Result							
A(AW)	ii		Cannot test							
B(BW)	ii		Cannot test							
C(WG)	ii	0.0514	Difference is not quite statistically significant							
D(WG)	ii	0.0046	Difference is very statistically significant							
Е	ii	0.0068	Difference is very statistically significant							
F	ii	0.7635	Difference is not statistically significant							

Two of the four cases that could be tested at low dose were found to have a statistically significant difference between the creep dynamic elastic modulus and the control dynamic elastic modulus, whereas two did not have statistically significant differences. This result is more equivocal than the high

dose statistical "t" testing and suggests the differences between the creep and control specimen modulus became more apparent at high doses (Table 8).

4.2 Irradiated Dynamic Elastic Modulus (by ultrasonic velocity/time of flight)

As explained in Section 4.1, the differences in the relative changes in dynamic elastic modulus between control and creep specimens are more easily illustrated if fractional changes in modulus are calculated and plotted as a function of dose. The fractional change in modulus was defined in Equation 1. Figure 32 to Figure 37 show the dynamic elastic modulus (ultrasonic velocity/tof) fractional changes for the various grades in AGC-1.

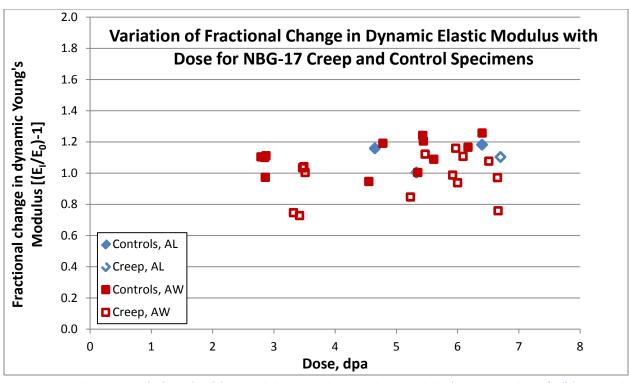


Figure 32. Grade A, NBG-17 (AL) and (AW) fractional changes in dynamic elastic modulus (ultrasonic velocity/tof) for control and creep (stressed) specimens.

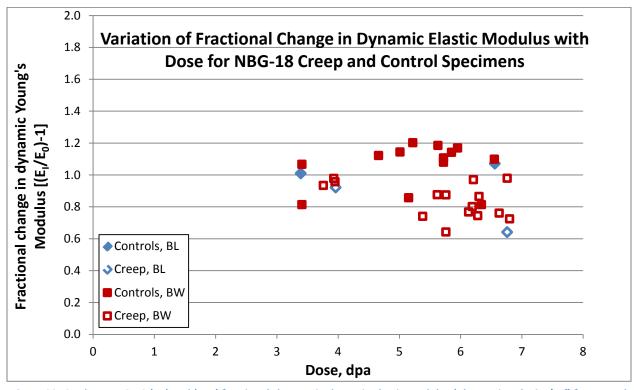


Figure 33. Grade B, NBG-18 (AL) and (AW) fractional changes in dynamic elastic modulus (ultrasonic velocity/tof) for control and creep (stressed) specimens.

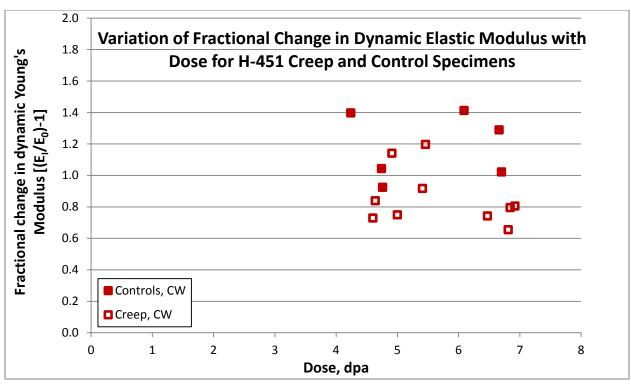


Figure 34. Grade C, H-451 fractional changes in dynamic elastic modulus (ultrasonic velocity/tof) for control and creep (stressed) specimens.

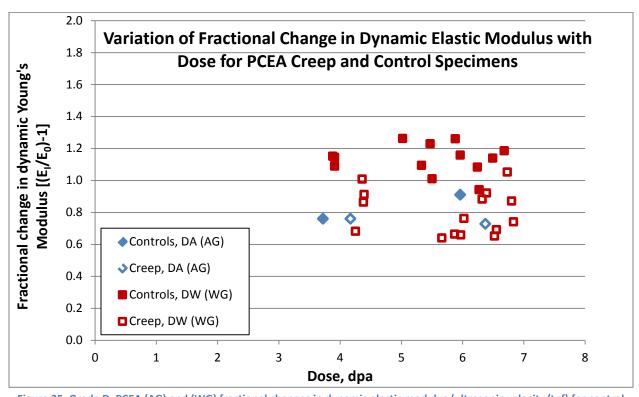


Figure 35. Grade D, PCEA (AG) and (WG) fractional changes in dynamic elastic modulus (ultrasonic velocity/tof) for control and creep (stressed) specimens.

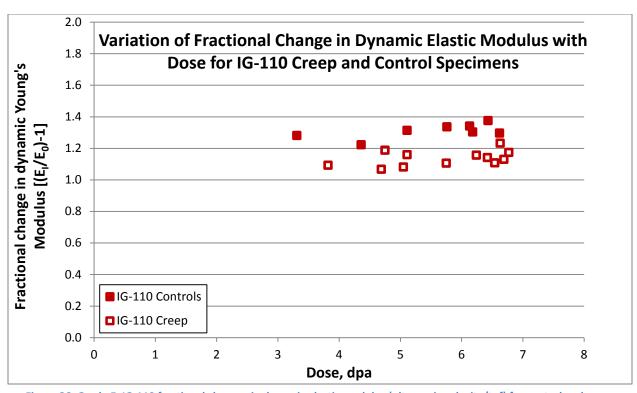


Figure 36. Grade E, IG-110 fractional changes in dynamic elastic modulus (ultrasonic velocity/tof) for control and creep (stressed) specimens.

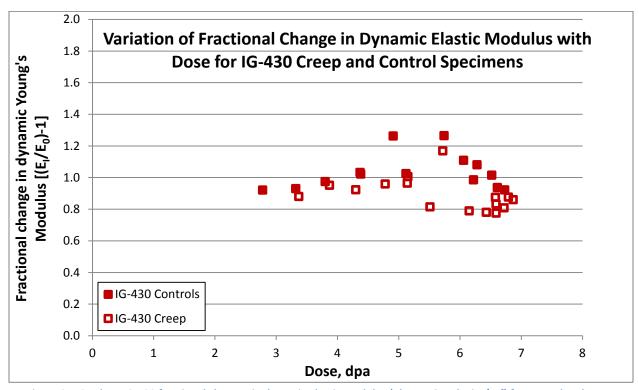


Figure 37. Grade F, IG-430 fractional changes in dynamic elastic modulus (ultrasonic velocity/tof) for control and creep (stressed) specimens.

