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A Experimental Plan for EDF Energy Creep Rabbit Graphite Irradiations – Rev. 2 (replaces Rev. 0 ORNL/TM-2014/49)

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July 2014

OAK RIDGE NATIONAL LABORATORY
MANAGED BY UT-BATTELLE FOR THE US DEPARTMENT OF ENERGY

Experimental Plan for EDF Energy Creep Rabbit Graphite Irradiations - Rev. 2 (replaces Rev. 0 ORNL/TM/2013/49).

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July 2014

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Experimental Plan for EDF Energy Creep Rabbit Graphite Irradiations

SUMMARY

The results obtained through this experimentation will assist in the development and validation of future models of irradiation induced creep of graphite by providing the following data:

- Inert creep stain data from low to lifetime AGR fluence
- Inert creep-property data (especially CTE) from low to lifetime AGR fluence
- Effect of oxidation on creep modulus (by indirect comparison with experiment 1 and direct comparison with experiment 3 – NB. Experiment 1 and 3 are not covered here)
- Data to develop a mechanistic understanding, including
    - Appropriate creep modulus (including pinning and high dose effects on structure)
    - Investigation of CTE-creep strain behavior under inert conditions
    - Information on the effect of applied stress/creep strain on crystallite orientation (requires XRD)
    - Effect of creep strain on micro-porosity (requires tomography & microscopy)

This document describes the experimental work planned to meet the requirements of project technical specification [1] and EDF Energy requests for additional Pre-IE work. The PIE work is described in detail in this revision (Section 8 and 9).

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ABBREVIATIONS

AG	Against-grain
AGR	Advanced Gas-cooled Reactor
ASME	American Society for Mechanical Engineers
ASTM	American Society for Testing and Material
CTE	Coefficient of Thermal Expansion
DNE	DIDO Nickel Equivalent
DPA	Displacements per atom
DYM	Dynamic Modulus
EDF E	EDF Energy Ltd.
HFIR	High Flux Isotope Reactor
NB	Nota Bena (note well)
NBS	National Bureau of Standards
NIST	National Institute of Standards and Technology
ORNL	Oak Ridge National Laboratory
PIE	Post irradiation examination
Pre-IE	Pre-irradiation examination
RR	Round robin
TIG	Tungsten-inert-gas
TOF	Time of Flight
WFO	Work for Others
WG	With-grain
XRD	X-ray diffraction

1 OBJECTIVES

The objective of the EDF Energy creep irradiation capsules is to provide data to support an improved understanding of certain aspects of the creep behavior, particularly at very high neutron dose.

2 INTRODUCTION

The EDF Energy creep rabbit capsules are HFIR target region capsules that will provide data for the high dose creep behavior of AGR moderator graphite. The creep rabbits will be irradiated in the target region of HFIR for five irradiation periods over several years. The capsules design irradiation temperatures is 420°C. This creep irradiation series is Experiment 2 in EDF Energy's experimental notation [1].

The data obtained for each grade of graphite included in the HTV capsule include:

- Dimensional, volume and density changes
- Creep strain
- Elastic modulus (E) from sonic velocity
- Coefficient of Thermal Expansion (CTE)
- Microstructural characterization via XRD and tomography.

The EDF Energy graphite specimens have a rectangular cross-section with nominal dimensions of 5.5 mm x 6.0 mm and a length of 12.5 mm. Temperature control is achieved by setting the initial gas gaps in the capsule. The capsules will be sealed and filled with inert gas. A full design assessment of the thermal design of the capsule has been provided to EDF Energy.

The experimental results will assist in the development and validation of future models of irradiation induced creep of graphite by providing the following data:

- Inert creep strain data from low to lifetime AGR fluence
- Inert creep-property data (especially CTE) from low to lifetime AGR fluence
- Effect of oxidation on creep modulus (by indirect comparison with experiment 1 and direct comparison with experiment 3 – N.B., Experiment 1 and 3 are not covered here)
- Data to develop a mechanistic understanding, including
    - Appropriate creep modulus (effect of pinning)
    - Investigation of CTE-creep strain behavior under inert conditions
    - Information on the effect of creep strain on crystallite orientation (requires XRD)
    - Effect of creep strain in micro-porosity (requires tomography & microscopy)

This document describes the experimental work conducted to meet the requirements of project technical specification [1] and EDF Energy requests for additional Pre-IE work, as well as the planned PIE work.

3 CREEP RABBIT REQUIREMENTS

The key technical requirements of the experiment are:

- Virgin graphite IM1-24 supplied by EDF Energy shall be used
- One stressed and one unstressed specimens per capsule
- Four capsules per irradiation segment (five during first irradiation segment – calibration rabbit)
- Specimen geometry is 6x5.5x12.5 mm (longer dimension in the stressed direction)
- Applied creep stress = 10 MPa compressive stress
- Irradiation temperature = 420°C
- Allowable irradiation temperature variation ± 40°C
- SiC monitors to be used – see subsequent discussion in Section 9
- Five irradiation segments:
  - 1st irradiation 4 X 10^{20} n/cm^2 EDN (<1 cycle in HFIR target)
  - 2nd irradiation 30 X 10^{20} n/cm^2 EDN (<3 cycles in HFIR target)
  - 3rd irradiation 45 X 10^{20} n/cm^2 EDN (<4 cycles in HFIR target)
  - 4th irradiation 45 X 10^{20} n/cm^2 EDN (<4 cycles in HFIR target)
  - 5th irradiation 45 X 10^{20} n/cm^2 EDN (<4 cycles in HFIR target)

Table 1 Required irradiation conditions per irradiation period at HIFR

Irradiation Segment	Neutron Dose		Design Irradiation Temperature	HFIR Cycles @ Peak
	EDN	DPA	°C	
1	4.00E+20	0.525	420	< 1
2	3.00E+21	3.939	420	< 2
3	4.50E+21	5.909	420	< 3
4	4.50E+21	5.909	420	< 3
5	4.50E+21	5.909	420	< 3

The fast neutron flux for each Rabbit position/channel is given in Table 2.

Table 2 HFIR channel/position rabbit approximate neutron fluxes [E>0.1 MeV]

