Strain Mapping in Graphite and MAX phase during High-Temperature Disc Compression via In-Situ Synchrotron X-ray Radiography and Diffraction

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Abstract.
Graphite and MAX Phase ceramics are materials under consideration as key core components in high temperature Gen IV reactors. Their structural integrity is dependent on the evolution of their material properties under elevated temperature and neutron irradiation, and also their performance under mechanical load at high temperatures. Test techniques are needed to measure the mechanical properties of small samples, which are suitable for irradiated materials. In this work, small 'Brazilian' discs (5 and 3 mm diameter) of the fine-grained graphite SNG742 and the MAX Phase ceramic Ti_2AlC were tested in diametral compression up to 80% failure load, at ambient and elevated temperature, achieved by resistance heating, up to 950°C. The testing geometry produces a compressive-tensile biaxial stress state at the centre of the discs. Simultaneous synchrotron X-ray diffraction in transmission and digital image correlation of radiographs enabled mapping of the elastic and bulk strains respectively at high spatial resolution within the specimens. The analysis aims to correlate these spatially to investigate the development of non-linear behaviour in tension due to damage mechanisms, and their possible temperature dependent. Ultimately, this approach will be applied to investigate the damage tolerance of neutron irradiated materials at elevated temperatures.

Introduction.

Generation IV fission reactors will operate at higher temperatures than previous reactors and with much longer lifetimes (60-80 year design life); some designs will operate at temperatures over 1000°C. Graphite and MAX phase (nanolaminated ternary carbides/nitrides) are tolerant to high temperatures and irradiation and thus are candidates for structural materials in Gen IV systems such as molten salt, liquid metal and gas-cooled reactors. In these environments they may experience mechanical property degradation due to neutron irradiation and coolant interactions and will be exposed to static and dynamic loading from thermal and irradiation gradients and external forces. To assure the structural integrity and safe economic lifetime of future advanced reactors, it is critical to understand how high temperatures and irradiation will affect the properties of their materials, and to select and design microstructures with optimal performance. Previous work on AGR graphite has used neutron diffraction and optical image correlation to study deformation up to 850°C, with strain mapping by digital volume correlation (DVC) of computed x-ray tomographs and selected area diffraction at room temperature [1]. High temperature nanoindentation (to 600°C) and cross-sectional TEM demonstrate a change in the graphite crystal deformation mechanism [2]. Basal plane kinking at high temperature caused pseudo-ductility, which may explain increased tensile strength at high temperatures. Max phases owe their damage tolerance to the same mechanism of kinking, and experience increased ductility above 800°C [3,4]. The aim of this study was to observe the effect of temperature on the nonlinear relationship between stress and strain in fine grained graphite and a MAX phase ceramic, and also investigate whether the occurrence of kinking can be detected by observation of textural changes using in situ diffraction under load.

Methodology.

Material and loading configuration. Discs of the fine grained graphite SNG742 (5 mm diameter, 2.5 mm thick), and the MAX phase Ti_2AlC (3 mm diameter, 1.5 mm thick) conforming to the 'Brazilian' disc design were paired with curved anvils of Nimonic 80A to a design derived from ASTM D8289–19 (Fig.1) to generate a compressive-tensile biaxial stress state at the centre of the sample when under compressive loading from two diametrically opposed positions. The curved nature of the anvils aids in reducing the likelihood of contact stress related early tensile failure when compared to other techniques.

$$\sigma_{sts} \approx \frac{P}{\pi LR}\left[1 - \left(\frac{b}{R}\right)^2\right] \quad (1)$$

Utilising Formula 1 [5,6], where σ_{sts} = splitting tensile strength, P = maximum applied load, L = thickness of the specimen, D = diameter of the specimen, b = circumference of contact length and R = specimen radius, the aforementioned sample geometries were calculated to allow for loading to failure within the 3kN load cell limit of the Electro-Thermal Mechanical Test (ETMT) rig on the i12-JEEP beamline at Diamond Light Source.

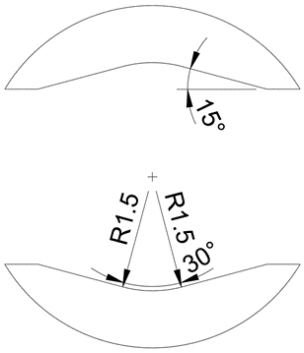

Figure 1. Diagram of the curved part of the Nimonic 80A anvils for the Ti_2AlC sample. R1.5 is the radius of the sample. Loading is along the vertical axis.