The largest dynamic modulus (by ultrasonic velocity/tof) fractional changes were observed for the extruded grade H-451 WG control specimens (Figure 34) and the smallest were observed for the vibro-molded grade NBG-18 (BW) controls (Figure 32). Examination of the fractional change plots in Figure 32 to Figure 37 indicates the fractional changes were smaller for the creep specimens than for the control specimens for all grades and orientations.

4.2.1 Dynamic Elastic Modulus Statistical Testing (by ultrasonic velocity/time of flight)

To establish whether there was a measurable statistical difference between the creep and control specimen dynamic modulus values (by ultrasonic velocity/tof), statistical significance testing ^{11,12} of the dynamic modulus data was conducted. To analyze the dynamic modulus data (by ultrasonic velocity/tof), a series of significant questions (Section 2.4.2) were posed (Table 5).

The modulus (tof) and dose of the three highest dose specimens are given in Table 12.

Table 12. Unirradiated dynamic elastic modulus means (ultrasonic/tof method) and the dynamic elastic modulus (ultrasonic/tof method) of the three highest dose creep and control specimens for each grade of graphite in AGC-1.

	Grade	3	Irradiated Modulus Statistics for "t" Testing					
			Controls (3	_				
		Unirradiated	dose	e) 	Creep	eep (3 highest dose)		
		Modulus, GPa						
			Modulus,	Dose,	Modulus,	Dose,	Creep	
ID	Name		GPa	dpa	GPa	dpa	Strain, %	
Α	NBG-17	n=30	20.76	5.61	20.64	6.51	-1.42	
			21.53	6.17	19.59	6.65	-1.63	
			22.43	6.40	17.48	6.66	-2.00	
	MEAN	9.94	21.57	6.06	19.24	6.61	-1.68	
	S.D.	0.100	0.83	0.41	1.61	0.08	0.29	
В	NBG-18	n=30	23.12	5.95	18.77	6.63	-1.35	
			19.34	6.34	18.38	6.8	-1.72	
			22.37	6.55	21.10	6.76	-2.03	
	MEAN	10.66	21.61	6.28	19.42	6.73	-1.70	
	S.D.	0.102	2.00	0.30	1.47	0.09	0.34	
С	H-451(WG)	n=22	18.25	6.66	14.39	6.92	-2.10	
			16.11	6.70	13.19	6.81	-3.09	
			19.23	6.09	14.31	6.84	-1.89	
	MEAN	7.97	17.86	6.48	13.96	6.86	-2.36	
	S.D.	0.102	1.60	0.34	0.67	0.06	0.64	
D	PCEA(WG)	n=31	18.28	6.27	19.31	6.73	-1.63	
			20.13	6.49	17.61	6.8	-2.04	
			20.56	6.68	16.39	6.83	-2.42	
	MEAN	9.41	19.66	6.48	17.77	6.79	-2.03	
	S.D.	0.152	1.21	0.21	1.47	0.05	0.40	
Е	IG-110	n=22	18.52	6.18	17.95	6.63	-1.94	
			19.10	6.43	17.13	6.69	-2.76	
			18.46	6.62	17.48	6.77	-2.53	
	MEAN	8.04	18.69	6.41	17.52	6.70	-2.41	
	S.D.	0.108	0.35	0.22	0.41	0.07	0.42	
F	IG-430	n=34	19.44	6.52	17.95	6.87	-2.56	
			18.70	6.61	17.46	6.72	-1.75	
			18.55	6.73	18.11	6.79	-1.70	
	MEAN	9.65	18.90	6.62	17.84	6.79	-2.00	
	S.D.	0.176	0.48	0.11	0.34	0.08	0.48	

Before significance testing of the specimen tof dynamic elastic modulus could commence, it was necessary to establish whether the chosen creep and control specimens had comparable irradiation doses. An unpaired student's "t" test was employed to test the statistical significance of the differences (Table 5, Question iv). Table 13 gives the results for the t-testing of the neutron doses.

Table 13. Dose "t" test outcomes.

	UNPAIRED "t" TEST RESULTS FOR DOSE							
		Statistics						
		Parameters						
Grade	Grade Test Two-Tailed "P" Two-Tailed Test Result							
A(AW)	iv	0.0973	Difference is not quite statistically significant					
B(AW)	iv	0.0676	Difference is not quite statistically significant					
C(WG)	iv	0.7488	Difference is not statistically significant					
D(WG)	iv	0.1987	Difference is not statistically significant					
Е	iv	0.0952	Difference is not quite statistically significant					
F	iv	0.0964	Difference is not quite statistically significant					

In each of the cases tested, the difference in the doses was not statically significant or not quite statistically significant. Since the physical property values to be significance tested are a function of neutron dose (and dose inflicted structural damage), it was imperative to establish the doses are comparable. Hence, the highest doses of the creep and control specimens of each graphite grade may be considered similar.

After establishing the doses were comparable, the data in Table 12 was statistically tested. The results of dynamic elastic modulus (velocity/tof) statistical significance testing are given in Table 14.

In all cases, the tof dynamic elastic modulus of the irradiated (control) and the irradiated/stressed (creep) specimen was significantly greater than the unirradiated dynamic elastic modulus (Questions i and ii). This clearly indicates that irradiation increases the dynamic elastic modulus.

As previously discussed, the initial increase in modulus (at lower dose levels) has been attributed to dislocation line pinning by irradiation induced defects in the graphite crystallite regions¹³⁻¹⁷. Subsequent changes in modulus occur due to strain induced structure (porosity) changes in the bulk graphite¹³⁻¹⁷.

From Table 14, only H-451, IG-110, and IG-430 graphite grades showed a significant difference between the two data sets (Table 5, Question iii). In the remaining three cases, the difference between the control and creep specimen values was not statistically significant. This and the ff modulus data suggest that irradiation creep has a small grade-specific, additional effect (a small reduction) on the dynamic elastic modulus over the dose range examined in AGC-1.

Table 14. Significance testing results for dynamic elastic modulus (velocity/tof) at high dose.

