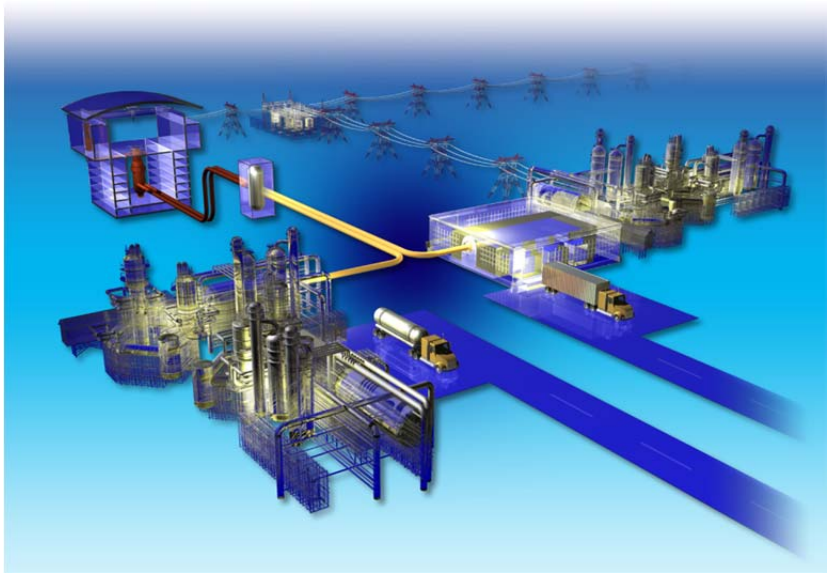
AGC-1 PIE Experimental Plan

Tim Burchell

Oak Ridge National Laboratory

March 2012



Prepared for

Office of Nuclear Energy Science and Technology

Prepared by

OAK RIDGE NATIONAL LABORATORY

Oak Ridge, Tennessee 37831

managed by

UT-BATTELLE, LLC

for the

U.S. DEPARTMENT OF ENERGY

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SUMMARY

Here we report the Post-Irradiation Examination planned for the AGC-1 creep samples. The key data obtained for each grade of graphite included in the AGC-1capsule are:

- Dimensions, volume, mass and density changes
- Creep strain
- Electrical resistivity
- Elastic constants (E, G, υ) from sonic velocity
- Dynamic modulus (from fundamental frequency of vibration)
- Coefficient of thermal expansion
- Microstructural characterization
- Fracture strength

The effect of creep strain on the above properties will be determined.

These data are critical to the design of the Next Generation Nuclear Plant (NGNP). Moreover, the data supports ongoing work in the area of model development, e.g., irradiation effects models such as dimensional change and creep. The data will be used to help underpin the existing American Society of Mechanical Engineers (ASME) design code for graphite core components.

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ABBREVIATIONS

AGC	Advanced Graphite Creep
ASME	American Society of Mechanical Engineers
ASTM	American Society for Testing and Materials
ATR	Advanced Test Reactor
ER	Electrical Resistivity
INL	Idaho National Laboratory
LAMDA	Low Activation Materials Development and Analysis
MFC	Materials and Fuels Complex
ORNL	Oak Ridge National Laboratory
PIE	Post-Irradiation Examination
Pre-IE	Pre-Irradiation Examination

1 INTRODUCTION

The AGC-1 experiment is the first of a series of graphite creep capsules to be irradiated in the Advanced Teat Reactor (ATR) at Idaho National Laboratory (INL) in support of the Next Generation Nuclear Plant (NGNP). Since the AGC-1 capsule was first conceived [1] it has undergone a number of design changes, requiring a revision of the original layout [2]. Here we describe the post irradiation examination (PIE) of the AGC-1 creep specimens to be conducted in the Low Activation Materials Development and Analysis (LAMDA) facility at the Oak Ridge National Laboratory (ORNL). This experimental plan describes the testing to be performed on the graphite creep specimens and SiC monitors only.

