Experimental report

17/02/2020

Proposal: **1-02-241**

Council: 4/2018

Title: High-resolution neutron diffraction residual stress analysis of nuclear graphite

Research area: Materials

This proposal is a new proposal

Main proposer: **Thomas ZILLHARDT**

Experimental team: Abdalrhaman MOHAMED
Thomas ZILLHARDT
Phil EARP

Local contacts: Thilo PIRLING
Sandra CABEZA

Samples: Graphite

Instrument	Requested days	Allocated days	From	To
SALSA	6	4	18/07/2019	22/07/2019

Abstract:

Nuclear graphites have a large number of defects over a very wide range of scales from nm to mm. These defects, and how they evolve with the microstructure, are important to understand the onset of sub-critical cracking and fracture of graphite components in the UK Advanced Gas Reactors. Micro-cracking affects the accommodation of strain at stress concentrations in quasi-brittle materials, like nuclear graphites, and we will perform high resolution studies of the effects of microcracking on the elastic properties and internal strains in Gilsocarbon graphite.

	Experiment title: **High-resolution neutron diffraction residual stress analysis of nuclear graphite**	**Experiment number:** 1-02-241
Beamline: ID11	**Date of experiment:** from: 18/07/2019 to: 22/07/2019	**Date of report:** Monday, 17 February 2020 *Received at ILL:*
Shifts: 12	**Local contact(s):** Thilo PIRLING, Sandra CABEZA	

Names and affiliations of applicants (* indicates experimentalists):

Thomas Zillhardt*, Department of Materials, University of Oxford

James Marrow, Department of Materials, University of Oxford

1 Introduction

The purpose of this experiment is to detect the evolution of strain and to demonstrate whether there is any residual strain upon mechanical loading and unloading and to quantify these residual stresses. Neutron scattering was used to help identify where damage might be accumulated and provide information on stress relaxation and recovery at notches. The reason for choosing the instrument Salsa at the Institut Laue-Langevin in France is the brightness of the source. As it is a nuclear reactor, the acquisition time is reduced. It takes 20 minutes on STFC ENGINX to perform a single acquisition with 4 x 4 mm collimators, compared to 2 minutes on Salsa with 2 x 2 mm collimators. It is to be noted however that the dual-bank configuration of ENGINX allows two orthogonal strain components to be collected at the same time, whereas Salsa only has one detector bank.

2 Experimental details

Due to time constraints and complications, this experiment was divided into two parts. They both used the same experimental setup at the exception of the collimators. While the first part aimed at providing acquisitions with a higher resolution using the 0.6x0.6x2mm collimator set, the second part made use of a 2x2x2mm setup in order to perform a larger number of acquisitions in the remaining time allocated.
A DIC camera was installed horizontally towards the sample and at an angle. The ideal setup for image correlation is to have the camera axis perpendicular to the speckle pattern plane. Because of the pillars of the Instron rig and the size of the collimators, this was found to be not possible for this experiment, which therefore required correction for the angle. This was done using two methods, a direct measurement of the angle between the camera axis and the perpendicular to the speckle pattern, and the grid correction. The latter consists in covering the sample with a grid of known dimensions in order to work out the image deformation resulting from the angle.

It is to be noted that due to delays and complications, detailed hereafter, a number of amendments had to be made to the original proposed experiment. An additional day of beamtime was granted by the instrument scientists in order to complete a few additional acquisitions, however this was not enough to achieve our objectives. In particular, it is crucial for our study to obtain both the radial and tangential components of strain. The layout used in the first part of the experiment meant that we could not rotate the sample table with the Instron fitted to obtain the tangential component.

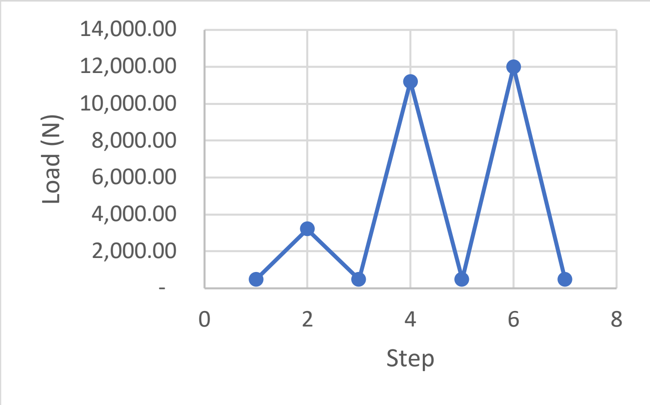

Figure 1: Loading steps for 36 points map of slit-notched specimen.