	UNPAIRED "t" TEST RESULTS FOR DYNAMIC ELASTIC MODULUS (ULTRASONIC/TOF METHOD)								
Grade	Test	Statistics Parameter Two- Tailed "P"	Two-Tailed Test Result						
A(WG) A(WG)	i ii	<0.0001 <0.0001	Difference is extremely statistically significant Difference is extremely statistically significant						
A(WG)	iii	0.0898	Difference is not quite statistically significant						
B(WG)	i	<0.0001	Difference is extremely statistically significant						
B(WG)	ii	<0.0001	Difference is extremely statistically significant						
B(WG)	iii	0.2012	Difference is not statistically significant						
C(WG)	i	<0.0001	Difference is extremely statistically significant						
C(WG)	ii	<0.0001	Difference is extremely statistically significant						
C(WG)	iii	0.0176	Difference is statistically significant						
D(WG)	i	<0.0001	Difference is extremely statistically significant						
D(WG)	ii	<0.0001	Difference is extremely statistically significant						
D(WG)	iii	0.1607	Difference is not statistically significant						
E	i	<0.0001	Difference is extremely statistically significant						
Е	ii	<0.0001	Difference is extremely statistically significant						
E	iii	0.0198	Difference is statistically significant						
F	i	<0.0001	Difference is extremely statistically significant						
F	ii	<0.0001	Difference is extremely statistically significant						
F	iii	0.0355	Difference is statistically significant						

In order for E_{dyn} creep to be less than the E_{dyn} control, it is necessary to postulate the formation of additional pores/cracks in the creep specimens that are aligned in such a way that the coefficient of thermal expansion (CTE) is not affected and that E_{dyn} is reduced for creep specimens. Such pores might also exist on the control samples, but the effect is more pronounced in the case of compressive creep. In a creep sample, more non CTE impacting porosity is created. This would tend to delay the volume turnaround for the case of compressive creep, as has been speculated, but is in disagreement with prior observations that creep appears to be volume conserving in the AGC-1 dose range⁶. Unfortunately, the AGC-1 dose range is insufficient to attain volume turn-around.

5 Quality Assurance

The described technical work scope and related activities were conducted in accordance with the applicable quality assurance requirements of ASME NQA-1-2008/1a-2009, *Quality Assurance Requirements for Nuclear Facilities*. Work scope at ORNL was performed under the activity-specific requirements of the applicable ORNL nuclear R&D quality assurance plan (QAP) numbered QAP-ORNL-NR&D-01 and entitled *Quality Assurance Plan for Nuclear Research and Development Conducted at the Oak Ridge National Laboratory*. This QAP is based on and compliant with ASME NQA-1-2008/1a-2009.

6 Conclusions

Regardless of measurement technique, the value of dynamic elastic modulus (E_{dyn}) increases substantially (by approximately 70–100%) after irradiation at AGC-1 dose range levels. The initial increase in E_{dyn} has previously been attributed to an irradiation-induced defect acting as additional pinning points that impede dislocation movement through the graphite crystallites¹³⁻¹⁷. Under current theory, any subsequent E_{dyn} increase or decrease with additional dose are understood to be a result of the changes to the pore structure and correlate roughly with the graphite volume change behavior¹³⁻¹⁷. In the dose range of AGC-1, volume turnaround is not achieved (with the possible exception of IG-430), so the E_{dyn} value is not expected to display this high dose drop in modulus.

Compressive creep strain does appear to have a small effect on dynamic elastic modulus, reducing the modulus at the higher doses as volume turnaround is approached. However, statistical testing showed mixed results in the difference of dose effects on dynamic elastic modulus in the case of creep versus control specimens. This was true for both dynamic elastic modulus test methods. The high dose ff test results showed that at higher doses, the difference between creep $E_{\rm dyn}$ and control $E_{\rm dyn}$ was significant for four of the six graphite grades. These results are supported by the outcome of ultrasonic velocity/tof testing, where the two grades, H-451(C) and IG-430(F), not significantly different by ff testing, were found to be significantly different by tof testing.

7 Acknowledgements

This work is sponsored by the U.S. Department of Energy, Office of Nuclear Energy Science and Technology under a Memorandum Purchase Order between Idaho National Laboratory (managed by Battelle Energy Alliance, LLC) and Oak Ridge National Laboratory (managed by UT-Battelle, LLC).

The author wishes to thank Ashli Clark for her diligence performing AGC-1 experimental measurements.

8 Appendix

8.1 Dynamic Elastic Modulus (by fundamental frequency)

Table 15. Grade A, NBG-17 graphite mean dynamic elastic modulus (fundamental frequency method) for creep (stressed) and control samples.

Sample ID	GRADE	DOSE dpa	Irr. Temp °C	SPEC TYPE	CREEP STRAIN %	E _{dyn} (GPa)	Fractional change in Modulus [(Ei/E0)-1]
AL8-02	NBG-17 AL	4.65	585	Control	0.00	21.54	0.86
AL6-02 AL6-01	NBG-17 AL	6.40	667	Control	0.00	22.38	0.93
AL8-01	NBG-17 AL	5.33	655	Creep	-1.42	19.64	0.93
AL6-02	NBG-17 AL	6.70	703	Creep	-1.61	20.74	0.79
ALO-02	NDO-17 AL	0.70	703	Огеер	-1.01	20.74	0.13
AW6-01	NBG-17 AW	2.79	472	Control	0.00	20.55	0.84
AW13-01	NBG-17 AW	2.85	470	Control	0.00	20.36	0.82
AW5-01	NBG-17 AW	2.86	470	Control	0.00	20.71	0.85
AW2-03	NBG-17 AW	2.87	468	Control	0.00	20.75	0.85
AW7-01	NBG-17 AW	4.55	587	Control	0.00	21.60	0.93
AW4-03	NBG-17 AW	4.78	582	Control	0.00	21.45	0.92
AW5-03	NBG-17 AW	5.35	597	Control	0.00	22.52	1.01
AW12-03	NBG-17 AW	5.43	594	Control	0.00	21.62	0.93
AW2-02	NBG-17 AW	5.44	592	Control	0.00	21.47	0.92
AW10-01	NBG-17 AW	5.61	617	Control	0.00	20.96	0.87
AW2-01	NBG-17 AW	6.17	650	Control	0.00	21.02	0.88
AW9-03	NBG-17 AW	6.40	678	Control	0.00	22.25	0.99
AW6-03	NBG-17 AW	3.32	594	Creep	-0.69	19.27	0.72
AW1-03	NBG-17 AW	3.47	589	Creep	-0.72	19.56	0.75
AW4-02	NBG-17 AW	3.49	592	Creep	-0.90	19.49	0.74
AW6-02	NBG-17 AW	5.23	656	Creep	-1.05	19.04	0.70
AW12-01	NBG-17 AW	3.51	593	Creep	-1.09	19.01	0.70
AW5-02	NBG-17 AW	3.42	594	Creep	-1.10	19.08	0.70
AW1-02	NBG-17 AW	5.97	668	Creep	-1.13	20.63	0.84
AW4-01	NBG-17 AW	5.47	653	Creep	-1.28	20.33	0.82
AW1-01	NBG-17 AW	6.51	690	Creep	-1.42	20.09	0.80
AW9-01	NBG-17 AW	6.09	677	Creep	-1.47	20.16	0.80
AW7-03	NBG-17 AW	6.56	712	Creep	-1.62	18.73	0.67
AW13-02	NBG-17 AW	5.92	674	Creep	-1.85	18.98	0.70
AW10-03	NBG-17 AW	6.00	670	Creep	-1.89	19.57	0.75
AW10-02	NBG-17 AW	6.66	703	Creep	-2.01	19.85	0.77

Table 16. Grade B, NBG-18 graphite mean dynamic elastic modulus (fundamental frequency method) for creep (stressed) and control samples.