2 MATERIALS

A full description of the materials/specimens contained in this experiment may be found in the "AGC-1 Experimental Plan" [1] and in "A Revised AGC-1 Creep Capsule Layout" [2]. The graphite to be examined here are the AGC-1 creep specimens listed in Table 1. The pre-irradiation data [3] and sister specimens testing data [4] have previously been reported.

Table 1 Graphite grades and marking codes used for the AGC-1 series of creep specimens

Graphite Code Letter	Graphite Grade
A	NBG-17
B	NBG-18
C	H-451
D	PCEA
E	IG-110
F	IG-430

The graphite grades examined here all are in compliance with the ASTM specification for nuclear graphite [5].

3 MATERIAL POST-IRRADIATION EXAMINATION

Post-irradiation examination (PIE) will follow the workflow chart in Figure 1. Following irradiation in ATR the AGC-1 capsule will cool in the ponds followed by shipment to the hot cells. The capsule is disassembled in the hot cells at INL and the specimens and temperature monitors recovered and shipped to the Low Activation Material Development & Analysis (LAMDA) at ORNL. The specimens will undergo post irradiation examination (PIE) which will consist of visual inspection, dimensional measurements, creep strain, mass, and hence density, elastic constants (sonic velocity), dynamic Modulus by resonance frequency, electrical resistivity, and coefficient of thermal expansion. Additional microstructural characterization may be performed depending on schedule and budget. Fracture testing will also be conducted but the nature of testing (tensile, or compression) has not been determined at this time.

