# Simultaneous heating and compression of irradiated graphite during synchrotron microtomographic imaging



2017 J. Phys.: Conf. Ser. 849 012021

(http://iopscience.iop.org/1742-6596/849/1/012021)

IOP Conf. Series: Journal of Physics: Conf. Series **849** (2017) 012021 doi:10.1088/1742-6596/849/1/012021

# Simultaneous heating and compression of irradiated graphite during synchrotron microtomographic imaging

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**Abstract**. Nuclear graphite is used as a neutron moderator in fission power stations. To investigate the microstructural changes that occur during such use, it has been studied for the first time by X-ray microtomography with in situ heating and compression. This experiment was the first to involve simultaneous heating and mechanical loading of radioactive samples at Diamond Light Source, and represented the first study of radioactive materials at the Diamond-Manchester Imaging Branchline I13-2. Engineering methods and safety protocols were developed to ensure the safe containment of irradiated graphite as it was simultaneously compressed to 450N in a Deben 10kN Open-Frame Rig and heated to 300°C with dual focused infrared lamps. Central to safe containment was a double containment vessel which prevented escape of airborne particulates while enabling compression via a moveable ram and the transmission of infrared light to the sample. Temperature measurements were made in situ via thermocouple readout. During heating and compression, samples were simultaneously rotated and imaged with polychromatic X-rays. The resulting microtomograms are being studied via digital volume correlation to provide insights into how thermal expansion coefficients and microstructure are affected by irradiation history, load and heat. Such information will be key to improving the accuracy of graphite degradation models which inform safety margins at power stations.

## 1. Introduction

The Diamond-Manchester Imaging Branchline I13-2 at Diamond Light Source (DLS) provides high-flux, partially-coherent 5-35keV X-rays which are used primarily for tomographic imaging experiments. Such experiments are often highly bespoke and involve complex manipulation of sample environments.

Nuclear graphite bricks function as both moderators and structural components in fission power stations. Over time, radiation and oxidation degrade the graphite, leading to mass loss, distortion and eventual cracking. When distortion and cracking become too severe, it can become difficult to insert and remove control rods, thereby affecting the risk profile of power station operation. Owing to uncertainties about the mechanisms and impacts of graphite degradation, UK power stations operate with very conservative safety margins. Computational models of degradation (eg. EDF Energy's FEAT-DIFFUSE6 and [1]) inform these safety margins; such models rely upon data relating to coefficients of thermal expansion (CTEs) and the manner in which these vary as a function of temperature and pressure. To generate CTE data and investigate how microstructure is also affected by irradiation history, load and heat, we performed X-ray microtomography on nuclear graphite in a custom sample environment and are now analysing the results via digital volume correlation (DVC) [2]. Development and use of the sample environment posed a number of technical challenges relating to the safe containment of samples undergoing simultaneous heating and compression.

## 2. Experimental design

Cylindrical samples, of 5mm diameter and 6mm height, were trepanned from both unirradiated graphite bricks and irradiated bricks at a UK power station. Samples were pretreated offsite to boil-off radioactive gases.

Samples were mounted in double containment vessels which were produced for the experiment as a collaborative design between University of Manchester and Deben UK Ltd. Double containment was provided by two cylindrical glass walls which were sealed against upper and lower plates. The sample was mounted on a static platform and compressed from above by a ram. Ram movement was controlled externally, with seals maintained by bellows.

The containment vessels did not require a very high degree of air tightness, but did need to contain any airborne particulates that could be generated during the course of an experiment. Helium leak testing was conducted jointly by Deben and the DLS Vacuum Group.

Samples arrived at DLS mounted in individual containment vessels and were handled on site in accordance with DLS's internal procedures. Since Deben Open-Frame Rigs had not been used previously for synchrotron experiments and the containment vessels had not been used with these rigs before, dry runs were performed with unirradiated samples to test various elements of the setup. Vessels were mounted inside the Open-Frame Rig, which was equipped with two Optron 150W IR Spot (Optron GmbH, Germany) focused infrared lamps (figure 1). The whole vessel was free to rotate inside the rig. To prevent sample cracking and associated particulate generation, the rig had been programmed with a compression limit and the option for torsion disabled.

