Mechanical testing techniques suitable for high temperature characterisation of irradiated nuclear graphite grades

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Abstract. Within a nuclear reactor, graphite properties are strongly modified by irradiation, chronic oxidation and elevated temperatures. To ensure safe and economic operation it is necessary to measure the mechanical properties, but the volumes of material available for such tests are highly constrained. The development of tests with smaller specimen sizes would maximise the information available, but must be validated to ensure good quality data is obtained even where the microstructures might be relatively coarse. A key benefit of using graphite moderation in a fission reactor is the ability to reach higher temperatures. Test methods must therefore also be validated at these temperatures if the complete behaviour of a graphite is to be understood. Two mechanical testing techniques are explored for small sample characterisation: diametral compression and shear punch. Both techniques were applied to at least two grades of graphite with microstructures ranging from medium to ultra-fine grained. A comparison of diametral compression behaviour across the three microstructures will be explored and the first demonstration of shear punch testing of graphite will be presented both at room temperature and elevated temperatures.

Background

Nuclear graphite is used for fission reactor moderation and is a key technology in Generation IV+ reactor designs, including the (very) high temperature reactors ((V)HTR), some small modular reactors and molten salt reactors [1]. Throughout the reactor lifetime the graphite is subjected to harsh conditions of fast neutron irradiation and chronic oxidation at high temperatures. This environment causes a dramatic evolution of properties [2]. Understanding the behaviour of a reactor requires accurate determination of these evolutions, and hence precise measurements both before and after irradiation.

Nuclear graphite behaves as a composite of two graphitised phases: the filler particles and the binder. The bulk form is a strain-softening quasi-brittle material with a strongly asymmetric failure locus; it is around four times stronger in compression than tension. Graphite has refractory properties, with both the stiffness and strength increasing as the temperature rises, contrary to most engineering materials [3]. The common goal for manufactures of modern nuclear graphites is to arrange the strongly anisotropic crystals to create a bulk material with isotropic mechanical and thermal properties.

During operation, reactor properties should be monitored and forward predictions made to ensure safe and economic operation. The properties of graphite under irradiation are known to be highly sensitive to the manufacturing parameters. As such, any new source of graphite intended for reactor use must also be qualified by accelerated irradiation programmes in a material test reactor (MTR). The volumes of material available for characterisation, particularly for qualification but also for Gen. IV+ operation, are highly constrained.

For the UK's current graphite-moderated reactors, the strength and stiffness must be measured from samples of the order of 6 x 6 x 19 mm [4]–[6]. MTR programmes vary, and with a limited volume available during each irradiation cycle it is desirable to irradiate as a small a sample as possible, but typical sample geometries are of the order of diameter 8 x 6 mm cylinders [7]. There is therefore a drive to demonstrate, develop and standardise new techniques for mechanical characterisation based on smaller geometries. Two techniques under development will be explored.

Small sample techniques

Diametral compression was used for Magnox graphite PIE for decades and was recently standardised for graphite in ASTM-8298, for testing as-manufactured graphite at room temperature [8]. This technique estimates the tensile strength of disc specimens, for diameters between 6 and 19 mm. For MTR programmes adoption of this technique is revolutionary, allowing many more samples to be included in the programme, compared to reliance on traditional tensile or flexural methods. However, there is scant diametral compression data for fine grain graphites degraded by any combination of irradiation, oxidation or elevated temperature. This information is vital for ensuring that diametral compression can be used as a qualification technique. The diametral compression technique has been applied to three grades of as-manufactured graphite, and discs of diameter 6, 8 and 12 mm. These tests were monitored with in situ electronic speckle pattern interferometry (ESPI) to determine the tensile failure strain on the sample surface. From this data the effect of geometry and microstructural coarseness can be determined on the failure behaviour. This is in preparation for characterising ex-reactor material taken from the UK's nuclear fleet.

It would be a great advantage to adopt an even finer-scale technique for mechanical characterisation. A technique based on a disc geometry with a sub-millimetre thickness would increase MTR capsule loading factors by an order of magnitude. Originally developed for metallic systems, shear punch measurements can be made on discs of diameter 8 x 0.5 mm, in line with the ASTM-standardised geometry for small punch testing (see ref [9]). The small sample geometry and contained apparatus makes shear punch an ideal approach to adopt for high temperature strength testing. The first demonstration of shear punch testing on graphitic materials will be presented, and furthermore, the capability to test the graphite grades at high temperatures with this technique.

Conclusion

The constraints of nuclear irradiation drive an ever-increasing need for characterisation technique miniaturisation. To support Gen IV+ reactor systems, two techniques for measuring the mechanical properties of graphite will be presented. The data presented will include first-of-its-kind demonstrations of shear punch testing at high temperatures.

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