Table 101 Grade 1	, ribe 10 grapinie	The array name etc.	The modulus (rundu	mentar mege	lency methody re	r creep (stressed)	Fractional
Sample ID	GRADE	DOSE dpa	Irr. Temp °C	SPEC	CREEP	E _{dyn} (GPa)	change in
Sample 10	UNADL	DOSE upa	iii. reiiip C	TYPE	STRAIN %	L _{dyn} (GFa)	Modulus
							[(Ei/E0)-1]
BL7-02	NBG-18 BL	3.39	505	Control	0	22.26	0.79
BL6-02	NBG-18 BL	6.56	678	Control	0	22.29	0.79
BL7-01	NBG-18 BL	3.96	559	Creep	-1.06	20.14	0.62
BL6-03	NBG-18 BL	6.76	714	Creep	-2.07	19.74	0.58
BW8-03	NBG-18 BW	3.24	509	Control	0	21.51	
BW1-02	NBG-18 BW	3.41	504	Control	0	21.67	0.78
BW11-02	NBG-18 BW	3.41	505	Control	0	21.73	0.79
BW7-01	NBG-18 BW	4.66	585	Control	0	22.06	0.81
BW10-02	NBG-18 BW	5.01	585	Control	0	22.17	
BW3-01	NBG-18 BW	5.15	582	Control	0	22.39	0.84
BW8-02	NBG-18 BW	5.22	599	Control	0	22.87	0.88
BW5-03	NBG-18 BW	5.63	617	Control	0	23.25	0.91
BW5-02	NBG-18 BW	5.72	611	Control	0	21.99	0.81
BW12-01	NBG-18 BW	5.72	613	Control	0	21.92	0.80
BW10-01	NBG-18 BW	5.85	638	Control	0	22.32	0.83
BW8-01	NBG-18 BW	5.95	658	Control	0	22.82	0.88
BW1-03	NBG-18 BW	6.34	664	Control	0	21.23	0.74
BW11-03	NBG-18 BW	6.51	674	Control	0	21.62	0.78
BW2-03	NBG-18 BW	6.55	675	Control	0	22.30	0.83
BW7-03	NBG-18 BW	3.76	603	Creep	-0.62	20.21	0.66
BW12-02	NBG-18 BW	3.93	597	Creep	-0.72	20.34	0.67
BW9-03	NBG-18 BW	3.95	599	Creep	-0.90	20.12	0.65
BW3-02	NBG-18 BW	6.19	671	Creep	-1.19	21.40	0.76
BW7-02	NBG-18 BW	6.30	698	Creep	-1.20	19.52	0.60
BW1-01	NBG-18 BW	6.63	700	Creep	-1.35	20.93	0.72
BW9-02	NBG-18 BW	5.62	668	Creep	-1.37	19.43	0.60
BW2-02	NBG-18 BW	5.76	665	Creep	-1.38	20.92	0.72
BW12-03	NBG-18 BW	5.76	675	Creep	-1.48	19.35	0.59
BW9-01	NBG-18 BW	6.28	686	Creep	-1.49	19.17	0.58
BW5-01	NBG-18 BW	5.38	656	Creep	-1.64	19.44	0.60
BW2-01	NBG-18 BW	6.80	709	Creep	-1.72	20.46	0.68
BW11-01	NBG-18 BW	6.21	674	Creep	-1.80	20.29	0.67
BW3-03	NBG-18 BW	6.13	678	Creep	-1.82	19.59	0.61
BW10-03	NBG-18 BW	6.76	709	Creep	-2.03	20.29	0.67

Table 17. Grade C, H-451 graphite mean dynamic elastic modulus (fundamental frequency method) for creep (stressed) and control samples.

Sample ID	GRADE	DOSE dpa	Irr. Temp °C	SPEC TYPE	CREEP STRAIN %	E _{dyn} (GPa)	Fractional change in Modulus [(Ei/E0)-1]
CW13-01	H-451 (WG)	4.24	567	Control	0	19.21	1.17
CW14-02	H-451 (WG)	4.74	582	Control	0	18.48	1.09
CW8-03	H-451 (WG)	4.76	580	Control	0	17.01	0.92
CW10-03	H-451 (WG)	6.09	657	Control	0	18.13	1.05
CW10-02	H-451 (WG)	6.66	681	Control	0	17.76	1.01
CW8-02	H-451 (WG)	6.70	674	Control	0	17.18	0.94
CW11-02	H-451 (WG)	4.60	628	Creep	-1.19	15.84	0.79
CW11-01	H-451 (WG)	4.91	642	Creep	-1.21	16.44	0.86
CW7-03	H-451 (WG)	5.41	649	Creep	-1.35	16.50	0.87
CW14-01	H-451 (WG)	4.64	627	Creep	-1.42	16.69	0.89
CW12-02	H-451 (WG)	5.00	641	Creep	-1.79	15.41	0.74
CW7-01	H-451 (WG)	6.84	706	Creep	-1.89	16.12	0.82
CW13-02	H-451 (WG)	6.92	708	Creep	-2.10	16.83	0.90
CW13-03	H-451 (WG)	5.46	653	Creep	-2.25	16.31	0.84
CW10-01	H-451 (WG)	6.47	697	Creep	-2.61	15.20	0.72
CW9-03	H-451 (WG)	6.81	712	Creep	-3.09	14.52	0.64

Table 18. Grade D, PCEA graphite mean dynamic elastic modulus (fundamental frequency method) for creep (stressed) and control samples.