						PTP		Outer Ring Target			Hydraulic Tube		
							fast flux			fast flux			fast flux
		From	From	Q		flux	1.11E+15		flux	1.09E+15		flux	8.90E+14
	z	C-L (in)	C-L (cm)	pf(z)	Pos	pf(z)		Pos	pf(z)		Pos	pf(z)	
	10.8	10.8	27.43	0.443		0.272	3.02E+14		0.259	2.82E+14		0.355	3.16E+14
	10.6	10.6	26.92	0.457		0.298	3.31E+14		0.286	3.12E+14	-	0.378	3.37E+14
Active	10.4	10.4	26.42	0.470		0.325	3.61E+14		0.313	3.41E+14	9	0.402	3.58E+14
Fuel	10.2	10.2	25.91	0.484		0.351	3.89E+14		0.339	3.69E+14	-	0.425	3.78E+14
Top	10	10	25.40	0.490		0.376	4.17E+14		0.365	3.97E+14		0.447	3.98E+14
	9.8	9.8	24.89	0.512		0.401	4.45E+14		0.390	4.25E+14		0.469	4.17E+14
	9.6	9.6	24.38	0.526		0.425	4.72E+14		0.414	4.52E+14		0.490	4.37E+14
	9.4	9.4	23.88	0.540		0.449	4.98E+14		0.438	4.78E+14		0.512	4.55E+14
	9.2	9.2	23.37	0.554		0.472	5.24E+14		0.462	5.04E+14		0.532	4.74E+14
	9	9	22.86	0.569		0.495	5.49E+14		0.485	5.29E+14		0.552	4.92E+14
	8.8	8.8	22.35	0.583		0.517	5.74E+14		0.508	5.54E+14		0.572	5.09E+14
	8.6	8.6	21.84	0.597	-	0.539	5.98E+14		0.530	5.78E+14		0.591	5.26E+14
	8.4	8.4	21.34	0.611	8	0.560	6.22E+14		0.552	6.01E+14		0.610	5.43E+14
	8.2	8.2	20.83	0.626	-	0.581	6.45E+14		0.573	6.24E+14		0.629	5.59E+14
	8	8	20.32	0.640		0.601	6.68E+14		0.593	6.47E+14	-	0.647	5.75E+14
	7.8	7.8	19.81	0.654		0.621	6.89E+14	-	0.613	6.69E+14	8	0.664	5.91E+14
	7.6	7.6	19.30	0.669		0.640	7.11E+14	7	0.633	6.90E+14	-	0.681	6.06E+14
	7.4	7.4	18.80	0.683		0.659	7.32E+14	-	0.652	7.11E+14		0.698	6.21E+14
	7.2	7.2	18.29	0.697		0.677	7.52E+14		0.671	7.31E+14		0.714	6.35E+14
	7	7	17.78	0.711		0.695	7.72E+14		0.689	7.51E+14		0.730	6.49E+14
	6.8	6.8	17.27	0.724		0.713	7.91E+14		0.706	7.70E+14		0.745	6.63E+14
	6.6	6.6	16.76	0.738		0.729	8.09E+14		0.723	7.88E+14		0.760	6.76E+14
	6.4	6.4	16.26	0.752		0.746	8.28E+14		0.740	8.06E+14		0.774	6.89E+14
	6.2	6.2	15.75	0.765		0.761	8.45E+14		0.756	8.24E+14		0.788	7.01E+14
	6	6	15.24	0.778	-	0.777	8.62E+14		0.771	8.41E+14		0.802	7.13E+14
	5.8	5.8	14.73	0.791	7	0.791	8.78E+14		0.786	8.57E+14		0.815	7.25E+14
	5.6	5.6	14.22	0.804	-	0.806	8.94E+14		0.801	8.73E+14		0.827	7.36E+14
	5.4	5.4	13.72	0.816		0.819	9.09E+14		0.815	8.88E+14	-	0.840	7.47E+14
	5.2	5.2	13.21	0.828		0.832	9.24E+14	-	0.828	9.03E+14	7	0.851	7.58E+14
	5	5	12.70	0.840		0.845	9.38E+14	6	0.841	9.17E+14	-	0.863	7.68E+14
	4.8	4.8	12.19	0.852		0.857	9.52E+14	-	0.854	9.30E+14		0.873	7.77E+14
	4.6	4.6	11.68	0.863		0.869	9.65E+14		0.866	9.43E+14		0.884	7.87E+14
	4.4	4.4	11.18	0.874		0.880	9.77E+14		0.877	9.56E+14		0.894	7.95E+14
	4.2	4.2	10.67	0.884		0.891	9.89E+14		0.888	9.68E+14		0.903	8.04E+14
	4	4	10.16	0.894		0.901	1.00E+15		0.898	9.79E+14		0.912	8.12E+14
	3.8	3.8	9.65	0.904		0.911	1.01E+15		0.908	9.90E+14		0.921	8.20E+14
	3.6	3.6	9.14	0.914		0.920	1.02E+15		0.918	1.00E+15		0.929	8.27E+14
	3.4	3.4	8.64	0.923	-	0.929	1.03E+15		0.927	1.01E+15		0.937	8.34E+14
	3.2	3.2	8.13	0.931	6	0.937	1.04E+15		0.935	1.02E+15		0.944	8.40E+14
	3	3	7.62	0.939	-	0.945	1.05E+15		0.943	1.03E+15		0.951	8.46E+14
	2.8	2.8	7.11	0.947		0.952	1.06E+15		0.950	1.04E+15	-	0.957	8.52E+14
	2.6	2.6	6.60	0.954		0.959	1.06E+15	-	0.957	1.04E+15	6	0.963	8.57E+14
	2.4	2.4	6.10	0.961		0.965	1.07E+15	5	0.963	1.05E+15	-	0.969	8.62E+14
	2.2	2.2	5.59	0.967		0.971	1.08E+15	-	0.969	1.06E+15		0.974	8.67E+14
	2	2	5.08	0.972		0.976	1.08E+15		0.975	1.06E+15		0.978	8.71E+14
	1.8	1.8	4.57	0.978		0.981	1.09E+15		0.979	1.07E+15		0.983	8.74E+14
	1.6	1.6	4.06	0.982		0.985	1.09E+15		0.984	1.07E+15		0.986	8.78E+14
	1.4	1.4	3.56	0.986		0.988	1.10E+15		0.988	1.08E+15		0.990	8.81E+14
	1.2	1.2	3.05	0.990		0.992	1.10E+15		0.991	1.08E+15		0.992	8.83E+14
	1	1	2.54	0.993		0.994	1.10E+15		0.994	1.08E+15		0.995	8.85E+14
	0.8	0.8	2.03	0.996	-	0.996	1.11E+15		0.996	1.09E+15		0.997	8.87E+14
	0.6	0.6	1.52	0.997	5	0.998	1.11E+15		0.998	1.09E+15		0.998	8.88E+14
	0.4	0.4	1.02	0.999	-	0.999	1.11E+15		0.999	1.09E+15		0.999	8.89E+14
	0.2	0.2	0.51	1.000		1.000	1.11E+15		1.000	1.09E+15	-	1.000	8.90E+14
C-L	0.0	0	0.00	1.000		1.000	1.11E+15	-	1.000	1.09E+15	5	1.000	8.90E+14
	-0.2	0.2	0.51	1.000		1.000	1.11E+15	4	1.000	1.09E+15	-	1.000	8.90E+14
	-0.4	0.4	1.02	0.999		0.999	1.11E+15	-	0.999	1.09E+15		0.999	8.89E+14
	-0.6	0.6	1.52	0.997		0.998	1.11E+15		0.998	1.09E+15		0.998	8.88E+14
	-0.8	0.8	2.03	0.996		0.996	1.11E+15		0.996	1.09E+15		0.997	8.87E+14
	-1	1	2.54	0.993		0.994	1.10E+15		0.994	1.08E+15		0.995	8.85E+14
	-1.2	1.2	3.05	0.990		0.992	1.10E+15		0.991	1.08E+15		0.992	8.83E+14
	-1.4	1.4	3.56	0.986		0.988	1.10E+15		0.988	1.08E+15		0.990	8.81E+14
	-1.6	1.6	4.06	0.982		0.985	1.09E+15		0.984	1.07E+15		0.986	8.78E+14
	-1.8	1.8	4.57	0.978		0.981	1.09E+15		0.979	1.07E+15		0.983	8.74E+14
	-2	2	5.08	0.972	-	0.976	1.08E+15		0.975	1.06E+15		0.978	8.71E+14
	-2.2	2.2	5.59	0.967	4	0.971	1.08E+15		0.969	1.06E+15		0.974	8.67E+14
	-2.4	2.4	6.10	0.961	-	0.965	1.07E+15		0.963	1.05E+15	-	0.969	8.62E+14
	-2.6	2.6	6.60	0.954		0.959	1.06E+15	-	0.957	1.04E+15	4	0.963	8.57E+14
	-2.8	2.8	7.11	0.947		0.952	1.06E+15	3	0.950	1.04E+15	-	0.957	8.52E+14
	-3	3	7.62	0.939		0.945	1.05E+15	-	0.943	1.03E+15		0.951	8.46E+14
	-3.2	3.2	8.13	0.931		0.937	1.04E+15		0.935	1.02E+15		0.944	8.40E+14
	-3.4	3.4	8.64	0.923		0.929	1.03E+15		0.927	1.01E+15		0.937	8.34E+14
	-3.6	3.6	9.14	0.914		0.920	1.02E+15		0.918	1.00E+15		0.929	8.27E+14
	-3.8	3.8	9.65	0.904		0.911	1.01E+15		0.908	9.90E+14		0.921	8.20E+14
	-4	4	10.16	0.894		0.901	1.00E+15		0.898	9.79E+14		0.912	8.12E+14
	-4.2	4.2	10.67	0.884		0.891	9.89E+14		0.888	9.68E+14		0.903	8.04E+14
	-4.4	4.4	11.18	0.874	-	0.880	9.77E+14		0.877	9.56E+14		0.894	7.95E+14
	-4.6	4.6	11.68	0.863	3	0.869	9.65E+14		0.866	9.43E+14		0.884	7.87E+14
	-4.8	4.8	12.19	0.852	-	0.857	9.52E+14		0.854	9.30E+14		0.873	7.77E+14
	-5	5	12.70	0.840		0.845	9.38E+14		0.841	9.17E+14	-	0.863	7.68E+14
	-5.2	5.2	13.21	0.828		0.832	9.24E+14	-	0.828	9.03E+14	3	0.851	7.58E+14
	-5.4	5.4	13.72	0.816		0.819	9.09E+14	2	0.815	8.88E+14	-	0.840	7.47E+14
	-5.6	5.6	14.22	0.804		0.806	8.94E+14	-	0.801	8.73E+14		0.827	7.36E+14
	-5.8	5.8	14.73	0.791		0.791	8.78E+14		0.786	8.57E+14		0.815	7.25E+14
	-6	6	15.24	0.778		0.777	8.62E+14		0.771	8.41E+14		0.802	7.13E+14
	-6.2	6.2	15.75	0.765		0.761	8.45E+14		0.756	8.24E+14		0.788	7.01E+14
	-6.4	6.4	16.26	0.752		0.746	8.28E+14		0.740	8.06E+14		0.774	6.89E+14
	-6.6	6.6	16.76	0.738		0.729	8.09E+14		0.723	7.88E+14		0.760	6.76E+14
	-6.8	6.8	17.27	0.724		0.713	7.91E+14		0.706	7.70E+14		0.745	6.63E+14
	-7	7	17.78	0.711	-	0.695	7.72E+14		0.689	7.51E+14		0.730	6.49E+14
	-7.2	7.2	18.29	0.697	2	0.677	7.52E+14		0.671	7.31E+14		0.714	6.35E+14
	-7.4	7.4	18.80	0.683	-	0.659	7.32E+14		0.652	7.11E+14		0.698	6.21E+14
	-7.6	7.6	19.30	0.669		0.640	7.11E+14		0.633	6.90E+14		0.681	6.06E+14
	-7.8	7.8	19.81	0.654		0.621	6.89E+14	-	0.613	6.69E+14	-	0.664	5.91E+14
	-8	8	20.32	0.640		0.601	6.68E+14	1	0.593	6.47E+14	2	0.647	5.75E+14
	-8.2	8.2	20.83	0.626		0.581	6.45E+14	-	0.573	6.24E+14	-	0.629	5.59E+14
	-8.4	8.4	21.34	0.611		0.560	6.22E+14		0.552	6.01E+14		0.610	5.43E+14
	-8.6	8.6	21.84	0.597		0.539	5.98E+14		0.530	5.78E+14		0.591	5.26E+14
	-8.8	8.8	22.35	0.583		0.517	5.74E+14		0.508	5.54E+14		0.572	5.09E+14
	-9	9	22.86	0.569		0.495	5.49E+14		0.485	5.29E+14		0.552	4.92E+14
	-9.2	9.2	23.37	0.554		0.472	5.24E+14		0.462	5.04E+14		0.532	4.74E+14
	-9.4	9.4	23.88	0.540		0.449	4.98E+14		0.438	4.78E+14		0.512	4.55E+14
	-9.6	9.6	24.38	0.526		0.425	4.72E+14		0.414	4.52E+14		0.490	4.37E+14
	-9.8	9.8	24.89	0.512		0.401	4.45E+14		0.390	4.25E+14		0.469	4.17E+14
Bottom	10	10	25.40	0.490		0.376	4.17E+14		0.365	3.97E+14		0.447	3.98E+14
	-10.2	10.2	25.91	0.484		0.351	3.89E+14		0.339	3.69E+14	-	0.425	3.78E+14
	-10.4	10.4	26.42	0.470		0.325	3.61E+14		0.313	3.41E+14	1	0.402	3.58E+14
	-10.6	10.6	26.92	0.457		0.298	3.31E+14		0.286	3.12E+14	-	0.378	3.37E+14
	-10.8	10.8	27.43	0.443		0.272	3.02E+14		0.259	2.82E+14		0.355	3.16E+14
	-11	11	27.94	0.430		0.244	2.71E+14		0.231	2.52E+14		0.331	2.94E+14
	-11.2	11.2	28.45	0.417		0.216	2.40E+14		0.203	2.21E+14		0.306	2.72E+14
	-11.4	11.4	28.96	0.404		0.188	2.09E+14		0.174	1.90E+14		0.281	2.50E+14
	-11.6	11.6	29.46	0.391		0.159	1.77E+14		0.145	1.58E+14		0.255	2.27E+14

The second segment irradiations will be conducted in the HFIR Target Rod Rabbit Holder (TRRH) vertical channel positions 3, 4 or 5. There are 20 horizontal tubes, each of which contains up to 7 rabbits (Table 2 – Outer Ring Target flux). The EDF target for the second irradiation segment is 3 x 10^{21} n/cm^2 EDN (3.94 DPA). The estimated segment two doses for two or three HFIR cycles are given in Table 3. The flux varies along the capsule length for position 3/5 rabbit capsules from 1.14 x 10^{15} n/cm^2.s [E>0.1MeV] to 1.04 x 10^{15} n/cm^2.s [E>0.1MeV] (due to flux buckling). Position 4 is on the HFIR mid plane and the flux hardly varies along the capsule length, but there is slight radial variation. The position 4 rabbit peak flux is 1.04 x 10^{15} n/cm^2.s [E>0.1MeV] for group 2 rabbits and 1.10 x 10^{15} n/cm^2.s [E>0.1MeV] for group 4 rabbits. The second EDF irradiation segment shall be of two cycle's duration. Thus the mid capsule fluences for EDF capsules 1-4 are 3.52, 3.31, 3.52 and 3.50 DPA, respectively. Note, the HFIR Target is divided for administrative purposes into 6 radial zones or groups (numbered 1-6, based on flux), with group I being at the target center. The TRRH could be in groups 1-5 and the hydraulic tube is located in group 3. Thus each creep rabbit capsule occupies a single position within the HFIR core. N.B., there is only a calibration rabbit in irradiation segment 1.

Table 3 Rabbit flux and time for second (segment 2) HFIR irradiation

EDF Rabbit Numbers	HFIR Location	Axial location	In-Rabbit location	HFIR Flux [E>0.1 MeV]	Target Fluence EDN	Target Fluence	Target Fluence [E>.1 MeV]	HFIR Cycle length	Ideal # Cycles	2 Cycles			3 Cycles		
		cm		$n/cm^2.s$	n/cm^2	DPA	n/cm2	Days		n/cm^2 (EDN)	n/cm^2 [E>0.1 MeV]	DPA	n/cm^2 (EDN)	n/cm^2 [E>0.1 MeV]	DPA
EDF1	B5-3 (Group 5)	4.1	top	1.14E+15	3.00E+21	3.94	5.40E+21	25.25	2.17	2.77E+21	4.98E+21	3.63	4.16E+21	7.47E+21	5.45
	B5-3 (Group 5)	7.0	mid-plane	1.10E+15	3.00E+21	3.94	5.40E+21	25.25	2.24	2.68E+21	4.82E+21	3.52	4.03E+21	7.22E+21	5.27
	B5-3 (Group 5)	10.2	bottom	1.04E+15	3.00E+21	3.94	5.40E+21	25.25	2.38	2.53E+21	4.54E+21	3.31	3.79E+21	6.81E+21	4.97
EDF2	C3-4 (Group 2)	3.1	top	1.03E+15	3.00E+21	3.94	5.40E+21	25.25	2.40	2.50E+21	4.49E+21	3.28	3.76E+21	6.74E+21	4.92
	C3-4 (Group 2)	0.0	mid-plane	1.04E+15	3.00E+21	3.94	5.40E+21	25.25	2.38	2.53E+21	4.54E+21	3.31	3.79E+21	6.81E+21	4.97
	C3-4 (Group 2)	3.1	bottom	1.03E+15	3.00E+21	3.94	5.40E+21	25.25	2.40	2.50E+21	4.49E+21	3.28	3.76E+21	6.74E+21	4.92
EDF3	F7-3 (Group 5)	4.1	top	1.14E+15	3.00E+21	3.94	5.40E+21	25.25	2.17	2.77E+21	4.98E+21	3.63	4.16E+21	7.47E+21	5.45
	F7-3 (Group 5)	7.0	mid-plane	1.10E+15	3.00E+21	3.94	5.40E+21	25.25	2.24	2.68E+21	4.82E+21	3.52	4.03E+21	7.22E+21	5.27
	F7-3 (Group 5)	10.2	bottom	1.04E+15	3.00E+21	3.94	5.40E+21	25.25	2.38	2.53E+21	4.54E+21	3.31	3.79E+21	6.81E+21	4.97
EDF4	D2-4 (Group 4)	3.1	top	1.09E+15	3.00E+21	3.94	5.40E+21	25.25	2.27	2.65E+21	4.75E+21	3.47	3.97E+21	7.13E+21	5.21
	D2-4 (Group 4)	0.0	mid-plane	1.10E+15	3.00E+21	3.94	5.40E+21	25.25	2.25	2.67E+21	4.80E+21	3.50	4.01E+21	7.20E+21	5.26
	D2-4 (Group 4)	3.1	bottom	1.09E+15	3.00E+21	3.94	5.40E+21	25.25	2.27	2.65E+21	4.75E+21	3.47	3.97E+21	7.13E+21	5.21

4 MATERIALS AND SPECIMEN MARKING

4.1 Materials

The virgin graphite specimen to be irradiated in the EDF Energy creep rabbits are un-irradiated grade IM1-24. The specimens are from Hunterston Brick 8 (Quadrant 8/B/4) and have been machined by NRG (The Netherlands) and supplied to ORNL by EDF Energy (Fig. 1).