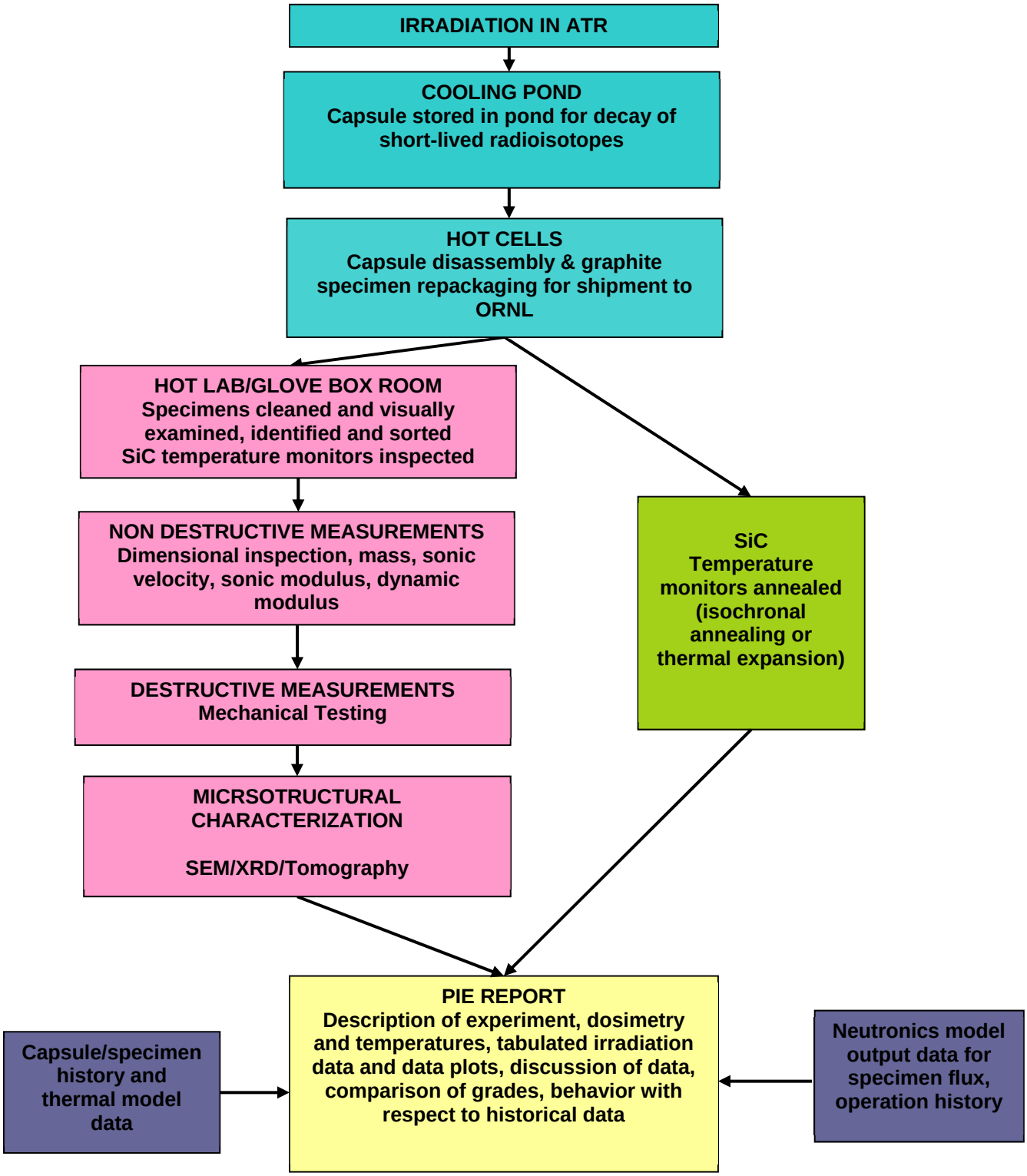


Figure 1 Post-irradiation data process flow chart for AGC-1 graphites

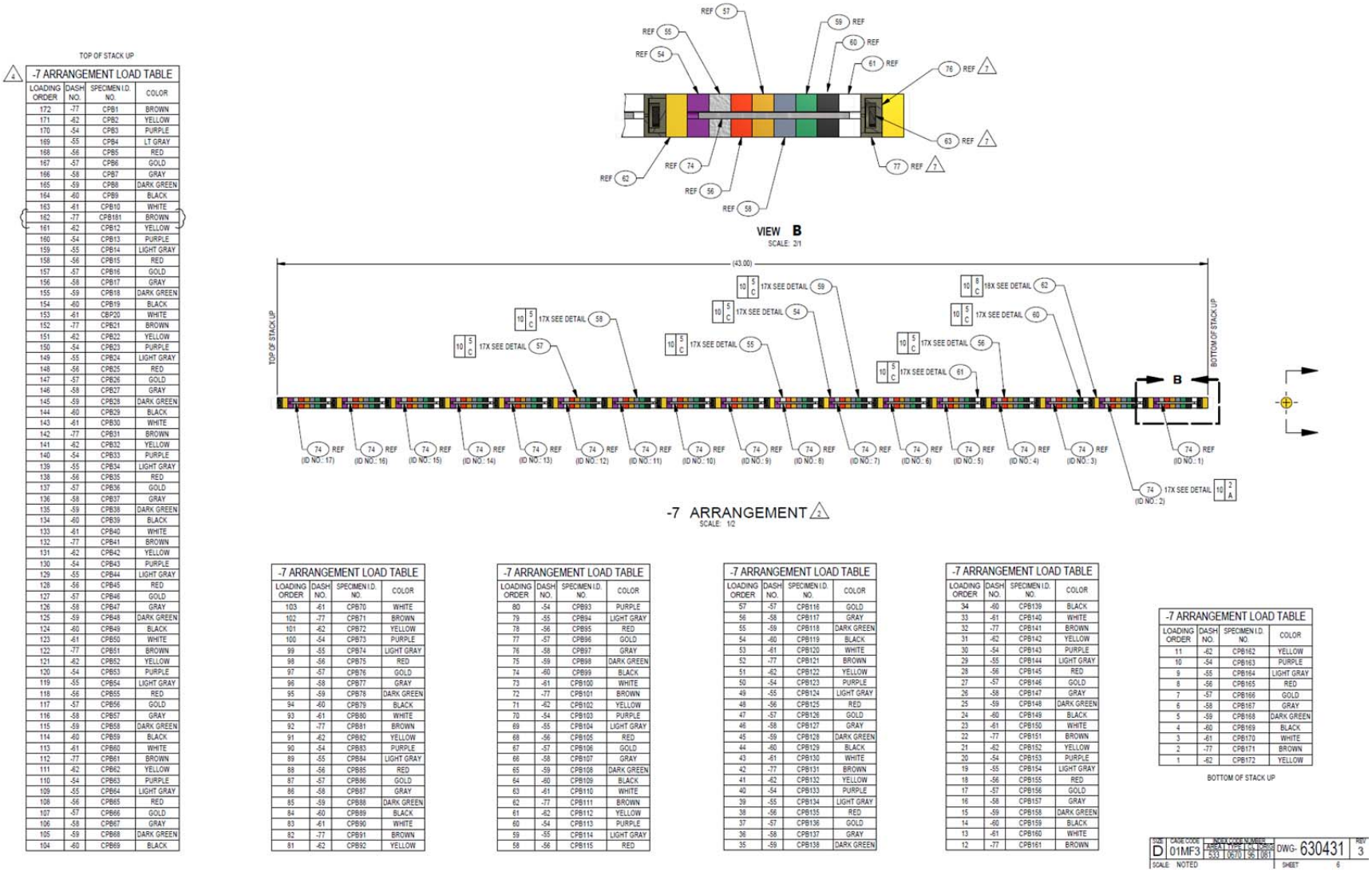
The specimen irradiation temperature will be determined from temperature monitors encapsulated within the specimens (Figure 2) in the AGC-1 central channel. The SiC monitors will be retrieved by INL and shipped to ORNL for testing. The irradiation temperature will be estimated by either (i) isochronal annealing followed by electrical resistivity (ER) determination using ASTM C611 [6], or (ii) thermal expansion. A plot of resistivity as a function of annealing temperature should display a marked change in resistivity when the annealing temperature exceeds the maximum irradiation temperature. The second method involves the continuous measurement of thermal expansion, in accordance with ASTM E228 [7] as the monitor is heated to a temperature in excess of the irradiation temperature. Again, the expansion data should exhibit a change in gradient when the irradiation temperature is exceeded.