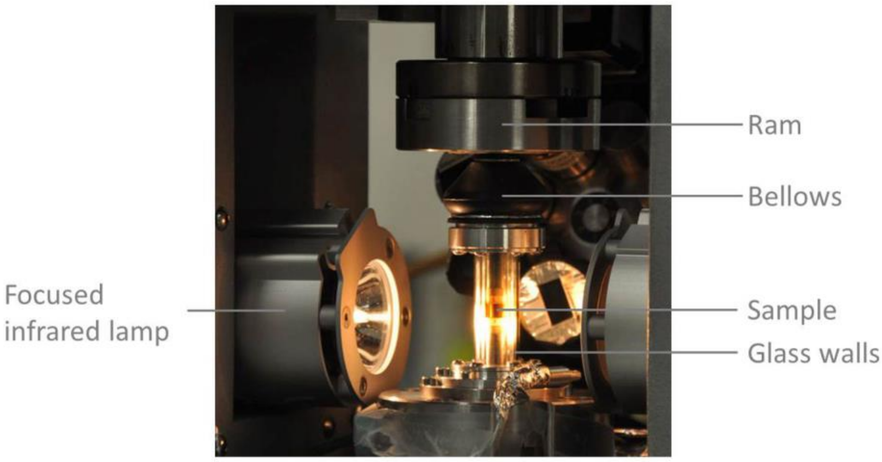


**Figure 1.** A double containment vessel in the Open-Frame Rig, with focused infrared lamps radiating orthogonally to the direction of the X-ray beam. A nuclear graphite sample can be seen undergoing compression in the vessel.

## 3. Microtomography

Samples were imaged with a partially-coherent, polychromatic 'pink' beam (5-35 keV) of parallel geometry generated by an undulator of 5mm gap. The beam was reflected from the platinum stripe of a grazing-incidence focusing mirror and filtered with 950µm pyrolytic graphite and 2mm aluminium. 2,001 projection images, each of 75ms exposure time, were acquired at equally-spaced angles over 180° of stepped rotation by a pco.edge 5.5 (PCO AG, Germany) detector. The image recorded at 180°

was compared to the image recorded at 0° to check for various experimental problems [3]. The detector was coupled to a 500µm $CdWO_4$ scintillator and a visual light microscope, providing 2.5× total magnification, a field of view of 6.7 × 5.6 mm (2560 × 2160 pixels) and an effective pixel size of 2.6µm. A propagation distance of approximately 70mm was used, providing a moderate level of in-line phase contrast.

## 4. Preliminary results

Tomographic slices of unirradiated and irradiated graphite are shown in figure 2. These images demonstrate widespread changes in microstructure caused by irradiation and radiolytic oxidation, including increased porosity and filler-particle compression. CTEs were calculated from displacements of features as measured by DVC. For the unirradiated samples, CTEs were found to increase with increasing compressive load. Other results, as well as image analysis and DVC methods, will be presented in an upcoming publication.

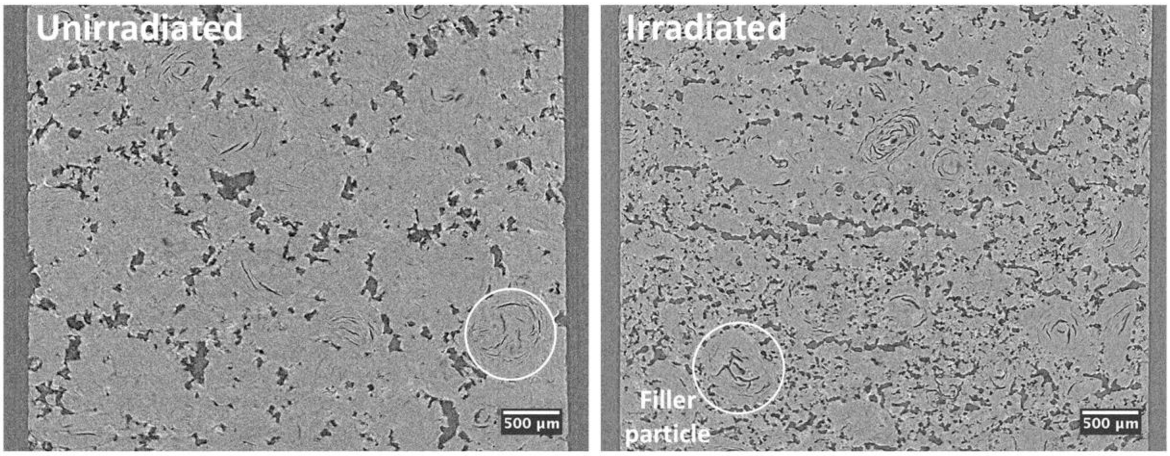


**Figure 2.** Comparison of unirradiated and irradiated graphite. Both samples had been raised to a temperature of 300°C and loaded to 450N. Note the increased porosity and compression of filler particles associated with irradiation.

## 5. Acknowledgements

We acknowledge financial support from the Innovate UK project *The Influence of Graphite Irradiation Creep on Plant Life Optimisation* (101437) and thank EDF Energy for the graphite samples. We thank DLS and the Diamond-Manchester Collaboration for beamtime at I13-2 under proposal MT12200. We would like to express our gratitude to K Wanelik and A A Wilson for helping to commission the Open-Frame Rig and integrate it into the DLS data acquisition systems, L Lea and colleagues in the DLS Vacuum Group for containment vessel testing, S Logan for technician support, and R Doull of the DLS Health Physics Group for assistance with health and safety matters.

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