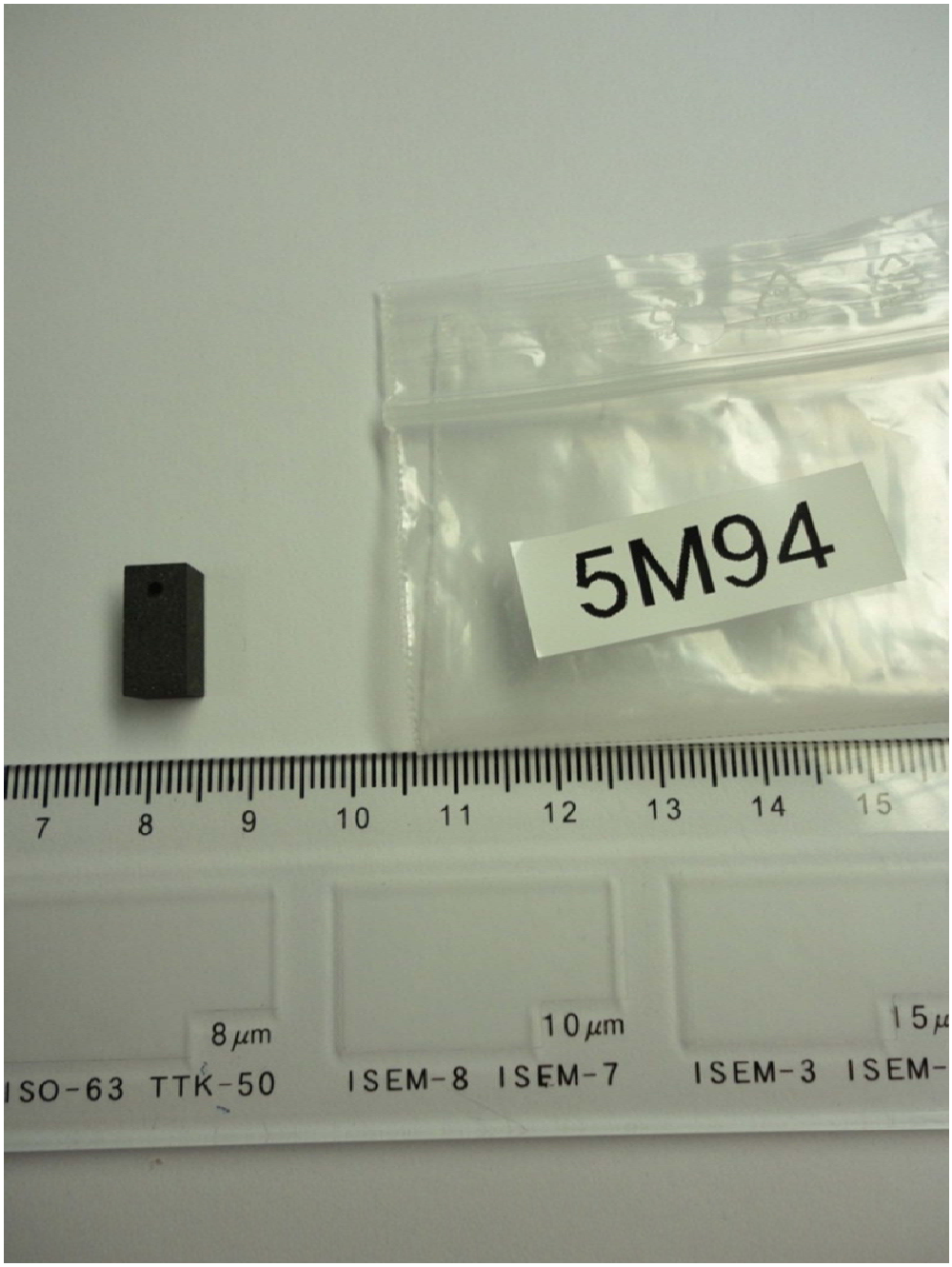

Figure 1 EDF Energy "spare" specimen

The EDF Energy specimen identities are given in Table 4. Each specimen has a LASER engraved "dot" on one of the 6 x 12.5 mm faces to indicate specimen orientation. In all cases the length of the specimen is machined parallel to the axial brick direction and the LASER "dot" is in the upper half of the hoop face (Fig. 1).

Table 4 Specimens supplied by EDF Energy and their associated identities

Sample ID	Purpose	Packaging	LASER Marking
5M68	RR/Pre-IE	Engraved Al can	U1
5M70	RR/Pre-IE	Engraved Al can	U2
5M72	RR/Pre-IE	Engraved Al can	U3
5M74	RR/Pre-IE	Engraved Al can	U4
5M76	RR/Pre-IE	Engraved Al can	U5
5M78	RR/Pre-IE	Engraved Al can	U6
5M80	RR/Pre-IE	Engraved Al can	S1
5M82	RR/Pre-IE	Engraved Al can	S2
5M84	RR/Pre-IE	Engraved Al can	S3
5M86	RR/Pre-IE	Engraved Al can	S4
5M88	RR/Pre-IE	Engraved Al can	S5
5M90	RR/Pre-IE	Engraved Al can	S6
5M91	Chemical Analysis/Capsule Design	Grip seal bag	-
5M92	Chemical Analysis/Capsule Design	Grip seal bag	-
5M93	Chemical Analysis/Capsule Design	Grip seal bag	-
5M94	Chemical Analysis/Capsule Design	Grip seal bag	-
5M95	Chemical Analysis/Capsule Design	Grip seal bag	-

4.2 Specimen Marking

The EDF Energy specimens shall be LASER engraved with a unique number. The specimen marking code is denoted in Table 4. The specimen number shall be LASER engraved centrally on the "dot" face as indicated in Fig. 2 & 3. The specimen identities are linked to engraved Al-can sample ID numbers in Table 4.

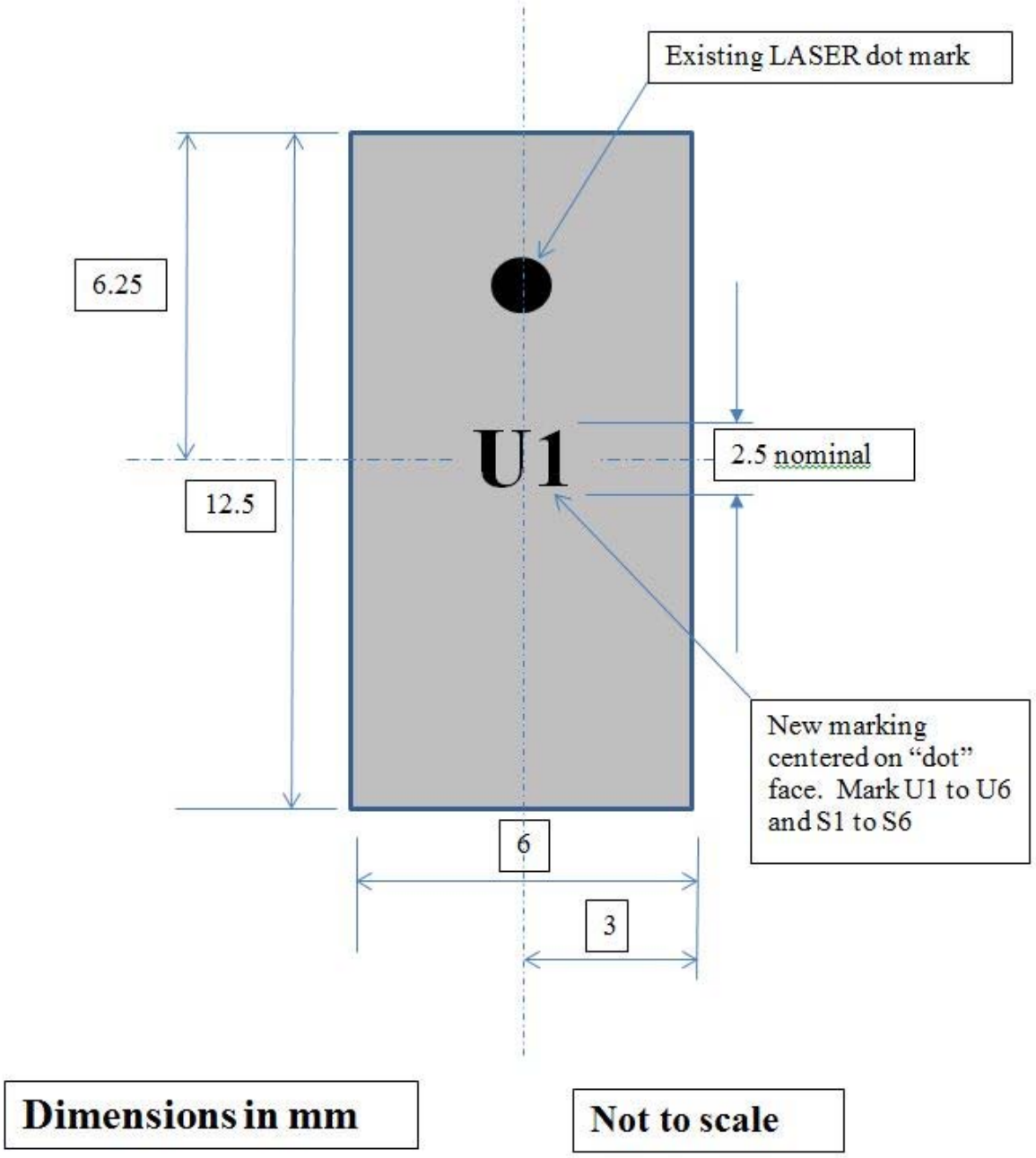

Figure 2 LASER marking scheme for EDF Energy specimens

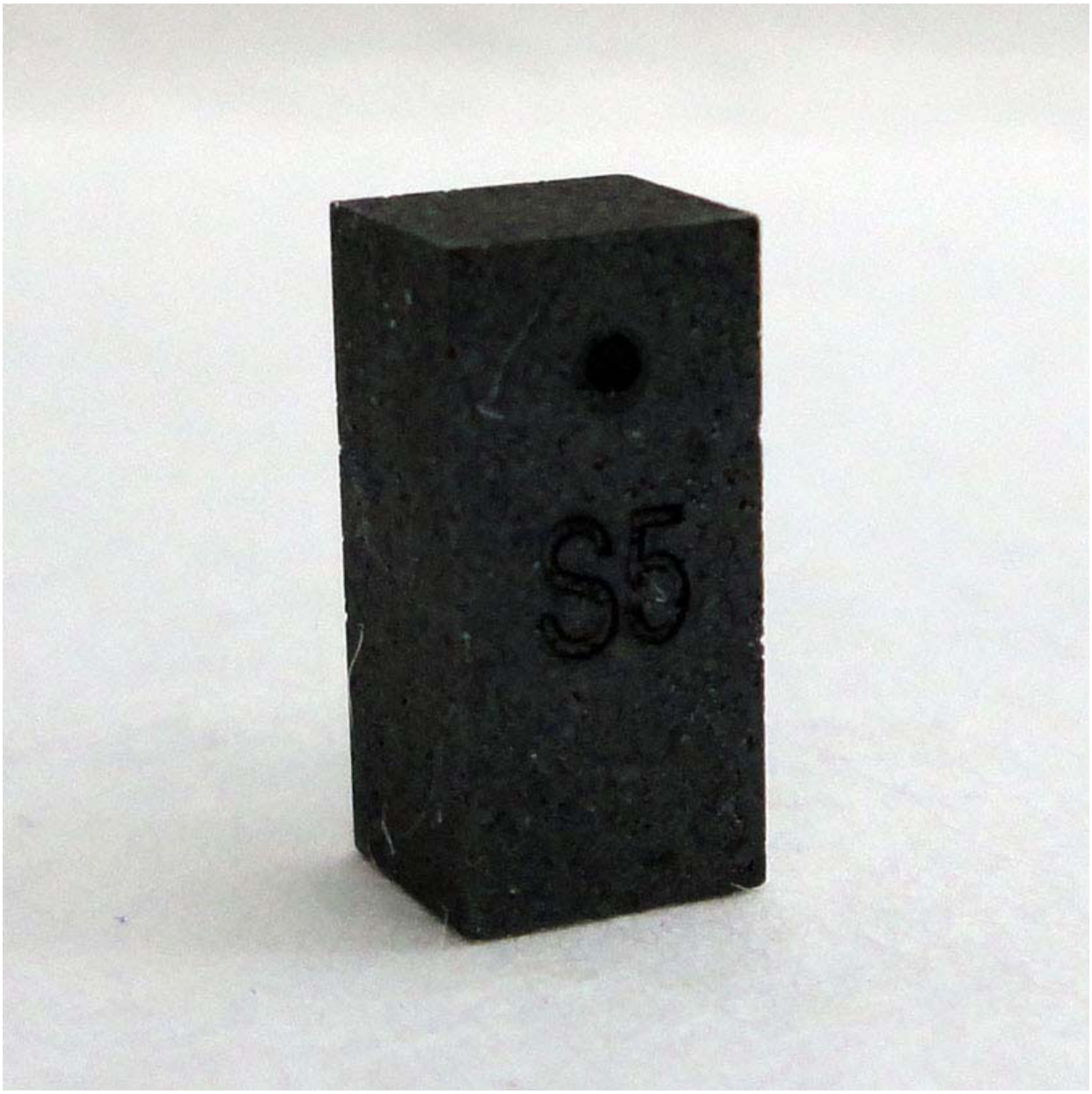

Figure 3 EDF Energy Specimen S5 indicting "dot" face

4.3 Specimen pair selection

Figure 4 shows the NRG specimen machining plan for the virgin experiments (E2a and E2) planned at NRG and ORNL.