The isochronal annealing temperature schedule is reported in Table 2.

Table 2 Annealing schedule for SiC temperature monitors

Annealing Temperature, °C	Heating Rate, °C/min	Time at Temperature, minutes	Actions
400	2.5	30	Cool to RT, Measure ER
450	2.5	30	Cool to RT, Measure ER
500	2.5	30	Cool to RT, Measure ER
550	2.5	30	Cool to RT, Measure ER
600	2.5	30	Cool to RT, Measure ER
625	2.5	30	Cool to RT, Measure ER
650	2.5	30	Cool to RT, Measure ER
675	2.5	30	Cool to RT, Measure ER
700	2.5	30	Cool to RT, Measure ER
725	2.5	30	Cool to RT, Measure ER
750	2.5	30	Cool to RT, Measure ER
775	2.5	30	Cool to RT, Measure ER
800	2.5	30	Cool to RT, Measure ER
850	2.5	30	Cool to RT, Measure ER
900	2.5	30	Cool to RT, Measure ER

Isochronal annealing furnace runs will be heated at 2.5 °C/minute, followed by a 30 minute dwell at temperature, cooled to room temperature and ER measured [6]. The experimental thermometry data will be contained in a final PIE report. Some of the SiC monitors were found broken on receipt from INL and thus were too short for ER measurement. These shorter monitors will be subjected to thermal expansion measurement. The SiC temperature monitors were heated at a rate of 2.5°C/minute to 1000°C while expansion was continuously measured according toE228 [7]. Table 3 indicates the SiC numbers, tube ID's, and the experimental measurements to be made.