					ncy method/for cree	(0.0000)	Fractional
Sample ID	GRADE	DOSE dna	Irr. Temp °C	SPEC TYPE	CREEP	E _{dyn} (GPa)	change in
	CIVIDE	BOSE apa		31 20 111 2	STRAIN %	Layn (Ci a)	Modulus
							[(Ei/E0)-1]
DA702	PCEA-AG	3.72	538	Control	0	20.36	1.27
DA601	PCEA-AG	5.96	632	Control	0	15.56	0.73
DA701	PCEA-AG	4.17	612	Creep	-1.17	14.60	0.63
DA602	PCEA-AG	6.37	680	Creep	-1.88	15.61	0.74
DW10-02	PCEA-WG	3.88	534	Control	0	20.44	0.86
DW2-03	PCEA-WG	3.91	533	Control	0	20.42	0.86
DW5-01	PCEA-WG	3.91	534	Control	0	19.52	0.78
DW6-01	PCEA-WG	5.02	585	Control	0	21.53	0.96
DW9-01	PCEA-WG	5.33	597	Control	0	21.05	0.92
DW4-01	PCEA-WG	5.47	594	Control	0	20.83	0.90
DW7-02	PCEA-WG	5.50	618	Control	0	22.02	1.01
DW5-03	PCEA-WG	5.88	638	Control	0	21.41	0.95
DW2-02	PCEA-WG	5.96	634	Control	0	20.45	0.86
DW8-03	PCEA-WG	6.24	670	Control	0	20.80	0.89
DW7-01	PCEA-WG	6.27	679	Control	0	20.82	0.90
DW2-01	PCEA-WG	6.49	672	Control	0	20.03	0.82
DW4-03	PCEA-WG	6.68	674	Control	0	20.52	0.87
DW1-03	PCEA-WG	4.36	606	Creep	-0.95	18.58	0.69
DW8-02	PCEA-WG	4.25	611	Creep	-1.25	17.75	0.62
DW3-03	PCEA-WG	4.38	609	Creep	-1.32	18.04	0.64
DW10-01	PCEA-WG	4.39	610	Creep	-1.59	17.42	0.59
DW6-02	PCEA-WG	5.97	679	Creep	-1.61	18.13	0.65
DW1-01	PCEA-WG	6.73	706	Creep	-1.63	18.89	0.72
DW6-03	PCEA-WG	6.52	714	Creep	-1.66	17.68	0.61
DW3-02	PCEA-WG	6.02	670	Creep	-1.75	18.56	0.69
DW8-01	PCEA-WG	5.87	674	Creep	-1.85	17.59	0.60
DW7-03	PCEA-WG	6.55	706	Creep	-2.04	17.85	0.63
DW3-01	PCEA-WG	6.80	711	Creep	-2.04	18.32	0.67
DW1-02	PCEA-WG	6.39	683	Creep	-2.40	17.89	0.63
DW9-03	PCEA-WG	6.83	710	Creep	-2.42	18.13	0.65
DW5-02	PCEA-WG	5.66	669	Creep	-2.42	17.06	0.55
DW11-01	PCEA-WG	6.32	687	Creep	-2.54	17.12	0.56

Table 19. Grade E, IG-110 graphite mean dynamic elastic modulus (fundamental frequency method) for creep (stressed) and control samples.

Sample ID	GRADE	DOSE dpa	Irr. Temp °C	SPEC TYPE	CREEP STRAIN %	E _{dyn} (GPa)	Fractional change in Modulus [(Ei/E0)-1]
EW9-02	IG-110	3.31	507	Control	0	18.43	1.08
EW2-03	IG-110	4.36	562	Control	0	17.67	0.99
EW10-01	IG-110	5.11	582	Control	0	18.43	1.08
EW5-03	IG-110	5.76	613	Control	0	18.66	1.10
EW8-01	IG-110	6.13	671	Control	0	18.45	1.08
EW5-02	IG-110	6.18	653	Control	0	18.35	1.07
EW10-03	IG-110	6.43	678	Control	0	19.47	1.19
EW9-01	IG-110	6.62	681	Control	0	18.71	1.11
EW2-02	IG-110	4.75	621	Creep	-1.21	16.94	0.91
EW8-03	IG-110	3.82	601	Creep	-1.31	16.72	0.88
EW2-01	IG-110	5.11	635	Creep	-1.32	16.99	0.91
EW7-01	IG-110	6.42	708	Creep	-1.82	17.17	0.93
EW4-01	IG-110	4.69	628	Creep	-1.92	16.50	0.86
EW6-03	IG-110	6.63	713	Creep	-1.94	17.50	0.97
EW5-01	IG-110	6.24	674	Creep	-2.12	16.96	0.91
EW6-01	IG-110	5.05	642	Creep	-2.17	16.39	0.85
EW9-03	IG-110	5.75	666	Creep	-2.31	17.32	0.95
EW8-02	IG-110	6.77	712	Creep	-2.53	17.25	0.94
EW10-02	IG-110	6.69	713	Creep	-2.76	16.91	0.90
EW4-02	IG-110	6.54	693	Creep	-2.78	16.59	0.87

Table 20. Grade F, IG-430 graphite mean dynamic elastic modulus (fundamental frequency method) for creep (stressed) and control samples.

Table	20. Grade F, 1G-450 g	graphite mean dynami	c elastic modulus (lui	iuamentai irequen	cy method) for the	eep (stresseu) and	control samples.
Sample ID	GRADE	DOSE dpa	Irr. Temp °C	SPEC TYPE	CREEP STRAIN %	E _{dyn} (GPa)	Fractional change in Modulus [(Ei/E0)-1]
EW4 04	IC 420	0.70	470	Control	0	40.04	0.00
FW4-01	IG-430	2.78	472	Control	0	18.61	0.96
FW7-03	IG-430	3.32	508	Control	0	18.26	0.93
FW7-02	IG-430	3.80	537	Control	0	19.10	1.02
FW3-03	IG-430	4.37	564	Control	0	19.47	1.05
FW9-01	IG-430	4.91	587	Control	0	21.71	1.29
FW2-01	IG-430	5.12	580	Control	0	19.65	1.07
FW5-03	IG-430 IG-430	5.74	639	Control	0	21.55	1.27 1.16
FW10-02 FW3-02	IG-430	6.06 6.22	656 653	Control Control	0	20.51 19.09	1.01
FW7-01	IG-430	6.28	670	Control	0	19.54	1.06
FW10-01	IG-430	6.52	593	Control	0	19.73	1.08
FW1-03	IG-430	6.61	669	Control	0	18.63	0.96
FW1-03	IG-430	6.73	662	Control	0	18.39	0.96
1 00 11-03	10-430	0.73	002	Control	0	10.59	0.94
FW12-01	IG-430	3.37	593	Creep	-0.92	17.98	0.90
FW8-02	IG-430	5.51	669	Creep	-0.95	17.29	0.82
FW13-01	IG-430	5.72	662	Creep	-1.09	20.91	1.21
FW4-02	IG-430	6.15	688	Creep	-1.18	17.08	0.80
FW5-02	IG-430	3.87	602	Creep	-1.21	18.89	0.99
FW2-03	IG-430	5.15	639	Creep	-1.28	18.96	1.00
FW3-01	IG-430	4.78	625	Creep	-1.34	19.73	1.08
FW5-01	IG-430	4.30	612	Creep	-1.35	18.75	0.98
FW8-01	IG-430	6.59	716	Creep	-1.45	17.51	0.85
FW11-01	IG-430	5.14	639	Creep	-1.54	18.44	0.95
FW11-02	IG-430	4.78	626	Creep	-1.54	18.77	0.98
FW9-03	IG-430	6.43	697	Creep	-1.55	17.01	0.79
FW1-01	IG-430	6.79	708	Creep	-1.70	17.86	0.88
FW9-02	IG-430	6.72	714	Creep	-1.75	17.13	0.81
FW2-02	IG-430	6.58	693	Creep	-1.83	17.74	0.87
FW4-03	IG-430	6.59	707	Creep	-2.04	17.17	0.81
FW10-03	IG-430	6.87	708	Creep	-2.56	17.83	0.88