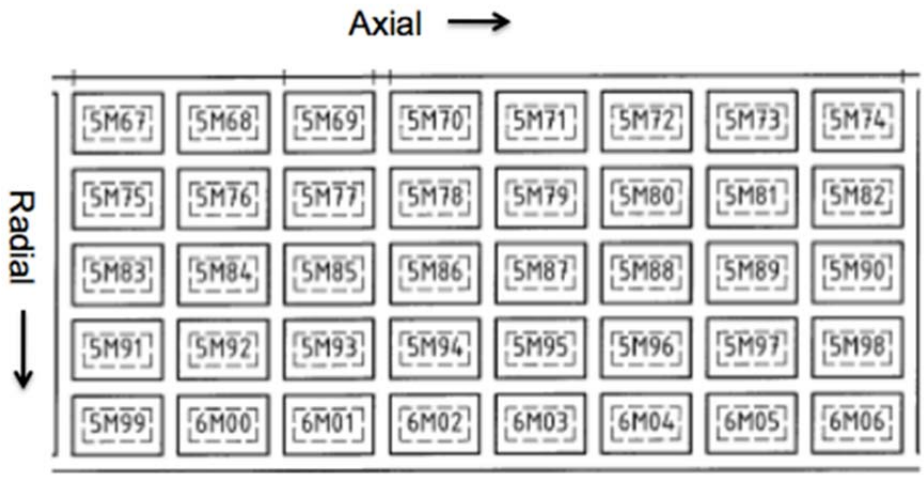

Figure 4 Specimen layout and cutting plan

Radially adjacent specimens have been paired for each facility e.g. 5M67 and 5M75 at NRG. Also the pairs at NRG and ORNL are vertically adjacent i.e. P1 NRG (5M67 and 5M75) is vertically adjacent to P1 ORNL (5M68 and 5M76). This will facilitate comparison of creep strains later in the experimental program.

Table 5 ORNL Specimen pairings

ORNL				
Pair	**Code**	**Selection**	**Storage**	**I.D.**
P1a	5M68	RR/Pre-IE	Engraved Al can	U1
P1b	5M76	RR/Pre-IE	Engraved Al can	U5
P2a	5M70	RR/Pre-IE	Engraved Al can	U2
P2b	5M78	RR/Pre-IE	Engraved Al can	U6
P3a	5M72	RR/Pre-IE	Engraved Al can	U3
P3b	5M80	RR/Pre-IE	Engraved Al can	S1
P4a	5M74	RR/Pre-IE	Engraved Al can	U4
P4b	5M82	RR/Pre-IE	Engraved Al can	S2
P5a	5M84	Spare	Engraved Al can	S3
P5b	5M86	Spare	Engraved Al can	S4
P6a	5M88	Spare	Engraved Al can	S5
P6b	5M90	Spare	Engraved Al can	S6

The spare pairs for ORNL that have been pre-characterized by NRG are also adjacent pairs but unfortunately the spare pairs that NRG have pre-characterized are not adjacent pairs. Therefore should additional pairs be required beyond the 4 selected pairs, e.g. to investigate effect of pre-stress on primary creep strain additional Pre-IE may be required to enable adjacent specimens to be selected for unload/load pairs etc.

P1-P5 will be subjected to further Pre-IE testing. P5 shall be installed in the calibration rabbit and P6 shall be retained by the capsule assembly team for demonstration purposes.

The specimen loading matrix is shown in Table 6.

Table 6 Specimen Loading matrix for the all irradiation segments

Loading Matrix EDF Specimens (07-18-2013)									
		Bellows pressurization data (1st irr. Segment)*							
		Force	Est. stress	Error					
Capsule No	Loaded	N	MPa	%	Spec ID		Control	Spec ID	Purpose
EDF01	P1a	137	9.6	-4.1	5M68		P1b	5M76	Creep Strain
EDF02	P2a	142	9.9	-0.6	5M70		P2b	5M78	Creep Strain
EDF03	P3a	132	9.2	-7.6	5M72		P3b	5M80	Creep Strain
EDF04	P4a	152	10.6	6.4	5M74		P4b	5M82	Creep Strain
EDF05	P5a	148	10.4	3.6	5M84		P5b	5M86	Calibration (thermal)

***The bellows load targets for subsequent irradiation cycles is yet to be established and depends on the extent of bellows annealing**

After assembly (bellows gas fill and weld sealing) the bellows force is measured by compressing the bellows. Thus the compressive creep load on the specimens is fixed and determined. The bellows are also tested post irradiation to provide confidence that the creep force has been maintained (Section 8). The pre- and post-irradiation bellows testing are repeated for every irradiation segment.

5 MATERIAL PRE-IRRADIATION EXAMINATION

5.1 Chemical Impurity Analysis

Spare specimens 5M91, 5M93 and 5M95 (Table 4) were submitted to Evans Analytical Inc. for destructive elemental chemical analysis. Satisfactory chemical analysis is a HFIR requirement. Low Cobalt concentration (less the 5 ppm) is particularly desirable. Chemical analysis has been completed and no impurity related impediment to irradiation is foreseen. These data have been supplied to EDF.

5.2 Pre-IE activity sequence

The order of conducting Pre-IE activities is reported below.

1. Visual inspection and digital photography (12 specimens, P1-P6)
2. Ultrasonically clean (briefly clean ultrasonically in ethanol bath)
3. Before Pre-Stress - Round Robin Pre-IE on all 8 specimens selected for irradiation (P1-P4):
    a. Dimensions & Mass
    b. CTE (RT-420°C)
    c. ER
    d. DYM E (Sonic velocity). (DYM to use dry probes – no couplants, 2.25 MHz probes to be used)
4. Before Pre-stress - Optional Pre-IE on 4 specimens only (i.e. P1 and P2)
    a. XRD and Pole Figures
    b. Digital tomography at lower resolution (full volume) with one highest resolution pass on mid-section (highest resolution) per sample
5. Pre-stress, 10.0 MPa compressive stress, two stress cycles (P1-P4)
6. Post Pre-Stress - Round Robin Pre-IE on all 8 specimens selected for irradiation (P1-P4):
    a. Dimensions & Mass
    b. CTE (RT-420°C)
    c. ER
    d. DYM E (Sonic velocity). (DYM to use dry probes – no couplants)
7. Post Pre-stress - Optional Pre-IE on all 8 specimens selected for irradiation (P1-P4):
    a. XRD and Pole Figures
    b. Digital tomography at lower resolution (full volume) with one highest resolution pass on mid-section (highest resolution) per sample
8. Encapsulate in creep rabbit capsules (P1-P4)

The experimental data will be contained in a final Pre-IE report. Detailed test methods are given below. Testing will follow the prescribed ASTM Guidelines [2].

5.2.1 Visual inspection & digital photography

All specimens will be visually inspected and digitally photographed before the start of Pre-IE or PIE. The purpose of the visual inspection/photography is to provide a qualitative appraisal and record of the condition of the sample and suitability for more detailed measurements. Any distinguishing features (including machining defects, porosity, fractured surfaces, etc.) will be recorded. Photography will be utilized to provide an accurate record of all distinguishing features. Digital photographs shall be taken so as to show three sides of the specimen per picture, the specimen shall be rotated between pictures, thus each specimen is captured in a pair of photographs. Prior to PIE (after segment 1) photography was switched to the Keyence LASER scanning microscope. Keyence images were taken on all eight faces of the specimens.

5.2.2 Pre-stress testing

Before specimen pre-stressing at 10MPa (section 5.2.3) the following tests shall be performed on the specimen pairs (Table 5) indicated below:

Specimen pairs P1-P4: Mass, dimension, CTE, DYM, ER

Specimen pairs P1-P2: XRD and pole figures, Tomography

5.2.3 Specimen pre-stress

Prior to Pre-IE and optional Pre-IE testing, the samples shall be pre-stressed in compression for a total of two cycles to 10 MPa maximum stress at each cycle. A 10MPa compressive stress equates to an applied compressive force of 330N. An MTS-Insight electro mechanical load frame (Fig 5) with a 2KN load cell shall be used. The crosshead speed will be 10μ/min.

We shall capture the load deflection curve from the crosshead displacement since the specimen is too small for a strain gauge or clip displacement gauge. A video camera set-up will capture the proof-stress test and allows for digital image correlation. Stress-strain curves are thus produced.

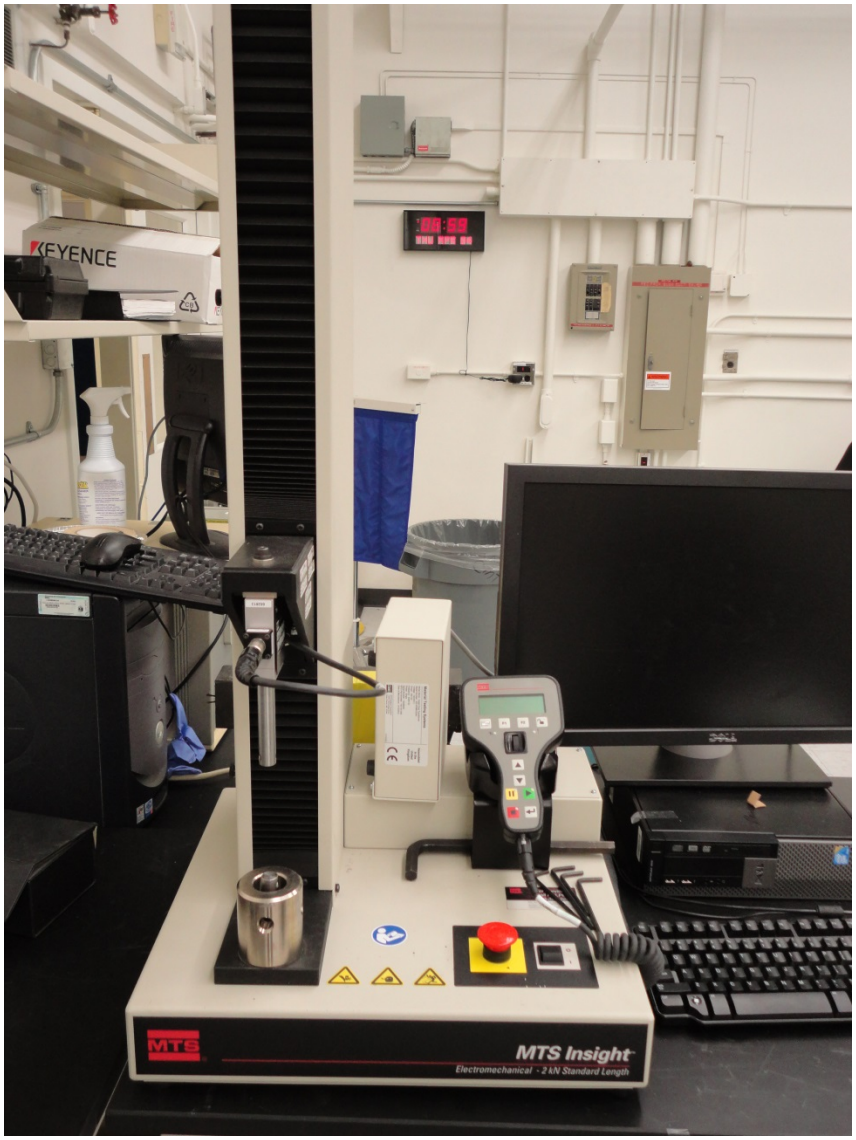

Figure 5 The "Insight" MTS load-frame to be used for pre-stressing

5.2.4 Specimen dimensions, mass, and density

The specimen dimensions shall be measured using calibrated, certified micrometers, with resolution of 0.001 mm, 0-25 mm range and 6.3 mm diameter anvils). The measurements are taken in accordance with ASTM C559-90 (Reapproved 1995) "Standard Method for Bulk Density by Physical Measurements of Manufactured Carbon and Graphite Articles" [3]. The mean specimen length is determined from four length measurements and the mean width and breadth each from four individual measurements equally spaced along the specimen length. In addition the Standard Operating Procedure: Specimen Dimensional Inspection shall be followed. The mass is determined using a calibrated microbalance, accurate to 0.1 mg, calibrated with

NIST (National Institute of Standard and Technology) traceable test weights. The bulk density calculated in accordance with ATSM C559 [3].

ASTM Standard C559 requires the specimen volume to be a minimum of 500 mm^3. The irradiated specimen geometries will fall below this threshold. Moreover, C559 also calls for the samples smallest dimension to be at least 10 times the length of the largest visible particle. Assuming the mean filler particle size of the graphite provided is 0.5 – 0.6 mm, the 5.5 mm specimen size is on the edge of this lower bound. Despite these specimen size issues we propose that dimensions, mass, and bulk density shall be determined according to ASTM C599 for all specimens.