-7 ARRANGEMENT LOAD TABLE

LOADING ORDER	DASH NO.	SPECIMEN I.D. NO.	COLOR
172	-77	CPB1	BROWN
171	-62	CPB2	YELLOW
170	-54	CPB3	PURPLE
169	-55	CPB4	LT GRAY
168	-56	CPB5	RED
167	-57	CPB6	GOLD
166	-58	CPB7	GRAY
165	-59	CPB8	DARK GREEN
164	-60	CPB9	BLACK
163	-61	CPB10	WHITE
162	-77	CPB181	BROWN
161	-62	CPB12	YELLOW
160	-54	CPB13	PURPLE
159	-55	CPB14	LIGHT GRAY
158	-56	CPB15	RED
157	-57	CPB16	GOLD
156	-58	CPB17	GRAY
155	-59	CPB18	DARK GREEN
154	-60	CPB19	BLACK
153	-61	CBP20	WHITE
152	-77	CPB21	BROWN
151	-62	CPB22	YELLOW
150	-54	CPB23	PURPLE
149	-55	CPB24	LIGHT GRAY
148	-56	CPB25	RED
147	-57	CPB26	GOLD
146	-58	CPB27	GRAY
145	-59	CPB28	DARK GREEN
144	-60	CPB29	BLACK
143	-61	CPB30	WHITE
142	-77	CPB31	BROWN
141	-62	CPB32	YELLOW
140	-54	CPB33	PURPLE
139	-55	CPB34	LIGHT GRAY
138	-56	CPB35	RED
137	-57	CPB36	GOLD
136	-58	CPB37	GRAY
135	-59	CPB38	DARK GREEN
134	-60	CPB39	BLACK
133	-61	CPB40	WHITE
132	-77	CPB41	BROWN
131	-62	CPB42	YELLOW
130	-54	CPB43	PURPLE
129	-55	CPB44	LIGHT GRAY
128	-56	CPB45	RED
127	-57	CPB46	GOLD
126	-58	CPB47	GRAY
125	-59	CPB48	DARK GREEN
124	-60	CPB49	BLACK
123	-61	CPB50	WHITE
122	-77	CPB51	BROWN
121	-62	CPB52	YELLOW
120	-54	CPB53	PURPLE
119	-55	CPB54	LIGHT GRAY
118	-56	CPB55	RED
117	-57	CPB56	GOLD
116	-58	CPB57	GRAY
115	-59	CPB58	DARK GREEN
114	-60	CPB59	BLACK
113	-61	CPB60	WHITE
112	-77	CPB61	BROWN
111	-62	CPB62	YELLOW
110	-54	CPB63	PURPLE
109	-55	CPB64	LIGHT GRAY
108	-56	CPB65	RED
107	-57	CPB66	GOLD
106	-58	CPB67	GRAY
105	-59	CPB68	DARK GREEN
104	-60	CPB69	BLACK

-7 ARRANGEMENT LOAD TABLE

LOADING ORDER	DASH NO.	SPECIMEN I.D. NO.	COLOR
103	-61	CPB70	WHITE
102	-77	CPB71	BROWN
101	-62	CPB72	YELLOW
100	-54	CPB73	PURPLE
99	-55	CPB74	LIGHT GRAY
98	-56	CPB75	RED
97	-57	CPB76	GOLD
96	-58	CPB77	GRAY
95	-59	CPB78	DARK GREEN
94	-60	CPB79	BLACK
93	-61	CPB80	WHITE
92	-77	CPB81	BROWN
91	-62	CPB82	YELLOW
90	-54	CPB83	PURPLE
89	-55	CPB84	LIGHT GRAY
88	-56	CPB85	RED
87	-57	CPB86	GOLD
86	-58	CPB87	GRAY
85	-59	CPB88	DARK GREEN
84	-60	CPB89	BLACK
83	-61	CPB90	WHITE
82	-77	CPB91	BROWN
81	-62	CPB92	YELLOW