8.2 Dynamic Elastic Modulus (by ultrasonic velocity/time of flight)

Table 21. Grade A, NBG-17 graphite mean Dynamic elastic modulus (ultrasonic velocity/tof) for creep (stressed) and control samples.

	7 Brapinte mean	Dynamic clastic	inodulus (dicius	offic veloci	cy/ tor/ for the	cp (stresset	a) and control sa
			Irr. Temp	SPEC	CREEP	F.	Fractional change in
Sample ID	GRADE	DOSE dpa	•		STRAIN	E_{dyn}	
	0	2 002 0.00	°C	TYPE		(GPa)	Modulus
					%		[(Ei/E0)-1]
							[([],[], []
A18.03	NDC 17.4C	4.65	FOF	Control	0.00	22.04	1.10
AL8-02	NBG-17 AG	4.65	585	Control	0.00	22.04	1.16
AL6-01	NBG-17 AG	6.40	667	Control	0.00	22.28	1.18
AL8-01	NBG-17 AG	5.33	655	Creep	-1.42	20.46	1.00
AL6-02	NBG-17 AG	6.70	703	Creep	-1.61	21.47	1.10
11115 04		2 =2	470		2.22	20.04	
AW6-01	NBG-17 WG	2.79	472	Control	0.00	20.91	1.10
AW13-01	NBG-17 WG	2.85	470	Control	0.00	20.87	1.10
AW5-01	NBG-17 WG	2.86	470	Control	0.00	19.61	0.97
AW2-03	NBG-17 WG	2.87	468	Control	0.00	20.99	1.11
AW7-01	NBG-17 WG	4.55	587	Control	0.00	19.34	0.95
AW4-03	NBG-17 WG	4.78	582	Control	0.00	21.78	1.19
AW5-03	NBG-17 WG	5.35	597	Control	0.00	19.92	1.00
AW12-03	NBG-17 WG	5.43	594	Control	0.00	22.28	1.24
AW2-02	NBG-17 WG	5.44	592	Control	0.00	21.93	1.21
AW10-01	NBG-17 WG	5.61	617	Control	0.00	20.76	1.09
AW2-01	NBG-17 WG	6.17	650	Control	0.00	21.53	1.17
AW9-03	NBG-17 WG	6.40	678	Control	0.00	22.43	1.26
AW6-03	NBG-17 WG	3.32	594	Creep	-0.75	17.35	0.75
AW1-03	NBG-17 WG	3.47	589	Creep	-0.72	20.22	1.03
AW4-02	NBG-17 WG	3.49	592	Creep	-0.90	20.30	1.04
AW6-02	NBG-17 WG	5.23	656	Creep	-1.05	18.36	0.85
AW12-01	NBG-17 WG	3.51	593	Creep	-1.09	19.92	1.00
AW5-02	NBG-17 WG	3.42	594	Creep	-1.10	17.17	0.73
AW1-02	NBG-17 WG	5.97	668	Creep	-1.13	21.46	1.16
AW4-01	NBG-17 WG	5.47	653	Creep	-1.28	21.09	1.12
AW1-01	NBG-17 WG	6.51	690	Creep	-1.42	20.64	1.08
AW9-01	NBG-17 WG	6.09	677	Creep	-1.47	20.94	1.11
AW7-03	NBG-17 WG	6.65	712	Creep	-1.62	19.59	0.97
AW13-02	NBG-17 WG	5.92	674	Creep	-1.85	19.75	0.99
AW10-03	NBG-17 WG	6.00	670	Creep	-1.89	19.26	0.94
AW10-02	NBG-17 WG	6.66	703	Creep	-2.01	17.48	0.76

Table 22. Grade B, NBG-18 graphite mean dynamic elastic modulus (ultrasonic velocity/tof) for creep (stressed) and control samples.

Sample ID	GRADE	DOSE dpa	Irr. Temp °C	SPEC TYPE	CREEP STRAIN %	E _{dyn} (GPa)	Fractional change in Modulus [(Ei/E0)-1]
BL7-02	NBG-18 AG	3.39	505	Control	0	21.70	1.01
BL6-02	NBG-18 AG	6.56	678	Control	0	22.36	1.07
BL7-01	NBG-18 AG	3.96	559	Creep	-1.06	20.74	0.92
BL6-03	NBG-18 AG	6.76	714	Creep	-2.07	17.73	0.64
BW1-02	NBG-18 WG	3.41	504	Control	0	19.34	0.81
BW11-02	NBG-18 WG	3.41	505	Control	0	22.03	1.07
BW7-01	NBG-18 WG	4.66	585	Control	0	22.62	1.12
BW10-02	NBG-18 WG	5.01	585	Control	0	22.86	1.14
BW3-01	NBG-18 WG	5.15	582	Control	0	19.79	0.86
BW8-02	NBG-18 WG	5.22	599	Control	0	23.48	1.20
BW5-03	NBG-18 WG	5.63	617	Control	0	23.29	1.18
BW5-02	NBG-18 WG	5.72	611	Control	0	22.18	1.08
BW12-01	NBG-18 WG	5.72	613	Control	0	22.46	1.11
BW10-01	NBG-18 WG	5.85	638	Control	0	22.84	1.14
BW8-01	NBG-18 WG	5.95	658	Control	0	23.12	1.17
BW1-03	NBG-18 WG	6.34	664	Control	0	19.34	0.81
BW2-03	NBG-18 WG	6.55	675	Control	0	22.37	1.10
BW7-03	NBG-18 WG	3.76	603	Creep	-0.62	20.62	0.93
BW12-02	NBG-18 WG	3.93	597	Creep	-0.72	21.09	0.98
BW9-03	NBG-18 WG	3.95	599	Creep	-0.90	20.87	0.96
BW3-02	NBG-18 WG	6.19	671	Creep	-1.19	19.21	0.80
BW7-02	NBG-18 WG	6.30	698	Creep	-1.20	19.88	0.86
BW1-01	NBG-18 WG	6.63	700	Creep	-1.35	18.77	0.76
BW9-02	NBG-18 WG	5.62	668	Creep	-1.37	20.00	0.88
BW2-02	NBG-18 WG	5.76	665	Creep	-1.38	19.99	0.88
BW12-03	NBG-18 WG	5.76	675	Creep	-1.48	17.51	0.64
BW9-01	NBG-18 WG	6.28	686	Creep	-1.49	18.60	0.74
BW5-01	NBG-18 WG	5.38	656	Creep	-1.64	18.56	0.74
BW2-01	NBG-18 WG	6.80	709	Creep	-1.72	18.38	0.72
BW11-01	NBG-18 WG	6.21	674	Creep	-1.80	21.01	0.97
BW3-03	NBG-18 WG	6.13	678	Creep	-1.82	18.84	0.77
BW10-03	NBG-18 WG	6.76	709	Creep	-2.03	21.10	0.98

