High temperature strain mapping in graphite and MAX phase using synchrotron x-ray diffraction and radiography

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Abstract. Graphite and MAX phase ceramics are both candidate materials for core components of Gen IV nuclear reactors, with MAX phase also generating interest for use in the aerospace industry. These materials must be demonstrated to maintain structural integrity, which depends on the material properties at elevated temperature. In this work, Brazilian disk specimens of SNG742 fine grained graphite and Ti_2AlC MAX Phase ceramic have been loaded close to failure in diametral compression at elevated temperatures up to 1050 °C with in-situ observations using Synchrotron X-ray radiation. Elastic strains and bulk strains were measured via diffraction mapping and digital image correlation of radiographs respectively. The spatial correlation of these strains has been examined in tension and compression to study damage mechanisms and their temperature dependence. This work constitutes an important step to building a testing regime applicable to investigating damage tolerance in neutron irradiated materials at elevated temperature.

Introduction

Generation IV reactors are designed to operate at higher core temperatures and for longer lifetimes (60-80yrs) than their previous counterparts, with some rectors designs such as the Very High Temperature Reactor expected to operate with a core outlet temperature of up to 950 °C [1]. Graphite and MAX Phase (quaternary nano-layered carbides) are tolerant to elevated temperature and high neutron doses. These materials are candidates for structural materials that will experience static and dynamic loading due to irradiation and thermally induced stresses and external loads, which can alter their mechanical properties. A previous work on AGR graphite used neutron diffraction and optical image correlation to study changes in its deformation behaviour up to 850°C, with strain mapping also at at room temperature by digital volume correlation (DVC) of computed synchrotron x-ray tomographs with selected area diffraction in transmission [2]. High temperature nanoindentation (to 600°C) and cross-sectional TEM also demonstrated a change in the graphite crystal deformation mechanism [3]. Basal plane kinking at high temperature caused pseudo-ductility that may explain increased tensile strength at high temperatures. Max phase ceramics owe their intrinsic damage tolerance to the same mechanism of kinking, and experience increased ductility above 800°C. It is therefore of interest to study the relationships between total strain and elastic strain at elevated temperatures to better understand how this is affected by crystal deformation mechanisms.

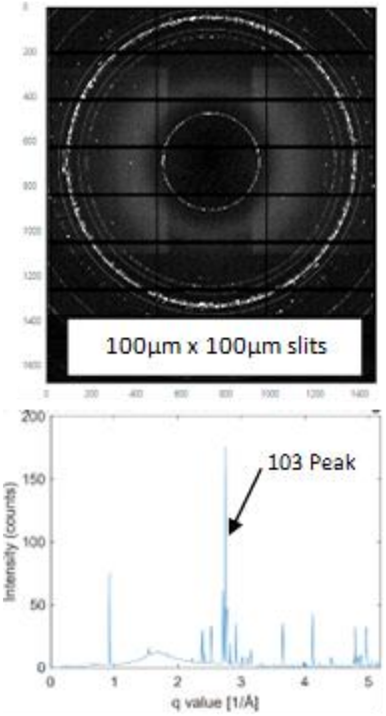

Figure 1: Top - raw Pilatus image, Ti_2AlC diffraction rings Bottom - processed and integrated spectrum

Experimental Materials and Methods

Sample Materials. The graphite samples were made from SNG742, a fine-grained graphite produced from a petroleum feedstock, manufactured by Sinosteel as part of the EPSRC UNIGRAF project. The Ti_2AlC MAX phase samples were produced by Leuven University (Belgium), by spark plasma sintering. The cylindrical samples (radius = thickness) were 2.5 mm and 1.5 mm in radius for the graphite and MAX Phase respectively.

Experimental Method. The experiment was conducted on the i12 JEEP beamline at the Diamond Light Source (Harwell, United Kingdom). Samples were loaded in diametral compression using an Instron Electro-thermal mechanical test system (ETMT), owned by the University of Manchester at Harwell group. Custom anvils were made to a design based on ASTM D8289-19; from Nimonic 80A for the graphite anvils and C1023 for the MAX Phase anvils. The anvil design includes a curved section of 30° arc, with the same radius of curvature as the sample radius. This design aims to reduce contract stress that can lead to early tensile failure when compared to other techniques. Loading in diametral