-7 ARRANGEMENT LOAD TABLE

LOADING ORDER	DASH NO.	SPECIMEN I.D. NO.	COLOR
80	-54	CPB93	PURPLE
79	-55	CPB94	LIGHT GRAY
78	-56	CPB95	RED
77	-57	CPB96	GOLD
76	-58	CPB97	GRAY
75	-59	CPB98	DARK GREEN
74	-60	CPB99	BLACK
73	-61	CPB100	WHITE
72	-77	CPB101	BROWN
71	-62	CPB102	YELLOW
70	-54	CPB103	PURPLE
69	-55	CPB104	LIGHT GRAY
68	-56	CPB105	RED
67	-57	CPB106	GOLD
66	-58	CPB107	GRAY
65	-59	CPB108	DARK GREEN
64	-60	CPB109	BLACK
63	-61	CPB110	WHITE
62	-77	CPB111	BROWN
61	-62	CPB112	YELLOW
60	-54	CPB113	PURPLE
59	-55	CPB114	LIGHT GRAY
58	-56	CPB115	RED

-7 ARRANGEMENT LOAD TABLE

LOADING ORDER	DASH NO.	SPECIMEN I.D. NO.	COLOR
57	-57	CPB116	GOLD
56	-58	CPB117	GRAY
55	-59	CPB118	DARK GREEN
54	-60	CPB119	BLACK
53	-61	CPB120	WHITE
52	-77	CPB121	BROWN
51	-62	CPB122	YELLOW
50	-54	CPB123	PURPLE
49	-55	CPB124	LIGHT GRAY
48	-56	CPB125	RED
47	-57	CPB126	GOLD
46	-58	CPB127	GRAY
45	-59	CPB128	DARK GREEN
44	-60	CPB129	BLACK
43	-61	CPB130	WHITE
42	-77	CPB131	BROWN
41	-62	CPB132	YELLOW
40	-54	CPB133	PURPLE
39	-55	CPB134	LIGHT GRAY
38	-56	CPB135	RED
37	-57	CPB136	GOLD
36	-58	CPB137	GRAY
35	-59	CPB138	DARK GREEN

-7 ARRANGEMENT LOAD TABLE

LOADING ORDER	DASH NO.	SPECIMEN I.D. NO.	COLOR
34	-60	CPB139	BLACK
33	-61	CPB140	WHITE
32	-77	CPB141	BROWN
31	-62	CPB142	YELLOW
30	-54	CPB143	PURPLE
29	-55	CPB144	LIGHT GRAY
28	-56	CPB145	RED
27	-57	CPB146	GOLD
26	-58	CPB147	GRAY
25	-59	CPB148	DARK GREEN
24	-60	CPB149	BLACK
23	-61	CPB150	WHITE
22	-77	CPB151	BROWN
21	-62	CPB152	YELLOW
20	-54	CPB153	PURPLE
19	-55	CPB154	LIGHT GRAY
18	-56	CPB155	RED
17	-57	CPB156	GOLD
16	-58	CPB157	GRAY
15	-59	CPB158	DARK GREEN
14	-60	CPB159	BLACK
13	-61	CPB160	WHITE
12	-77	CPB161	BROWN

-7 ARRANGEMENT LOAD TABLE

LOADING ORDER	DASH NO.	SPECIMEN I.D. NO.	COLOR
11	-62	CPB162	YELLOW
10	-54	CPB163	PURPLE
9	-55	CPB164	LIGHT GRAY
8	-56	CPB165	RED
7	-57	CPB166	GOLD
6	-58	CPB167	GRAY
5	-59	CPB168	DARK GREEN
4	-60	CPB169	BLACK
3	-61	CPB170	WHITE
2	-77	CPB171	BROWN
1	-62	CPB172	YELLOW

BOTTOM OF STACK UP

SIZE	CAGE CODE	INDEX CODE NUMBER				DWG.	REV
		AREA	TYPE	SQ	PERM		
D	01MF3	533	0670	96	081	630431	3
SCALE	NOTED					SHEET	6