ASTM C559 [3] calls for a mass and dimensional measurement accuracy within 0.05%. For these specimens (taking 5.5 mm width as the smallest dimension) 0.05% accuracy requires a resolution better than 2.75 μm. We shall use a digital micrometer with a resolution of 0.001 μm. For mass determination, a digital microbalance with a resolution of 0.1 mg shall be used (0.05% accuracy requires a resolution of better than ~0. 4 mg on a mass of ~0.75 g).

The specimens shall be wiped clean and oven dried per ASTM C559 prior to dimensions, mass, and density determination.

5.2.5 Coefficient of thermal expansion

Thermal expansion shall be obtained in accordance with ASTM E228-11. A Netzsch DIL 402 CD dilatometer shall be used (Figs. 6 and 7) to make the expansion measurements (s/n 219 7 038 G in-zone and 2197 041G out of zone). These dilatometers have horizontal push rods. All expansion shall be made using 25°C as the reference temperature. Average CTE shall be reported for each specimen from the second heating cycle for the temperature range 150-300°C and 150-420°C. The first heating cycle (at 20°C/min) shall be for settling the specimen prior to the testing (2^{nd}) cycle (at 5°C/min).

The constant ramp method shall be used with a heating and cooling rate of 5°C/min up to a maximum temperature of 425°C for all dilatometer tests. The specimen shall be heated twice (1^{st} & 2^{nd} cycles) with an adequate period between cycles to allow cooling back to < 25°C. The tests shall be conducted using titanium-gettered helium as the purge gas at a flow rate of 150 ml/min. Prior to the establishment of this purge flow rate, three cycles of evacuation and backfill shall be conducted. The nominal oxygen partial pressure of the exhaust stream from the dilatometer shall be below 10 ppm before each test is started. The graphite specimens are nominally 12.5 x 6.0 x 5.5 mm.

The system calibration (correction setting) runs for the DIL 402 CD dilatometer shall be made using Crystallox (fine grained alumina) rods of the same nominal 12.5 mm length as the graphite specimens. Reference values from the ASTM E228 standard are used for the Crystallox for the software-based correction calculations.

The accuracy of the system shall be checked by running tungsten rods before the series of graphite specimens. Stored Tungsten values from the NBS (NIST) SRM 737 certificate were used for comparison. The measured mean CTE results are compared to those calculated from the SRM certificate values. Measured values should be within ±10%. Tungsten was chosen for this evaluation because its mean CTE is very close to that of the graphite being tested.

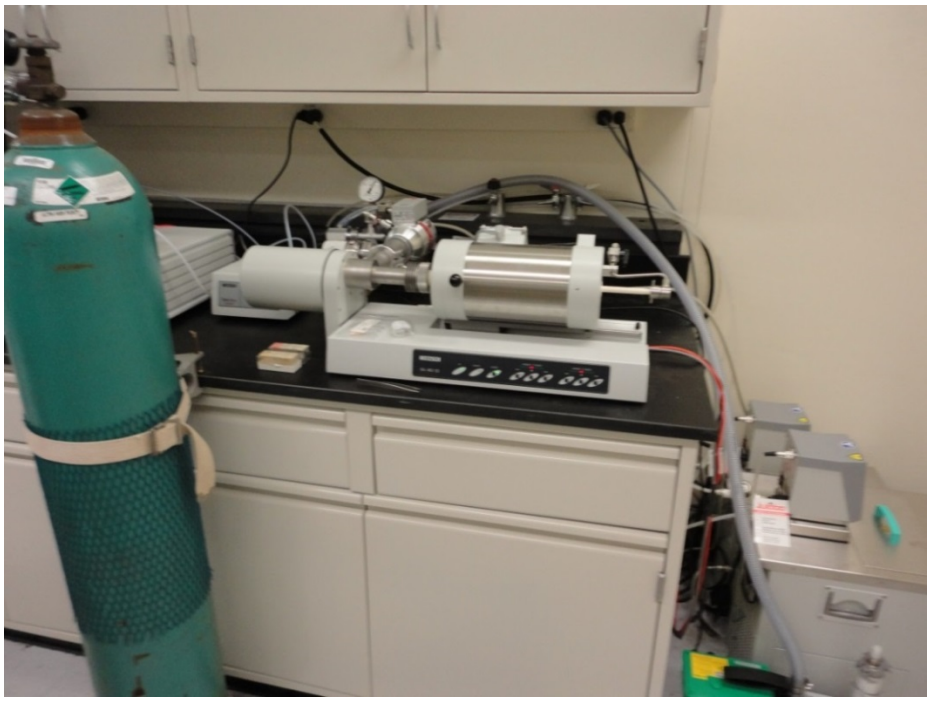

Figure 6 Netzsch 402 CD dilatometer

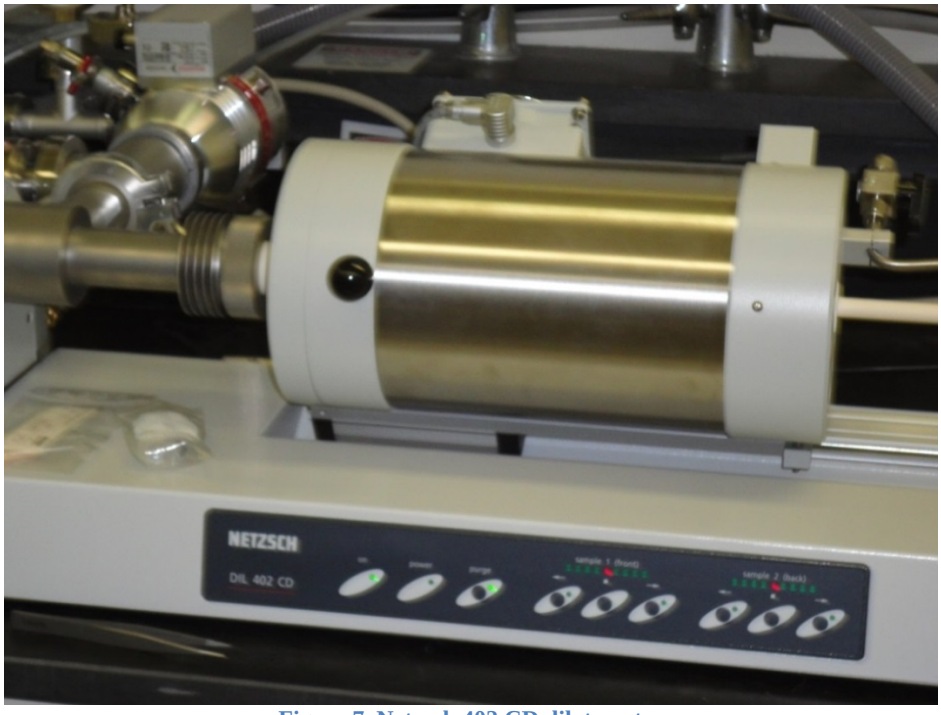

Figure 7 Netzsch 402 CD dilatometer

The mean linear coefficient of thermal expansion is calculated using the change in length of the specimen with increase in temperature. The following equations are used:

At any temperature T_1

$$\left(\frac{\Delta L}{L_0}\right)_c = \left(\frac{\Delta L}{L_0}\right)_m + A \quad (1)$$

Where

$$A = \left(\frac{\Delta L}{L_0}\right)_{cr} - \left(\frac{\Delta L}{L_0}\right)_{mr} \quad (2)$$

$$(\overline{\propto})_i = \frac{(\Delta L/L_0)_i}{\Delta T_i} \quad (3)$$

- ΔL change in length of specimen between temperatures T_i and T_0 [mm]
- L_0 original length of specimen at temperature T_0 [mm]
- A numerical calibration constant
- c calculated value
- cr calculated value for reference sample
- m measured expansion
- mr measured expansion of reference sample
- $(\overline{\propto})_i$ mean coefficient of thermal expansion [$\mu m.m^{-1}.°C^{-1}$ or $°C^{-1}$]
- ΔT_i temperature difference between T_1 and T_0 [°C]

Equation 1 is used to determine the linear thermal expansion of the test specimen, taking into consideration the expansion of the push rod material (note, push rod and holder are Alumina). Equation 3 is used to calculate the mean coefficient of thermal expansion.

5.2.6 Electrical resistivity

The test setup to be used employs a Keithley 2400 Source Meter (current supply), and a Keithley 2182 Nano-voltmeter. Multiple resistance measurements are made on 4 faces of each specimen to determine an average value. The four faces that are subject to measurement are the un-loaded faces of the stressed specimens and the corresponding faces of the reference specimens. Measurements will be made with both forward and reverse current direction on each graphite surface and with two currents, 50 and 100 MA. The specimen in inverted and the measurements repeated on the same face yielding a total of 8 measurements per face [5], 32 measurements per specimen. The resistance probe head is kept in the same position (not raised) between measurements for reversal of the polarity. It is raised when the specimen is rotated 180° on the same face or when the specimen is rotated to measure the next face. Supplemental instructions are provided to assist the test technician [6].

The experimental apparatus is illustrated in Figs. 8 and 9.

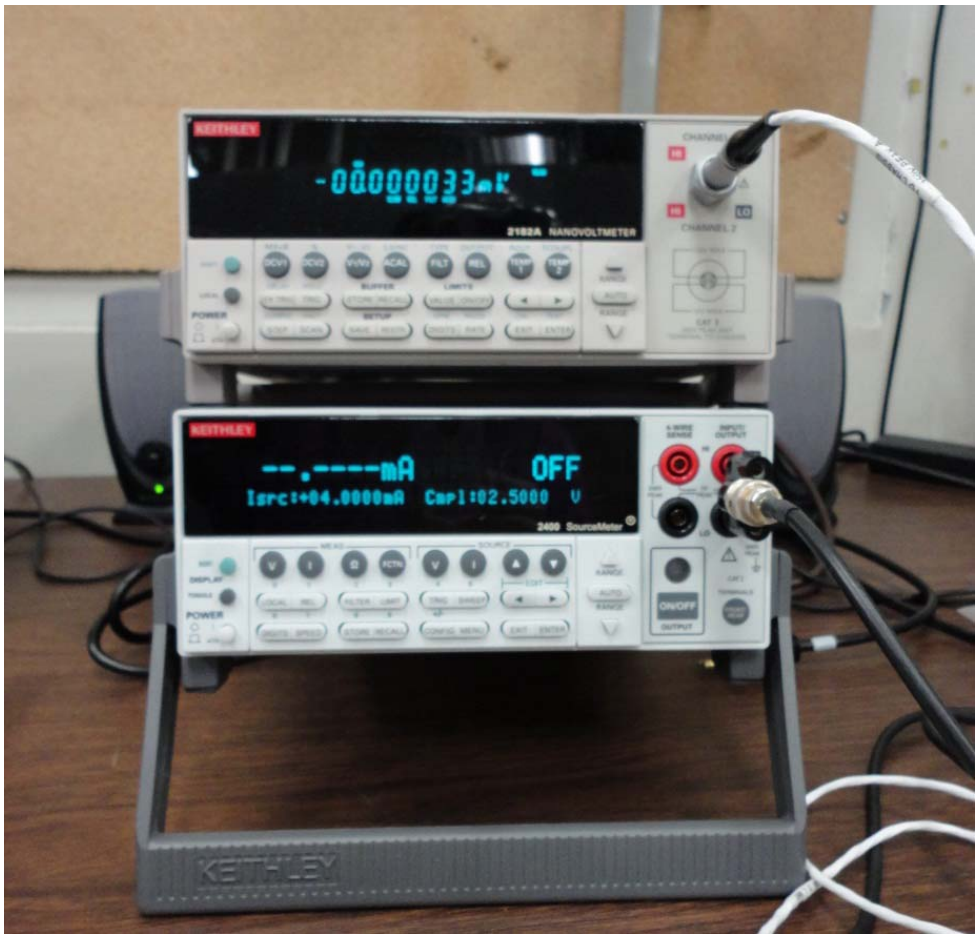

Figure 8 Electrical resistivity apparatus

A stand-mounted 4 point - probe designed and supplied by Jandel Engineering Limited (UK) shall be employed. The probe has 1.59 mm probe spacing's and the head is spring loaded to provide a constant probe application force. The probe will be coupled to our existing constant current source and nano-voltmeter. The probe is illustrated in Fig. 9.