Graphite has a low Bragg angle for the neutron elastic scattering of the 002 peak and the very first challenge upon setting up was to position the detector at the right theta without the detector housing touching the containment wall. The latter angle is the one at which we obtain the largest number of counts and is also the most significant peak as it represents the interplanar spacing of the stacked hexagonal structure and gives the best information on elastic strain. We successfully managed to acquire data for the 002 peak on the radial component and proceeded to a number of loading steps (Figure 1). The map was centred around the notch of the specimens, as shown in Figure 2.

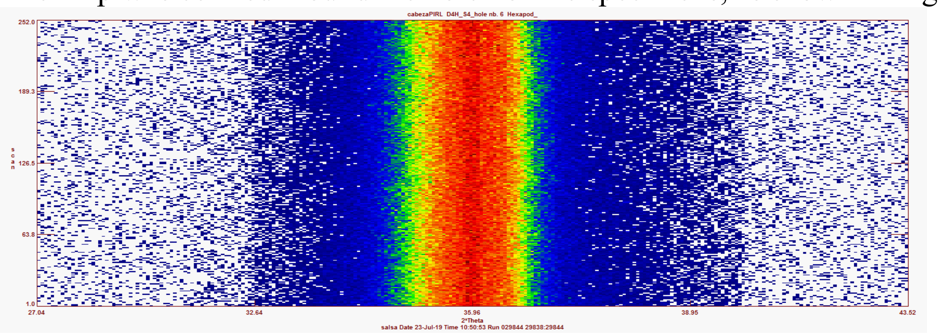

Figure 2. Raw data for 252 acquisitions of the second slit-notched sample. The ordinate axis represents scan numbers. The abscissa is the Bragg angle (2 theta); the (002) is studied at high resolution.

There are two main complications that affected the experiment. The first one was the unavailability of the Instron for mechanical loading, which went offline because of cooling failures and could not be brought back online quickly after these issues were resolved because of IT problems. This delayed the experiment by 10 hours. The second issue has motivated an application for extension as it prevented us from completing an important part of the experiment.

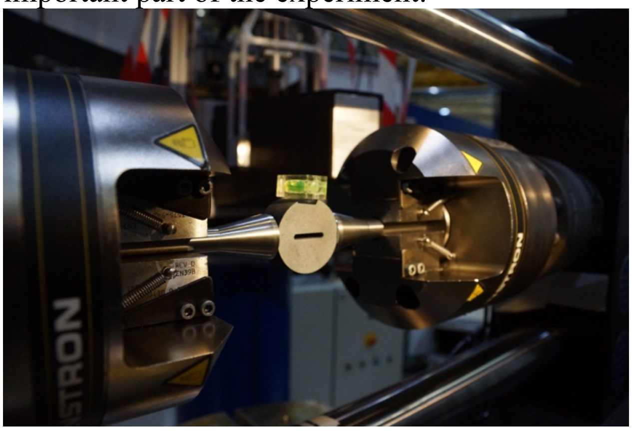

Figure 3: Experimental setup. The region of interest is around the notch, left of the slit.

The radial strain is relatively important in order to validate the experiment and observe the compressive strains close to the edges of the disc and by the notch, but we cannot make any conclusion without the tangential strains. To achieve this, we needed to rotate the sample table and the Instron loading rig by 90 degrees. We tried to perform this operation, but without a three-dimensional model of the instrument it was a complicated effort. We found out that the Instron could not be rotated with the 0.6x0.6

collimators as they are too large. This would however be possible with the 2x2x2mm collimators. An example of the setup is seen in Figure 3.