Figure 2 Drawing of AGC-1 capsule showing positions of SiC temperature monitors

Table 3 SiC temperature monitor information from AGC-1 (10-26-11) as supplied by INL

Number on Plastic Cryo Tube	Piggy back specimen associated with SiC TM in Plexiglass shipping tube from MFC.	SiC TM ID Number per INL Dwg. 630431 rev. 3.	Experimental Measurement Technique
1	CPB24	15	Elec. Resist.
2	CPB1-10	17	Elec. Resist.
3	CPB11-20	16	Elec. Resist.
4	CPB88	9	Therm. Expan.
5	CPB124, 125	5	Therm. Expan.
6	CPB76, 77, 79	10	Therm. Expan.
7	CPB156, 157	2	Elec. Resist.
8	CPB118, 119	6	Elec. Resist.
9	CPB167, 168	1	Elec. Resist.
10	CPB145, 144	3	Elec. Resist.
11	CPB110, 109	7	Elec. Resist.
12	CPB98, 99	8	Elec. Resist.
13	CPB68, 70	11	Elec. Resist.
14	CPB55, 57, 60	12	Elec. Resist.
15	CPB49, 47	13	Elec. Resist.
17	Packaged alone in bottle. By process of elimination this is either ID no. 14 or 4 and therefore no. 14 or 4 are missing.	14 or 4	Elec. Resist.

4 EXERIMENTAL PIE METHODS

The applicable ASTM test standards used here are listed in Table 4.

Table 4 Applicable ASTM test methods

Test Property	Standard Title	ASTM Standard	Ref No.
General	Testing Graphite and Boronated Graphite Materials for High-Temperature Gas-Cooled Nuclear Reactor Components	C781	[8]
Dimensions, mass, density	Bulk Density by Physical Measurements of Manufactured Carbon and Graphite Articles	C559	[9]
Electrical Resistivity	Electrical Resistivity of Manufactured Carbon and Graphite Articles at Room Temperature	C611	[6]
Dynamic Young's Modulus	Moduli of Elasticity and Fundamental Frequencies of Carbon and Graphite Materials by Sonic Resonance	C747	[10]
Dynamic Young's Modulus	Dynamic Young's Modulus, Shear Modulus, and Poisson's Ratio for Advance Ceramics by Impulse Excitation of Vibration	C1259	[11]
Yong's Modulus	Sonic Velocity in Manufactured Carbon and Graphite Materials for Use in Obtaining Young's Modulus	C769	[12]
Thermal Expansion	Linear Thermal Expansion of Thermal Materials with a Push-Rod Dilatometer	E228	[7]
Compressive Strength	Compressive Strength of Carbon and Graphite	C695	[13]
Tensile Strength	Tensile Stress-Strain of Carbon and Graphite	C749	[14]
Glued End Tensile Strength	Testing Graphite and Boronated Graphite Materials for High-Temperature Gas-Cooled Nuclear Reactor Components	C781 A4 (mod to C749)	[15]

The order of PIE testing shall be:

1. Visual inspection. photography
2. Dimensions, mass, density
3. Electrical resistivity
4. Dynamic Young's modulus by resonance
5. Sonic velocity and elastic constants
6. Thermal expansion, CTE
7. Strength
8. XRD, SANS, tomography, microscopy

Dimensional measurements will be taken according to the scheme in Figure 3 so as to assure complete repeatability from the pre-irradiation measurements. Similarly, specimen alignment (with respect to the engraved number) during PIE physical property measurements shall be the

same as previously employed during Pre-IE [3]. Any testing limitations in regard of specimen size shall be in accordance with C781[8].