Table 23. Grade C, H-451 graphite mean dynamic elastic modulus (ultrasonic velocity/tof) for creep (stressed) and control samples.

Sample ID	GRADE		Irr. Temp °C	SPEC TYPE	CREEP STRAIN %	E _{dyn} (GPa)	Fractional change in Modulus [(Ei/E0)-1]
CW13-01	H-451 (WG)	4.24	567	Control	0	19.11	1.40
CW14-02	H-451 (WG)	4.74	582	Control	0	16.28	1.04
CW8-03	H-451 (WG)	4.76	580	Control	0	15.34	0.92
CW10-03	H-451 (WG)	6.09	657	Control	0	19.23	1.41
CW10-02	H-451 (WG)	6.66	681	Control	0	18.25	1.29
CW8-02	H-451 (WG)	6.7	674	Control	0	16.11	1.02
CW11-02	H-451 (WG)	4.60	628	Creep	-1.19	13.78	0.73
CW11-01	H-451 (WG)	4.91	642	Creep	-1.21	17.06	1.14
CW7-03	H-451 (WG)	5.41	649	Creep	-1.35	15.28	0.92
CW14-01	H-451 (WG)	4.64	627	Creep	-1.42	14.65	0.84
CW12-02	H-451 (WG)	5.00	641	Creep	-1.79	13.94	0.75
CW7-01	H-451 (WG)	6.84	706	Creep	-1.89	14.31	0.79
CW13-02	H-451 (WG)	6.92	708	Creep	-2.10	14.39	0.81
CW13-03	H-451 (WG)	5.46	653	Creep	-2.25	17.51	1.20
CW10-01	H-451 (WG)	6.47	697	Creep	-2.61	13.89	0.74
CW9-03	H-451 (WG)	6.81	712	Creep	-3.09	13.19	0.66

Table 24. Grade D, PCEA graphite mean dynamic elastic modulus (ultrasonic velocity/tof) for creep (stressed) and control samples.

Sample ID		DOSE dpa	Irr. Temp °C	SPEC TYPE	CREEP STRAIN %	E _{dyn} (GPa)	Fractional change in Modulus [(Ei/E0)-1]
DA702	PCEA-AG	3.72	538	Control	0	15.15	0.76
DA601	PCEA-AG	5.96	632	Control	0	16.45	0.91
DA701	PCEA-AG	4.17	612	Creep	-1.168	15.15	0.76
DA602	PCEA-AG	6.37	680	Creep	-1.878	14.88	0.73
DW10-02	PCEA-WG	3.88	534	Control	0	20.24	1.15
DW2-03	PCEA-WG	3.91	533	Control	0	20.18	1.14
DW5-01	PCEA-WG	3.91	534	Control	0	19.66	1.09
DW6-01	PCEA-WG	5.02	585	Control	0	21.30	1.26
DW9-01	PCEA-WG	5.33	597	Control	0	19.71	1.09
DW4-01	PCEA-WG	5.47	594	Control	0	20.98	1.23
DW7-02	PCEA-WG	5.50	618	Control	0	18.91	1.01
DW5-03	PCEA-WG	5.88	638	Control	0	21.27	1.26
DW2-02	PCEA-WG	5.96	634	Control	0	20.31	1.16
DW8-03	PCEA-WG	6.24	670	Control	0	19.61	1.08
DW7-01	PCEA-WG	6.27	679	Control	0	18.28	0.94
DW2-01	PCEA-WG	6.49	672	Control	0	20.13	1.14
DW4-03	PCEA-WG	6.68	674	Control	0	20.56	1.19
DW1-03	PCEA-WG	4.36	606	Creep	-0.95	18.90	1.01
DW8-02	PCEA-WG	4.25	611	Creep	-1.25	15.83	0.68
DW3-03	PCEA-WG	4.38	609	Creep	-1.32	17.54	0.86
DW10-01	PCEA-WG	4.39	610	Creep	-1.59	18.00	0.91
DW6-02	PCEA-WG	5.97	679	Creep	-1.61	15.61	0.66
DW1-01	PCEA-WG	6.73	706	Creep	-1.63	19.31	1.05
DW6-03	PCEA-WG	6.52	714	Creep	-1.66	15.55	0.65
DW3-02	PCEA-WG	6.02	670	Creep	-1.75	16.58	0.76
DW8-01	PCEA-WG	5.87	674	Creep	-1.85	15.66	0.66
DW7-03	PCEA-WG	6.55	706	Creep	-2.04	15.92	0.69
DW3-01	PCEA-WG	6.80	711	Creep	-2.04	17.61	0.87
DW1-02	PCEA-WG	6.39	683	Creep	-2.40	18.08	0.92
DW9-03	PCEA-WG	6.83	710	Creep	-2.42	16.39	0.74
DW5-02	PCEA-WG	5.66	669	Creep	-2.42	15.44	0.64
DW11-01	PCEA-WG	6.32	687	Creep	-2.54	17.72	0.88

Table 25. Grade E, IG-110 graphite mean dynamic elastic modulus (ultrasonic velocity/tof) for creep (stressed) and control samples.