Measurement and analysis techniques. Each sample was loaded in 5 steps up to 80% of the typical failure load at room and elevated temperatures, of 800°C and 950°C for graphite and Ti_2AlC respectively, achieved via resistance heating in an argon environment. The sample was held under fixed displacement at each load step to record monochromatic radiographs and a monochromatic transmission diffraction map of a 2.5 x 2.5mm area using 0.1 x 0.1mm slit size. Digital image correlation (DIC) of the radiographs of the sample surface, augmented with a speckle pattern of high temperature paint containing 5-25µm diameter tungsten (W) powder, produces spatial maps of the two dimensional strain field (Fig. 3). Peak fitting of 10° azimuth cakes of the Bragg diffraction rings, (Fig. 2) observed using a 2D flat panel detector, was used to determine the peak shift relative to the pre-load. This allowed for the calculation of the normal lattice strains in the crystals of the graphite (basal plane, 0002) and MAX phase (103 plane). From this, the principal strain components can be extracted via the application of a least-squares fit of Mohr's circle to the azimuthal variation of the normal strain. Maps of the spatial variation of the lattice strains can then be produced and correlated with the total strains at the same locations. This analysis was previously applied in room temperature studies of a coarse grained graphite [1]. Post-test analysis (EBSD and TEM), at locations selected using the strain maps, will examine the relations between the temperature, local strain state and microstructure deformation.

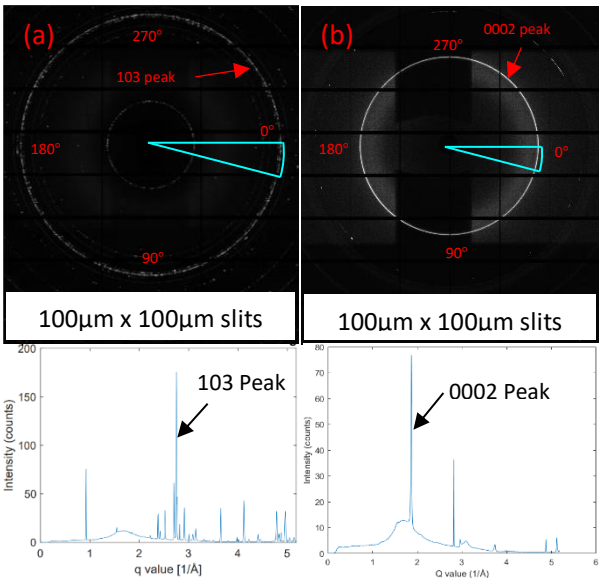

Figure 2. Bragg's rings collected at the centre of a diffraction map of (a) Ti2AlC and (b) SNG742 graphite. The 0°-10° azimuth cake is shown in blue. The corresponding diffractogram is featured below with the peak of interest highlighted.

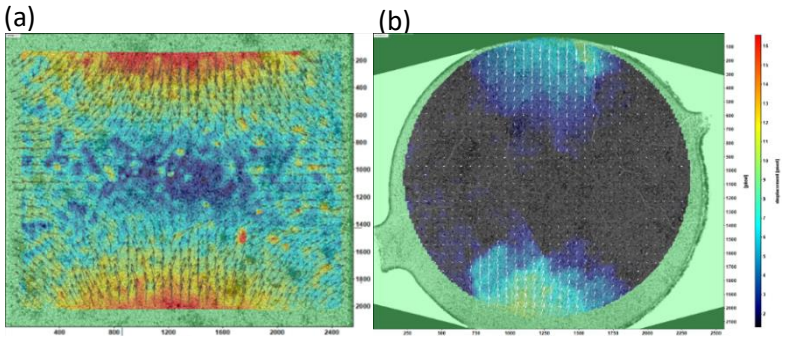

Figure 3. Displacement maps of (a) SNG742 graphite and (b) Ti_2AlC under 70% of failure load whilst at ambient temperature (21°C). Vectors indicate displacement direction, their lengths are the pixel displacement increased by a factor of 10. The centre of the sample is at the centre of the map and loading is along the vertical axis.

Conclusion.

Synchrotron X-ray radiography and diffraction have been used to map elastic and bulk strains in fine-grained graphite and Ti_2AlC MAX phase, at ambient and Gen IV fission reactor relevant temperatures. Maps of the elastic and bulk strains at high spatial resolution will guide the ongoing analysis which aims to correlate these strains spatially to investigate the development, possible temperature dependence of the damage mechanisms associated with non-linear behaviour in tension. The data from this study will form the baseline for future studies on irradiated materials.

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