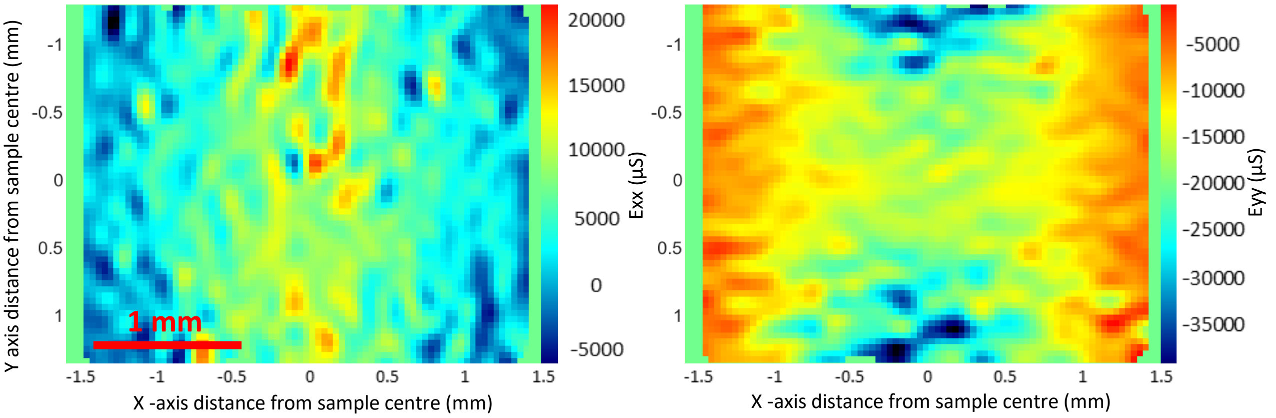

Figure 2: Total strain maps derived from DIC of radiographs. Graphite, 540 N nominal load, 800 °C

compression generates a biaxial stress state at the centre of the disk sample, allowing material properties to be investigated in tension and compression in a single test.

Monochromatic radiographs were collected using a PCO detector, with a pixel size of 1.30 µm. To create the speckle pattern necessary for high quality digital image correlation, the samples were sprayed with a high temperature paint doped with 5-25 µm diameter tungsten powder. Digital Image Correlation was performed using a least squares method via LaVision's DaVis software. The resulting strain maps are shown in Figure 2.

Monochromatic diffraction was taken using a flat panel Pilatus detector. Diffraction maps were taken over a 2.5 mm x 2.5 mm area with 0.1 mm x 0.1 mm slits and 0.1 mm spacing between map points. Sample detector distances were selected to optimise the collection of diffracted beam from (0002) for graphite and {103} for MAX Phase. The raw images from the detector were corrected for sample-detector distance, geometric structure of the detector and ring current normalisation before azimuth caking at 1° intervals in the DAWN software package. The peaks were fitted to a Psuedo-Voigt profile to measure their shifts in position with load from which the normal strains were calculated. Principle strains were then extracted by the application of a least-squares fit of Mohr's circle to the azimuthal variation of the normal strain. Example maps for the strains E_{xx} (0°) and E_{yy} (90°) in a graphite sample loaded to 540 N (nominal compressive stress at specimen centre of 82.5 MPa) at 800 °C are shown in Figure 3. Both the bulk and elastic strain maps show that the expected biaxial stress state was developed. The current analysis aims to spatially correlate the bulk and elastic strains as a function of temperature, with increasing load.

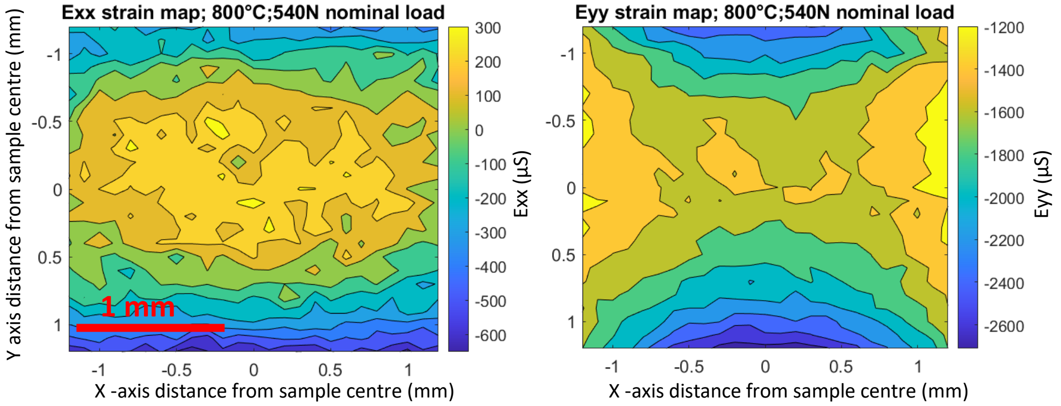

Figure 3: Elastic strain maps derived from diffraction data. Graphite, 540 N load, 800 °C

Conclusion

Synchrotron X-ray radiography and diffraction have been used to map elastic and bulk strains in fine-grained graphite and quaternary layered carbide MAX phase, at ambient and high temperatures. It has been shown that high spatial resolution bulk and elastic strain maps can be generated from these data. The spatial correlation of these two strain maps will elucidate the development of non-linear behaviour in tension due to damage, and the possible temperature dependence of this behaviour. The methodology developed during this experiment will form the foundation of future studies on neutron irradiated materials.

References

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