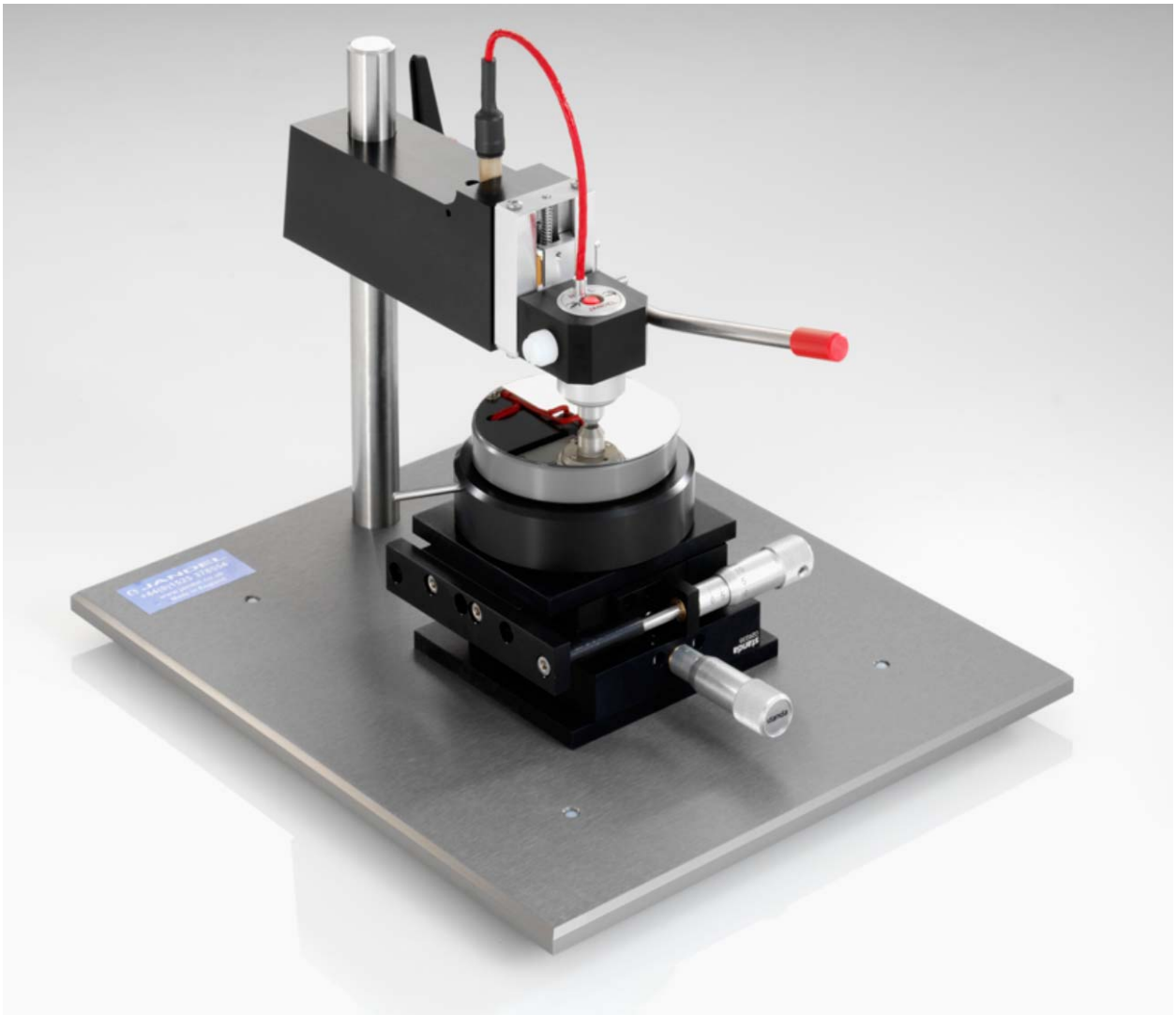

Figure 9 Jandel multi height 4-piont probe and test stand

ER measurements will be made initially with the probe in the specimen length on the face **opposite** the LASER dot and number marking in a central location (opposite LASER engraved number). Subsequently all four sides are tested. The probe details are as follows: 1.591 mm tip spacing, tungsten carbide probe tips, 200 μm tip radii, 30 g tip/spring load (30-60g range). The exact driver current setting will be established through trial and error before being fixed. The final value will be the mean of multiple readings (reversed current and rotated specimen) as per the ASTM procedure [5].

A correction factor based on two ratios is required. The first is based upon the distance between the edge of the specimen and the outer probe (this referred to as the "plane size" correction and accounts for the test plane not being an infinite plane. The second correction is for specimen thickness and again accounts for a specimen that is of finite thickness and cannot be considered "infinitely thick". Two aggregate correction factors are currently used, one calculated in

consultation with JANDEL (*F*=0.98505), and the other supplied by EDF and calculated for the equation given by Yamashita [7] (F=0.9929).

The following relationship gives the volume resistivity, ρ:

$$\rho = F2\pi s\left(\frac{V}{I}\right) \quad (4)$$

Where F = aggregate correction factor
S = probe spacing (0.159 cm)
V = Measured voltage drop from center probes (Volts)
I = Driver current from outer probes (Amps)

The value of the correction factor is taken as F= 0.9929 here.

5.2.7 Dynamic Young's Modulus (time-of-flight)

Young's modulus (DYM) is determined ultrasonically in accordance with C769 "Standard Test Method for Sonic Velocity in Manufactured Carbon and Graphite Materials for use in obtaining Young's Modulus" [7]. Supplemental instructions are provided to assist the test technician [8].

Young's modulus (E) is calculated from the velocity of a longitudinal sound wave. The velocity is calculated from measurements of the time of flight of a sound wave over a known specimen length. Three replicate measurements of time of flight are taken. The experimental apparatus is shown in Fig. 9. Two sets of Olympus ultrasonic probes are available. They have frequencies of 1 MHz and 2.25 MHz. The probes are 12.7 mm (½ inch) diameter. The probes are faced with multiple layers of replaceable soft rubber membranes so that they may be used without a couplant. Since the distance between the probe piezo-crystals is not zero, a face-thickness time correction can be expected and must be established for these probes.

The experimental apparatus (Fig. 10) consists of a Panametrics square wave pulser/receiver model 5077PD, S/N 01133702 integrated with a National Instruments Dual Trace Oscilloscope Card, Model PXI-5122, which is read with Lab View software.

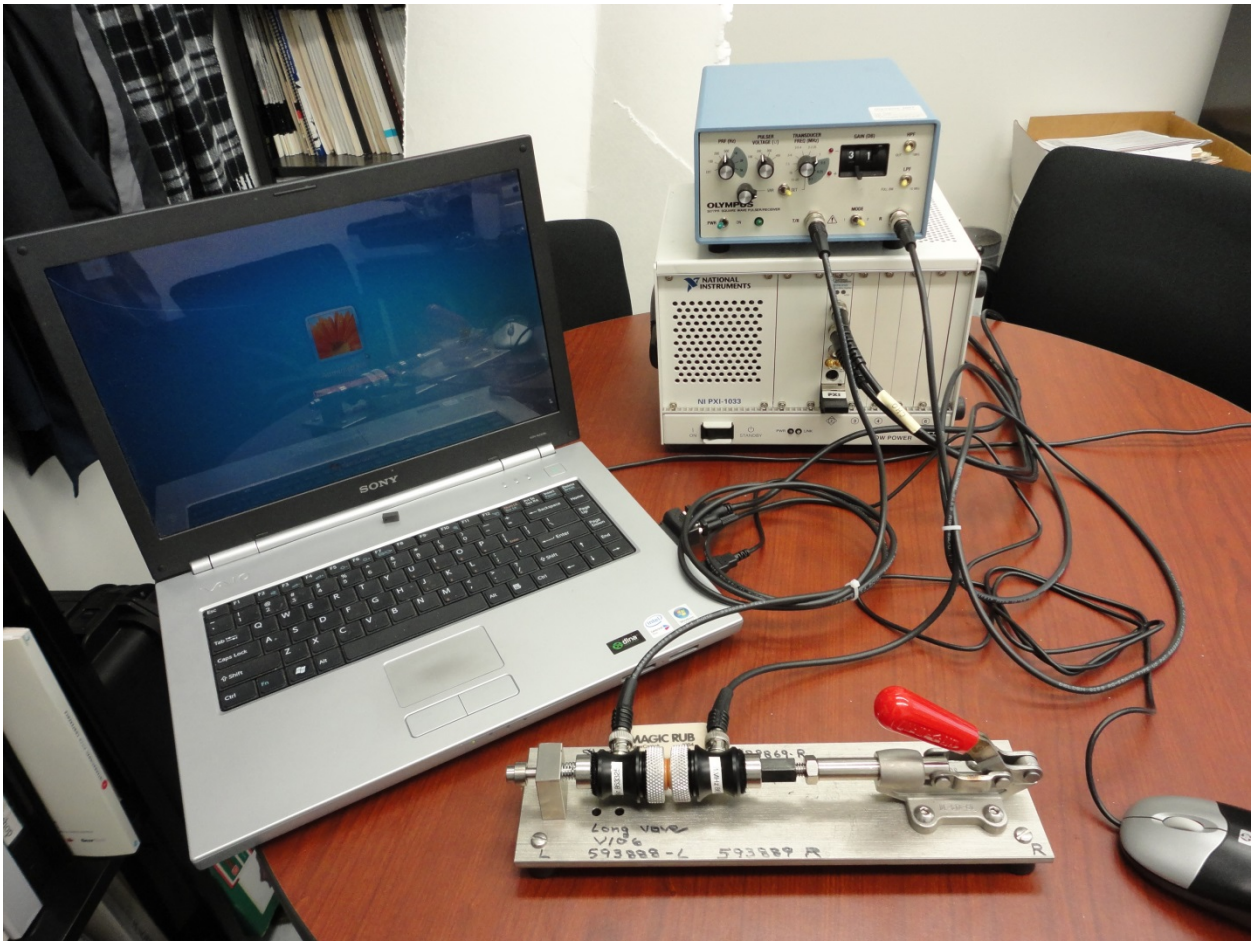

Figure 10 Experimental apparatus for the determination of time of flight and hence E (DYM)

The approximate Young's modulus (DYM) is determined from measurements of sonic velocity and calculated from equation 5 or 6:

ATSM C769 gives the approximate Young's modulus as

$$E = \rho \cdot v^2 \tag{5}$$

where E = Young's Modulus (Pa)
ρ = specimen bulk density (kg/m^3)
v = Longitudinal sonic velocity in the specimen (m/s)

However, Young's Modulus may be more precisely calculated for anisotropic materials from

$$E = \rho \cdot v^2 \cdot [(1+\mu)(1-2\mu)/(1-\mu)] \tag{6}$$

where E = Young's Modulus (Pa)
ρ = specimen bulk density (kg/m^3)
v = Longitudinal sonic velocity in the specimen (m/s), and
μ = Poisson's ratio.

Since Poisson's ratio is unknown for each of these materials a value of 0.2 is assumed, which corresponds to a correction factor in eq. 6 of 0.9 [7]

The specimen size should be sufficiently large compared to the wavelength of sound in the material. ASTM C769 suggests at least a factor of two and ideally a factor of five greater than the wavelength of sound in the material under test.

The wavelength (m) is given by

$$\lambda = c/f \tag{7}$$

where c is the velocity of sound in graphite (~2600 m/s) and f is the frequency of the sound wave. Thus a 1 MHz probe will produce a sound wave with a wavelength of 2.6 mm. In this instance the smallest specimen dimension is 5.5 mm. Thus we just exceed the recommended smallest specimen dimension and this method is considered applicable to the specimen geometry used here. Graphite's sound velocity increases on irradiation. A 60% increase in velocity, c, would thus result in a similar increase in wavelength. The specimen size guidance would thus be contravened. Depending upon the irradiated specimen sonic attenuation we should consider increasing the probe frequency to 2.25 MHz for our irradiated specimens.

Consequently, measurements shall be made at this stage at both probe frequencies (1 and 2.25 MHz).

The exact test protocol will be established when the new probes are available for experimentation, including determination of the probe face thickness correction.

Additionally, agreement was reached as to the time-of-flight measurement point on the sound wave peak signal during technical discussions during September 2013. The Pre-IE data taken prior to that date shall be reevaluated (archived digital data) since the zero point to be used was changed during discussions.

6 X-RAY DIFFRACTION STUDIES

Initially, pairs P1 and P2 shall be submitted to XRD and XRD-pole figure examination prior to the application of a pre-stress. Specimens shall be oriented with the "DOT" up and to the right during testing. Additionally, each creep and control specimen (P1 – P4) shall be subjected to XRD and XRD-pole figure examination after the pre-stress and prior to each irradiation. We shall determine the crystallographic c- and a-spacing and the crystal coherence length l_a and l_c. We have partly completed our XRD trial. A standard is being run to determine the constant in the Scherer equation used to determine l_a and l_c. Pole figures suggested slight anisotropy and have been repeated post ultrasonic cleaning.

7 X-RAY TOMOGRAPHY

Initially, specimen pairs P1 and P2 shall be submitted to full tomographic analysis at standard resolution and partial volume scanning at maximum resolution prior to the application of the pre-stress. Creep and control specimens (P1 – P4) will be subjected to full tomographic analysis at standard resolution and partial volume scanning at maximum resolution after pre-stressing and prior to irradiation. Tomography trials have been completed on spare specimen 5M-94.

8 GRAPHITE POST-IRRADIATION EXAMINATION

Post-irradiation examination (PIE) will follow the workflow chart in Figure 11. Following irradiation in HFIR the EDF Energy creep rabbits will cool in the ponds followed by shipment to the hot cells. The rabbit capsules are disassembled in the cells and the specimens and temperature monitors recovered and sent to the Low Activation Material Development & Analysis (LAMDA) Laboratory. The specimens will undergo post irradiation examination (PIE) which will consist of dimensional measurements, mass, and hence density [3], time-of-flight (hence DYM) [8] measurement, electrical resistivity (ER) [5], and determination of the coefficient of thermal expansion [4]. Additional microstructural characterization shall include XRD and tomography. Additional PIE details are provided below.