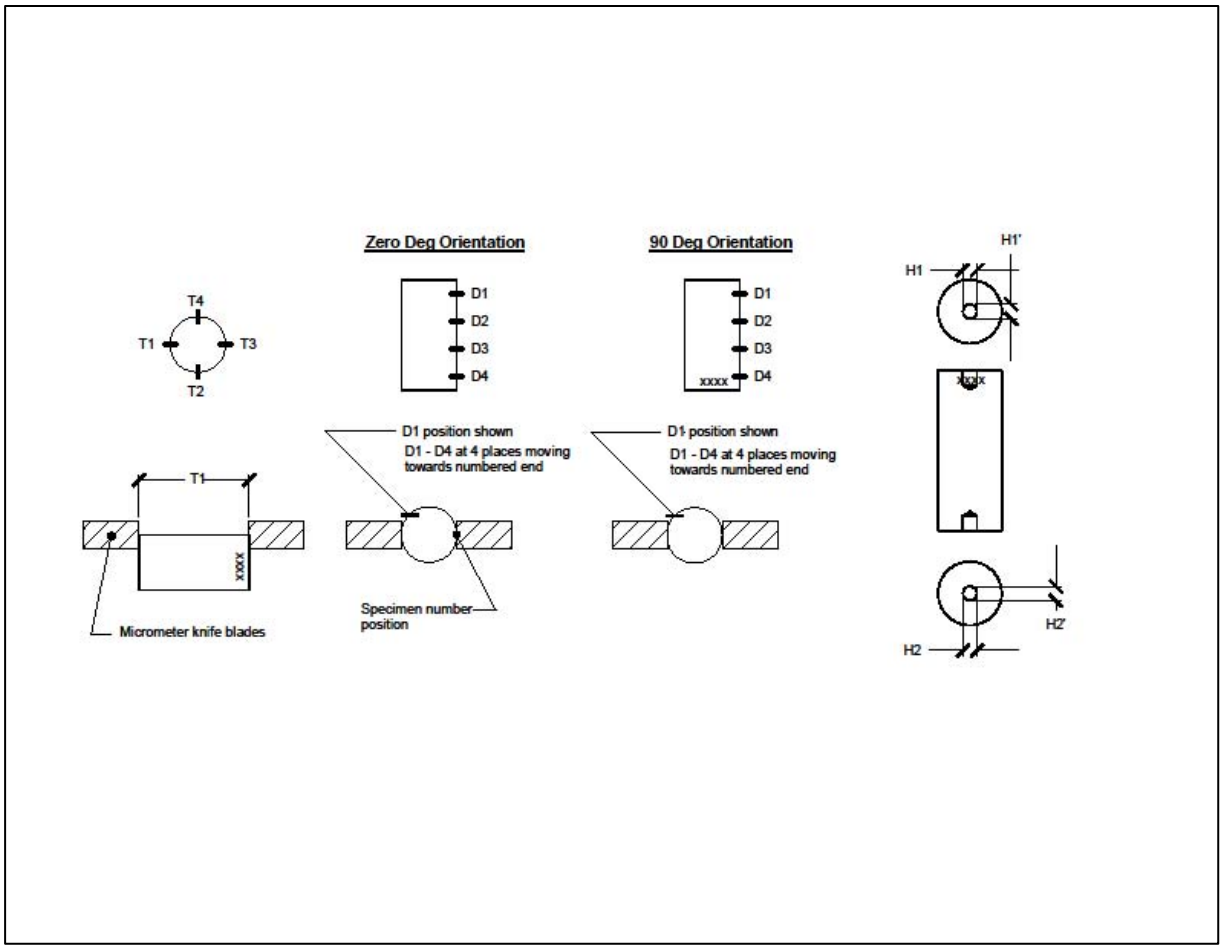


Figure 3 AGC-1 creep specimen measurement scheme

5 QUALITY ASSURANCE

The activities described in this plan will be conducted in accordance with the applicable requirements of the ASME/NQA-1-2000 national standard entitled *Quality Assurance Requirements for Nuclear Facility Applications.* Project and activity-specific information concerning ORNL's application of the standard's requirements is provided in Document #QAP-ORNL-NGNP-01 entitled *Quality Assurance Plan for the Next Generation Nuclear Plant Materials Program at Oak Ridge National Laboratory* [15].

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