3 Results

The results are incomplete as we were only able to obtain the radial component around the notches, i.e. parallel to the loading direction. However, these preliminary results tend to show that the information obtained using SALSA can be highly valuable, accurate and the instrument allows us to perform an experiment that we could not possibly perform somewhere else. Using the 2 x2 x 2 mm we obtained acquisitions in less than 3 minutes for points close to the notch, as the beam only had half of the sample attenuating, and up to 5 minutes for sample far from the notch. This is four times faster than our previous experiments, with a resolution twice higher. An example map, obtained after unloading, is shown in Figure 4, for the map presented in Figure 5. It should be noted that the absolute strain values have been obtained in reference to the theoretical value of the d-spacing for pure graphite, rather than the larger lattice spacing of Gilsocarbon graphire. The ongoing analysis will provide the more relevant relative strains, with the unload state as reference.

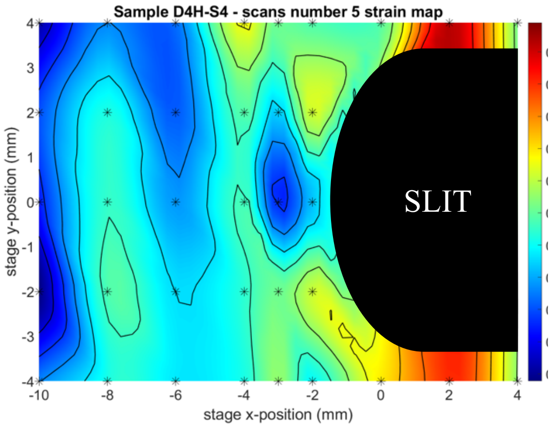

Figure 4: 36 points map, absolute strains (nominal scale), specimen with slit (The notch is to the centre right of the strain map and is aligned horizontally).

Because we want to see whether the elastic strains are fully recovered upon unloading and if relaxation of strains at the tip of the notch happens, which tends to indicate that microcracking or other irrecoverable damage mechanism occurs, we also need to obtain data for the tangential strains. Furthermore, we did not have time to scan the unnotched sample which would have allowed us to compare with other experiments we carried out at STFC ISIS ENGIN-X.

An application for extension has been submitted to ILL. In order to compensate for the difficulty of rotating the rig, and hence obtaining the tensile tangential component, we will change the geometry to a tensile notched specimen.

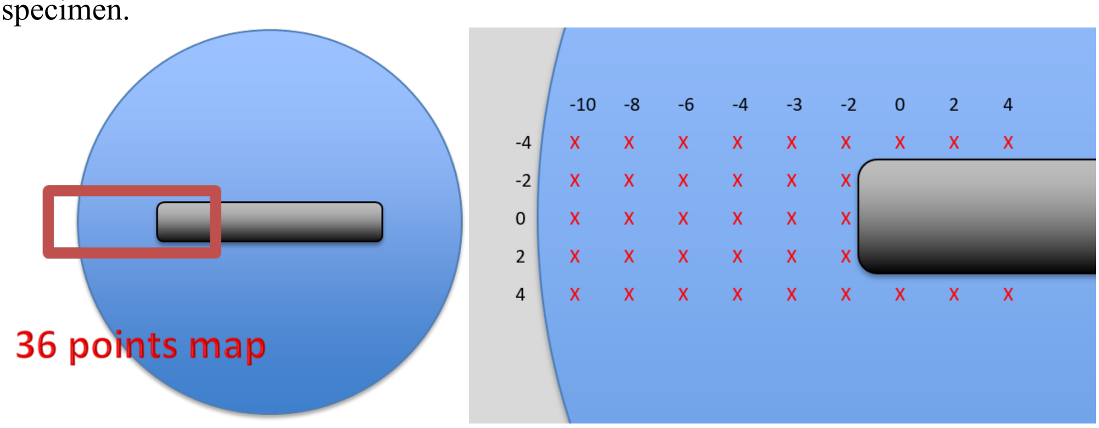

Figure 5: Map for 36 points.

4 Conclusions and future work

The instrument SALSA was the appropriate choice for our objectives and demonstrated its use and benefits in the study of brittle material fracture and notch sensitivity studies of nuclear graphite. It is reasonable to keep in mind that this instrument is particularly suited for metals, making this experiment quite challenging. In order to provide definitive conclusions, we will apply for an extension of four days.