Prior to full disassembly the capsule/bellows are compression tested in-cell to determine the exact bellows force exerted on the specimen. Bellows testing will occur in hot cell #6 using the existing load frame with a 1 kN load cell fitted. Bellows testing will occur at room temperature only so as to avoid the potential for (i) SiC annealing, and (ii) graphite specimen oxidation.

The specimen irradiation temperature will be determined from temperature monitors encapsulated within the specimens. Each capsule contains at least six monitors, each associated with the specimens. Additionally a SiC monitor shall be placed inside the bellows. Details of the SiC monitors sizes are given in Table 6.

The irradiation temperature will be estimated by isochronal annealing followed by thermal diffusivity determination at 100°C. Certain SiC monitors will be tested by thermal expansion/instantaneous CTE behavior. A plot of property as a function of annealing temperature should display a marked change in the property when the annealing temperature exceeds the last irradiation temperature. Detail of the SiC thermometry is given in Section 9.

The experimental data will be reported in a final PIE report after each irradiation/examination cycle.

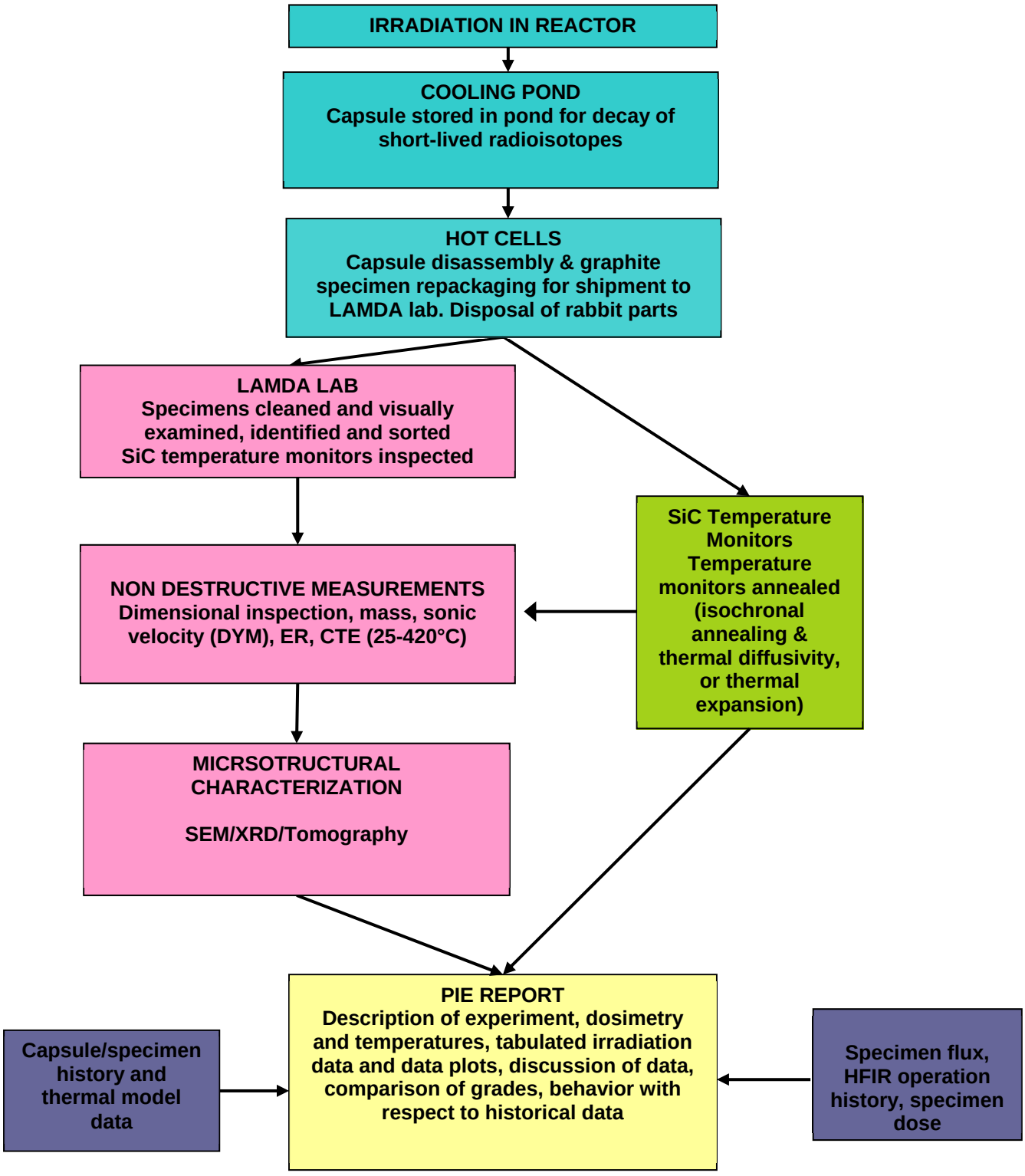

Figure 11 Post-irradiation data process flow chart for EDF Energy graphite specimens

8.1 PIE Test order

The capsule loading plan and specimen ID's are repeated in Table 7.

Table 7 Capsule Loading Plan and Specimen identities

Capsule No	SPEC PAIR	Spec ID		LOADED	Purpose
EDF01	P1a	5M68	U1	YES	Creep Strain
	P1b	5M76	U5	NO	Creep Strain
EDF02	P2a	5M70	U2	YES	Creep Strain
	P2b	5M78	U6	NO	Creep Strain
EDF03	P3a	5M72	U3	YES	Creep Strain
	P3b	5M80	SI	NO	Creep Strain
EDF04	P4a	5M74	U4	YES	Creep Strain
	P4b	5M82	S2	NO	Creep Strain
EDF05	P5a	5M84	S3	YES	Calibration (thermal)
	P5b	5M86	S4	NO	Calibration (thermal)

The order of PIE activities and testing shall be (check with LAMDA work package):

1. Cleaning ultrasonically in ethanol to remove any radiological contaminants
2. SiC monitor interrogation
3. Photography
4. Dimensions. Mass, density
5. X-ray Tomography (P1a/b and P3a/b)
6. Pole Figures (P1a/b and P3a/b)
7. XRD (all 8 specimens)
8. E_{dym} (TOF)
9. ER
10. CTE (25-420°C), 8 specimens both orientations (12.5 m and 6mm)

8.1.1 SiC Monitors

The SiC monitors will be interrogated to establish T_{irr}. Section 9 contains further details of the SiC monitor testing. Once T_{irr} has been determined the upper temperature for thermal expansion can be established. However, we assume T_{irr} is ~420°C and thus the specimen thermal expansion testing shall be over the temperature range RT-420°C.

8.1.2 Photography

The irradiated specimens shall be submitted for digital photography using the Keyence microscope. Plan view images of all 6 faces of each specimen are recorded.

8.1.3 Dimensional Measurements

The graphite specimen shall be subjected to full dimensional measurement using the same protocol as in the case of Pre-IE. (Section 5.2.4 refers).

8.1.4 Mass

The graphite specimen shall be subjected to Mass measurement using the same protocol as in the case of Pre-IE. (Section 5.2.4 refers). PIE Density may thus be calculated.

8.1.5 Electrical Resistivity

The graphite specimen shall be subjected to ER measurement using the same protocol as in the case of Pre-IE. (Section 5.2.6 refers).

8.1.6 Dynamic Young's Modulus (time-of-flight)

We shall calculate E_{dym} from the ultrasonic signal TOF. The same protocol will be followed as in the case of Pre-IE (section 5.2.7) except that measurements of TOF will only be conducted at 2.25 MHz frequency.

8.1.7 X-Ray Tomography

X-ray tomography shall be conducted on four specimens P1a, P1b, P3a and P3b. The specimens shall be submitted to full tomographic analysis at standard resolution and partial volume scanning at maximum resolution. The same experimental protocol as used for pre-IE shall be used for PIE (Section 7 refers).

8.1.8 X-Ray Diffraction (XRD)

The XRD parameters d, a, l_c, l_a shall be determined from 20°-120° Cukα scans performed on all 8 specimens, i.e., P1a/b, P2a/b, P3a/b and P4a/b.

Pole figures will be obtained from 20°-100° Cokα scans on 4 specimens only, i.e., P1a/b and P3a/b only.

The specimens shall be oriented with the 'DOT' up and to the right.

8.1.9 Coefficient of Thermal Expansion

The specimen post irradiation CTE shall be determined using the same experimental protocol as used for the Pre-IE (section 5.2.5 refers. The upper temperature for the expansion measurements shall be the irradiation temperature (assumed to be 420°C).

CTE shall be determined for all 8 specimens (i.e., P1a/b, P2a/b, P3a/b and P4a/b). Each specimen shall be tested in both orientations (12.5 mm and 6 mm dimension).

9 SiC THERMOMETRY

A calibration rabbit will be used during segment 1 irradiation only to determine the temperature variations in the rabbit capsule, i.e., with and without a central hole in the specimen. The sizes and locations of the various SiC temperature monitors to be used are shown in Table 8 and the monitors are illustrated in Figure 12.

The temperature monitors shall be read using the change in thermal diffusivity as a function of isochronal annealing temperature, or by continuous thermal expansion in the case of the bellows SiC and calibration rabbit SiCs.

Table 8 EDF Energy Creep capsule temperature monitors

SiC Geometry	SiC Dimensions (mm)	Number per capsule	SiC Location	Thermometry test method
Bars	0.5x1x12.5	4	2 per along graphite specimen flank	Expansion (optional)
Wafers	1x5.5x6	2	1 per at end of graphite specimens	Thermal diffusivity
Rods	1 dia. x 12	2	Inside specimen (central hole) CALIBRATION RABBIT ONLY (Irradiation Segment 1 only)	Expansion
Rods	3.1 dia. X 11	1	Inside bellows	Expansion