Sample ID		DOSE dpa	Irr. Temp °C	SPEC TYPE	CREEP STRAIN %	E _{dyn} (GPa)	Fractional change in Modulus [(Ei/E0)-1]
EW9-02	IG-110	3.31	507	Control	0	18.34	1.28
EW2-03	IG-110	4.36	562	Control	0	17.87	1.22
EW10-01	IG-110	5.11	582	Control	0	18.61	1.31
EW5-03	IG-110	5.76	613	Control	0	18.78	1.34
EW8-01	IG-110	6.13	671	Control	0	18.82	1.34
EW5-02	IG-110	6.18	653	Control	0	18.52	1.30
EW10-03	IG-110	6.43	678	Control	0	19.10	1.38
EW9-01	IG-110	6.62	681	Control	0	18.46	1.30
EW2-02	IG-110	4.75	621	Creep	-1.21	17.59	1.19
EW8-03	IG-110	3.82	601	Creep	-1.31	16.82	1.09
EW2-01	IG-110	5.11	635	Creep	-1.32	17.37	1.16
EW7-01	IG-110	6.42	708	Creep	-1.82	17.21	1.14
EW4-01	IG-110	4.69	628	Creep	-1.92	16.62	1.07
EW6-03	IG-110	6.63	713	Creep	-1.94	17.95	1.23
EW5-01	IG-110	6.24	674	Creep	-2.12	17.34	1.16
EW6-01	IG-110	5.05	642	Creep	-2.17	16.74	1.08
EW9-03	IG-110	5.75	666	Creep	-2.31	16.93	1.11
EW8-02	IG-110	6.77	712	Creep	-2.53	17.48	1.17
EW10-02	IG-110	6.69	713	Creep	-2.76	17.13	1.13
EW4-02	IG-110	6.54	693	Creep	-2.78	16.95	1.11

Table 26. Grade F, IG-430 graphite mean dynamic elastic modulus (ultrasonic velocity/tof) for creep (stressed) and control samples.

Sample ID	GRADE	DOSE dpa	Irr. Temp °C	SPEC TYPE	CREEP STRAIN %	E _{dyn} (GPa)	Fractional change in Modulus [(Ei/E0)-1]
FW4-01	IG-430	2.78	472	Control	0	18.54	0.92
FW7-03	IG-430	3.32	508	Control	0	18.63	0.93
FW7-02	IG-430	3.80	537	Control	0	19.06	0.97
FW3-03	IG-430	4.37	564	Control	0	19.61	1.03
FW9-01	IG-430	4.91	587	Control	0	21.84	1.26
FW2-01	IG-430	5.12	580	Control	0	19.55	1.03
FW5-03	IG-430	5.74	639	Control	0	21.85	1.26
FW10-02	IG-430	6.06	656	Control	0	20.35	1.11
FW3-02	IG-430	6.22	653	Control	0	19.16	0.99
FW7-01	IG-430	6.28	670	Control	0	20.08	1.08
FW10-01	IG-430	6.52	678	Control	0	19.44	1.01
FW1-03	IG-430	6.61	672	Control	0	18.70	0.94
FW11-03	IG-430	6.73	677	Control	0	18.55	0.92
FW12-01	IG-430	3.37	593	Creep	-0.92	18.14	0.88
FW8-02	IG-430	5.51	669	Creep	-0.95	17.51	0.81
FW13-01	IG-430	5.72	662	Creep	-1.09	20.93	1.17
FW4-02	IG-430	6.15	688	Creep	-1.18	17.26	0.79
FW5-02	IG-430	3.87	602	Creep	-1.21	18.83	0.95
FW2-03	IG-430	5.15	639	Creep	-1.28	19.36	1.01
FW3-01	IG-430	4.38	625	Creep	-1.34	19.52	1.02
FW5-01	IG-430	4.30	612	Creep	-1.35	18.55	0.92
FW8-01	IG-430	6.59	716	Creep	-1.45	17.69	0.83
FW11-01	IG-430	5.14	639	Creep	-1.54	18.96	0.96
FW11-02	IG-430	4.78	626	Creep	-1.54	18.91	0.96
FW9-03	IG-430	6.43	697	Creep	-1.55	17.18	0.78
FW1-01	IG-430	6.79	708	Creep	-1.70	18.11	0.88
FW9-02	IG-430	6.72	714	Creep	-1.75	17.46	0.81
FW2-02	IG-430	6.58	693	Creep	-1.83	18.10	0.88
FW4-03	IG-430	6.59	707	Creep	-2.04	17.13	0.78
FW10-03	IG-430	6.87	708	Creep	-2.56	17.95	0.86

9 Distribution

ORNL INL Department of Energy

Tim Burchell Judy Mount William Corwin

Anne Campbell Larry Hull Tom O'Connor

Cristian Contescu Travis Mitchell

Yutai Katoh David Swank

Mark Vance David Petti

Wei Ju Ren William Windes

10 References

¹ ORNL/TM-2015/377, T. Burchell, "AGC-1 Irradiation Induced Property Changes Analysis Report: Electrical Resistivity and Coefficient of Thermal Expansion," February 2016.

² ORNL/TM-2005/505, T. Burchell and R. Bratton, "Graphite irradiation creep capsule AGC-1 experimental plan," September 2006.

³ ORNL/TM-2009/009, T. Burchell "A revised AGC-1 creep capsule layout," November 2010.

⁴ ORNL/TM-2010/285, T. Burchell, J. Strizak, and M. Williams, "AGC-1 Specimen Pre-irradiation Data Report," October 2010.

⁵ ORNL/TM-2013/242, T. Burchell "AGC-1 Specimen Post Irradiation Data Report," September 2013.

⁶ ORNL/TM-2014/255, T. Burchell, "AGC-1 Creep Strain Data Analysis," September 2014.

⁷ ORNL/TM-2007/153, T. Burchell, R. Bratton, and W. Windes, "NGNP Graphite Selection and Acquisition Strategy," September 2007.

⁸ PLN-2497 Rev. 1, W. Windes, T. Burchell, and R. Bratton, "Graphite Technology Development Plan," October 2010.

⁹ ASTM Testing Standard C747-93 (Reapproved 2010)

¹⁰ ASTM Testing Standard C1259-08

¹¹ Student (William S. Gossett), "The Probable Error of a Mean," BIOMETRIKA Vol.6, No. 1, pp. 1-25 (1908).

¹² GraphPad Software, QuickCalcs, t test. http://www.graphpad.com/quickcalcs/ttest1/?Format=SD.

¹³ R. E. Nightingale., "Nuclear Graphite," Academic Press, New York (1962).

¹⁴ J. H. W. Simmons, "Radiation Damage in Graphite," Pergamon Press, Oxford (1965).

¹⁵ B. T. Kelly., "Physics of Graphite," *Applied Science Publishers*, London (1981).

¹⁶ T. Burchell, "Radiation Effects in Graphite and Carbon-Based Materials," *MRS Bulletin*, Vol. XXII, No. 2 pp. 29-35, February 1997.

¹⁷ Burchell, T., "Radiation Effects in Graphite," Comprehensive Nuclear Materials, Vol. 4, pp. 299-324 (2012).