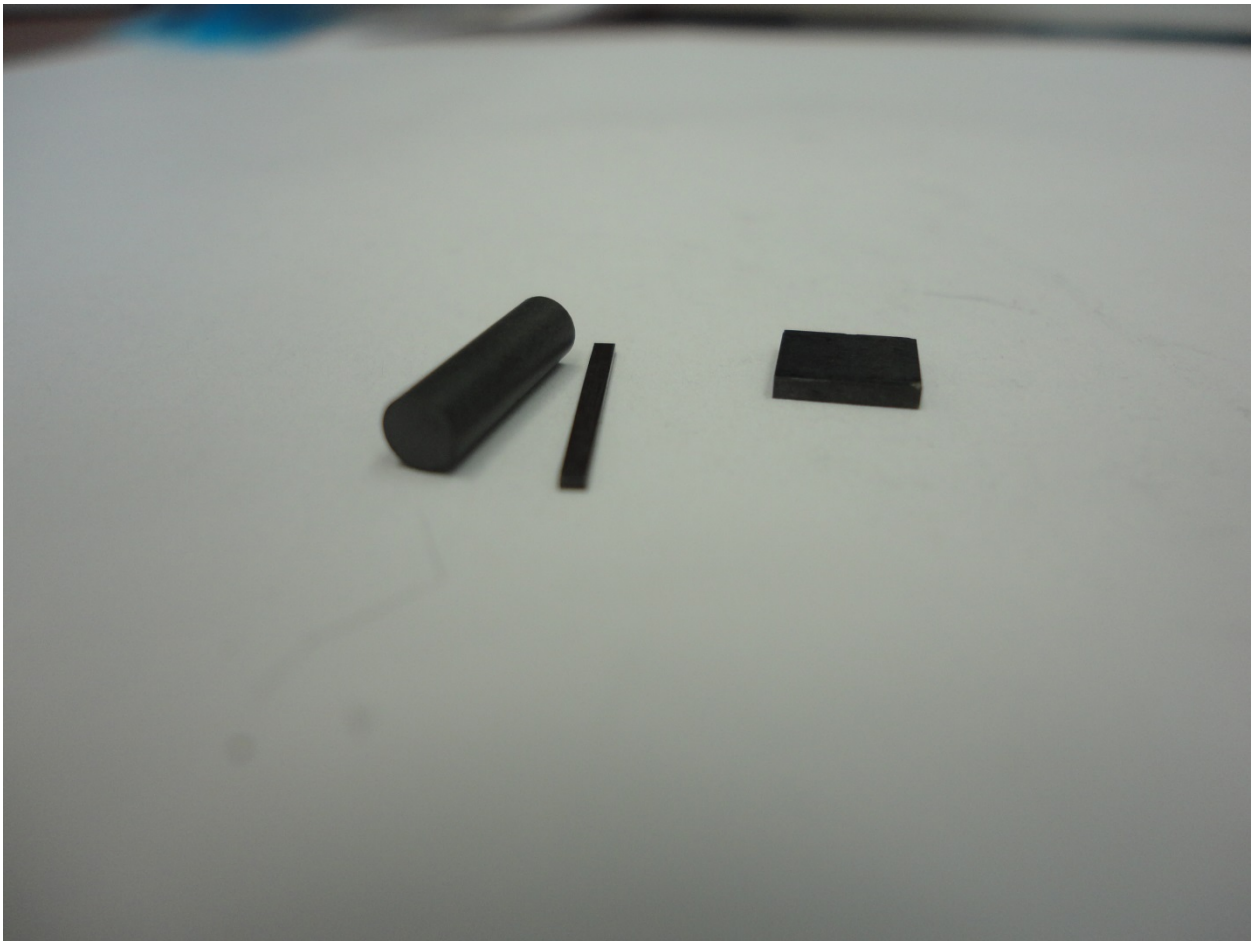

Figure 12 SiC monitors inserted in the Creep Rabbit Capsules

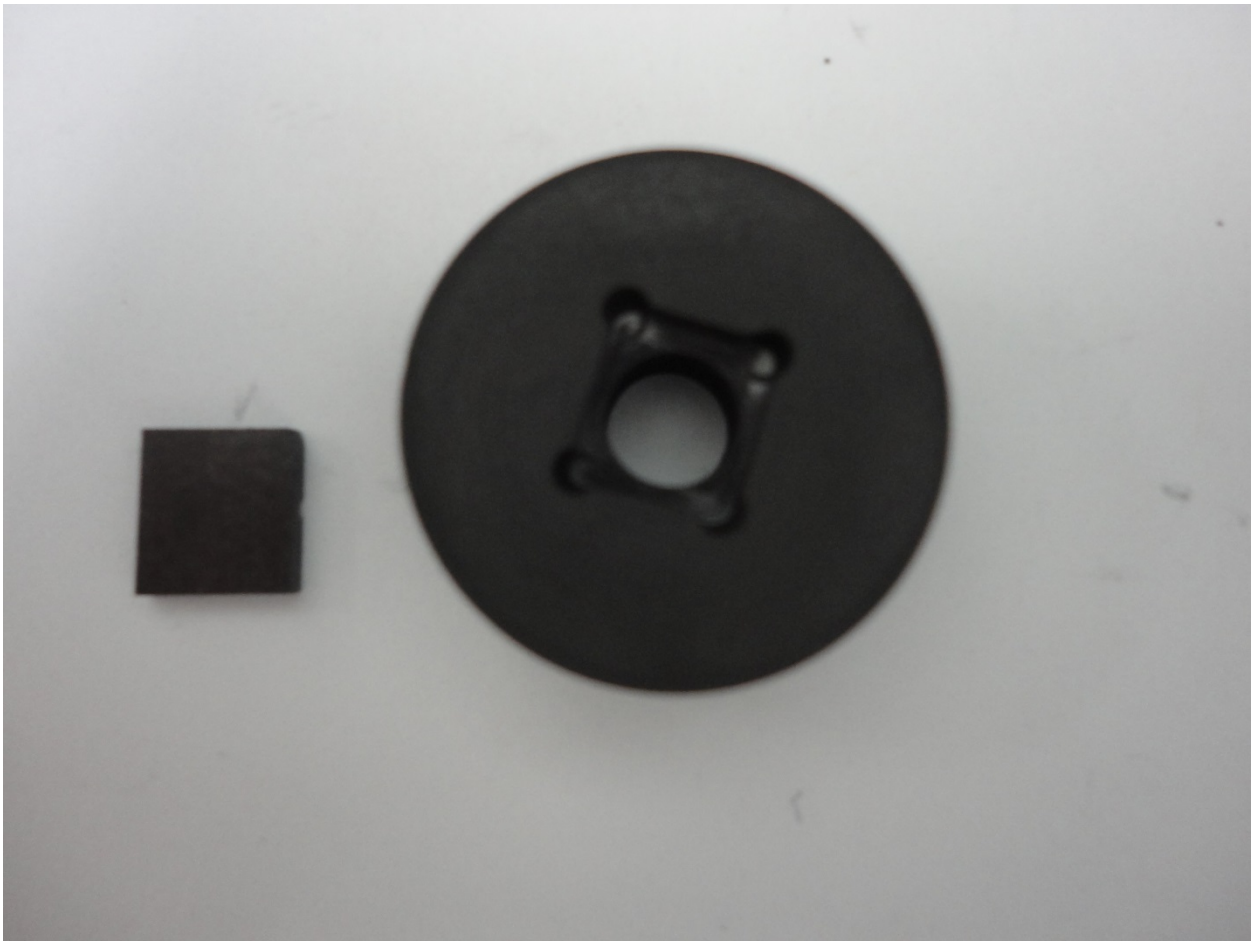

Figure 13 SiC wafer and thermal diffusivity fixture

The SiC wafers will be thermally annealed at set temperatures (Table 9) during PIE at a heating rate is 2°C/min. The wafers are held at the annealing temperature for sufficient time to take 2 diffusivity measurements. The wafers are then cooled to 100°C and the thermal diffusivity (at 100°C) determined. The diffusivity value reported shall be the mean of four diffusivity measurements (LASER pulses) at 100°C. The specimens are cooled at 20°C/min to 100°C. Specimen holders have been machined for the SiC thermal diffusivity testing (Figure 13). The desired annealing temperature/hold time schedule for the annealing of the SiCs is given in Table 9.

The bellows SiC and the calibration rabbit specimen centerline SiCs (if applicable) shall be continuously annealed while simultaneously measuring the thermal expansion. Since the SiCs are relatively short, good resolution is required and measurements shall be performed on the Netzsch dilatometer. The thermal expansion and instantaneous CTE shall be determined for the bellows and calibration SiCs over the temperature range 100°C to 700°C. The heating and cooling behavior shall be recorded. A heating rate of 1°C/min and a cooling rate of 2°C/min shall be employed. The SiC approximate dimensions are given in Table 8.

Table 9 Heating schedule for EDF rabbit capsule SiC temperature monitors diffusivity measurements

Annealing Temperature (°C)	Actions
100	Heat to annealing temp, hold, cool to 100°C, measure thermal diffusivity
150	Heat to annealing temp, hold, cool to 100°C, measure thermal diffusivity
200	Heat to annealing temp, hold, cool to 100°C, measure thermal diffusivity
250	Heat to annealing temp, hold, cool to 100°C, measure thermal diffusivity
280	Heat to annealing temp, hold, cool to 100°C, measure thermal diffusivity
290	Heat to annealing temp, hold, cool to 100°C, measure thermal diffusivity
300	Heat to annealing temp, hold, cool to 100°C, measure thermal diffusivity
310	Heat to annealing temp, hold, cool to 100°C, measure thermal diffusivity
320	Heat to annealing temp, hold, cool to 100°C, measure thermal diffusivity
330	Heat to annealing temp, hold, cool to 100°C, measure thermal diffusivity
340	Heat to annealing temp, hold, cool to 100°C, measure thermal diffusivity
350	Heat to annealing temp, hold, cool to 100°C, measure thermal diffusivity
360	Heat to annealing temp, hold, cool to 100°C, measure thermal diffusivity
370	Heat to annealing temp, hold, cool to 100°C, measure thermal diffusivity
380	Heat to annealing temp, hold, cool to 100°C, measure thermal diffusivity
390	Heat to annealing temp, hold, cool to 100°C, measure thermal diffusivity
400	Heat to annealing temp, hold, cool to 100°C, measure thermal diffusivity
410	Heat to annealing temp, hold, cool to 100°C, measure thermal diffusivity
420	Heat to annealing temp, hold, cool to 100°C, measure thermal diffusivity
430	Heat to annealing temp, hold, cool to 100°C, measure thermal diffusivity
440	Heat to annealing temp, hold, cool to 100°C, measure thermal diffusivity
450	Heat to annealing temp, hold, cool to 100°C, measure thermal diffusivity
460	Heat to annealing temp, hold, cool to 100°C, measure thermal diffusivity
475	Heat to annealing temp, hold, cool to 100°C, measure thermal diffusivity
500	Heat to annealing temp, hold, cool to 100°C, measure thermal diffusivity
600	Heat to annealing temp, hold, cool to 100°C, measure thermal diffusivity
700	Heat to annealing temp, hold, cool to 100°C, measure thermal diffusivity

A summary of the required SiC//Graphite specimen testing is given in Table 10.

Table 10 EDF specimens/SiCs PIE summary

EDF PIE TESTING																	
		Spec ID				Tasks to be performed in left to right order of testing. Note SiC testing most be completed before graphite specimen CTE is performed											
Capsule No	SPEC PAIR	EDF number	Number engraved on specimen	LOADED	Purpose	1 SiC Thermography	2 A Cleaning	2B SiC SORTING	3 Digital Photography	4 DIMENSIONS	5 MASS	6 XRD paramaters (Cu kα)	7 Pole Figures (Cokα)	8 Tomography	9 E_{dyn}(TOF)	10 ER	11 Thermal Expansion
						Wally	**Patty**	**Ashli/Tim**	**Stephanie**	**Ashli**	**Ashli**	**Robbie/Roza**	**Robbie/Roza**	**Michael/ Stephanie**	**Tim/Ashli**	**Tim/Ashli**	**Wally**
EDF01	P1a	5M68	U1	YES	Creep Strain	O	O	O	O	O	O	O	O	O	O	O	O
	P1b	5M76	U5	NO	Creep Strain		O	O	O	O	O	O	O	O	O	O	O
EDF02	P2a	5M70	U2	YES	Creep Strain	O	O	O	O	O	O	O	X	X	O	O	O
	P2b	5M78	U6	NO	Creep Strain		O	O	O	O	O	O	X	X	O	O	O
EDF03	P3a	5M72	U3	YES	Creep Strain	O	O	O	O	O	O	O	O	O	O	O	O
	P3b	5M80	SI	NO	Creep Strain		O	O	O	O	O	O	O	O	O	O	O
EDF04	P4a	5M74	U4	YES	Creep Strain	O	O	O	O	O	O	O	X	X	O	O	O
	P4b	5M82	S2	NO	Creep Strain		O	O	O	O	O	O	X	X	O	O	O
EDF05	P5a	5M84	S3	YES	Calibration (thermal)	O	SiC thermography: thermal diffusivty (Diff @ 100C): Bellows & Cal SiC by Expansion										
	P5b	5M86	S4	NO	Calibration (thermal)												

	NOTE									**Task 6**	**XRD Parameters c,a,lc and la, Cukα scan 20°to 100° min**						
	Task 1	**perform first to determine irradiation temerature ahead of graphite CTE**								**Task 7**	**Cokα scan 20° to 90° scan**						
		Tim has graphite holder insetrs for the SiC wafers								**Task 8**	**Michael and Stephanie**						
	Task2A	**Its OK to U/S clean in a flask of hot water or ethanol/alcohol**								**Task 9**	**2.25 MHz only Tim/Ashli**						
	Task 2B	**Sort the SiC's into type and test type and Operator**								**Task 10**	**Using JANDEL**						
	Task 3	**Ashli/Stephanie with Tim**								**Task 11**	**12.5 and 6 mm dimension orientations, perhaps a slower heating rate ? Do nothing untill Tirr established**						
	Task 4 & 5	**Straightforward. Existing technique**															

10 SPECIMEN RE-IRRADIATION

Following PIE examination (Table 10) the specimens shall be returned to LAMDA lab for re-encapsulation in new creep rabbits. The target neutron fluence for the second irradiation segment is given in Table 3. All anticipated permissions/procedures are in place for re-irradiation/re-encapsulation of the specimens in LAMDA

Capsule welds performed with irradiated samples in capsules represent a departure from our normal operating procedures since we have not recently re-irradiated specimens. Each rabbit capsule has three weld procedures performed in the welding labs in building 4508 after the inner assembly is complete and pre-irradiated graphite is placed in the capsule.

The required welds are:

1. A LASER weld to seal the bellows. This is conducted under pressure so the bellows is sealed with the appropriate internal pressure. A leak test is required after this weld. Leaking welds shall be repaired. We shall bench test the bellows at this point to assure the appropriate force is being developed and the load train articulates freely.
2. The rabbit assembly has an e-beam weld performed to join the end cap to the outer housing. This weld must be performed by a qualified/certified welder. Post weld leak testing must be performed.
3. The final weld procedure is a Tungsten-Inert- Gas (TIG) weld, which seals the capsule atmosphere. The TIG weld is performed in a glove box with the desired rabbit fill-gas and the glove box pumped down to high vacuum prior to being back-filled with the desired inert gas. This weld must be performed by a qualified/certified welder. Post weld leak testing must be performed.

The appropriate procedures for the welding operations involving irradiated specimens have been developed. A temporary c-zone will be established around the weld equipment. The weld operators are appropriately radiation worker trained.

11 QUALITY ASSURANCE

The activities described in this plan will be conducted in accordance with the applicable requirements of the ANSI/ISO/ASQ(E) Q9001-2008 standard entitled Quality Management Systems - Requirements, and the ASME NQA-1-2008 standard entitled Quality Assurance Requirements for Nuclear Facility Applications. Project and activity-specific information concerning ORNL's application of the each standard's requirements is provided in Document #QAPORNL-EDF-01 entitled Quality Assurance and Safety Management Plan for the Nuclear Science and Technology Activities Funded by EDF Nuclear Generation Ltd. and Conducted by or for the Oak Ridge National Laboratory.

12 REFERENCES

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