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Fatigue micromechanism characterisation in carbon fibre reinforced polymers using synchrotron radiation computed tomography

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ABSTRACT

In situ synchrotron radiation computed tomography (SRCT) has been used to evaluate fatigue damage micromechanisms in $[90/0]_s$ carbon fibre reinforced epoxy double-edge notched specimens. Interactions between cracks and toughening particles have been identified within the epoxy, particularly: particles de-bonding ahead of the main crack tip, creating a preferential damage path, and the bridging of cracks by un-failed ligaments. The critical mechanism of fatigue crack growth appears to be the degradation of bridging ligaments in the crack wake. Damage has been quantified in terms of crack opening and shear displacements, and the results have been compared with corresponding damage occurring due to quasi-static loading of the same materials. The removal of bridging ligaments in fatigue loading results in higher, more uniform crack opening (and shear) displacements and less serrated crack fronts. These observations have potential implications for material development, damage resistant and damage tolerant structural design approaches.

1. Introduction

Carbon fibre reinforced polymers (CFRPs) are well established as an important weight-reducing structural technology, particularly within the aerospace sector due to their high specific stiffness and strength [1]. CFRPs are widely identified as being very fatigue resistant, however, this advantage is often not entirely exploited in design, therefore, understanding the durability of these materials is of great interest. Fatigue design methodologies for composite laminates are not well-established due to their degradation via multiple interacting damage modes, including fibre/matrix debonding, matrix cracking, delamination, and fibre breaks [2,3]. Damage mechanisms have been extensively investigated using different approaches, resulting in various modeling strategies for predicting fatigue life [*e.g.* [4–16]]. However, reliable predictions based on physical observations remain key to improving CFRP structural design.

Different non-destructive techniques have been employed historically to detect fatigue damage in composites: for example, ultrasonic C-scans to evaluate delamination [17–19], acoustic emission to monitor the formation and growth of damage [20–23], thermography and thermo-elastic stress analysis to correlate damage and surface strains [24,25]. These methods have intrinsic limitations, such as the inability to provide direct information on type, size and orientation of damage, to resolve fine-scale failure events (e.g. fibre/matrix debonding and fibre breaks), and to provide a thorough three-dimensional representation of damage. In this respect, computed tomography (CT) has become established as a powerful technique for contemporary material science studies, facilitating multiscale analysis (macro-, meso-, micro- and nano-scale) of material structure and damage to be performed [26–31]. CT has been used on fibre-reinforced polymers to evaluate the local volume fraction and the orientation of reinforcement [32], to assess voids and internal damage in glass/epoxy [33,34], and to investigate fibre fracture of quartz/epoxy bundles [35]. Previous studies conducted on CFRPs have demonstrated that high resolution CT allows the imaging of damage at the scale of individual broken fibres [36], and that a voxel resolution on the order of 1 μm is reasonable to detect and distinguish primary damage modes [37]. Damage of carbon/epoxy subjected to impact and quasi-static loading has been quantified using synchrotron radiation computed tomography imaging, evidencing multiscale interacting 3D failure processes [37–40]. However, to the best of the authors' knowledge, there have been no *in situ* CT investigations of the fatigue behaviour of polymer matrix fibre composite materials.

In this paper, carbon/epoxy cross ply laminates containing toughening particles have been examined using *in situ* SRCT imaging to characterise the micromechanical fatigue behaviour and to

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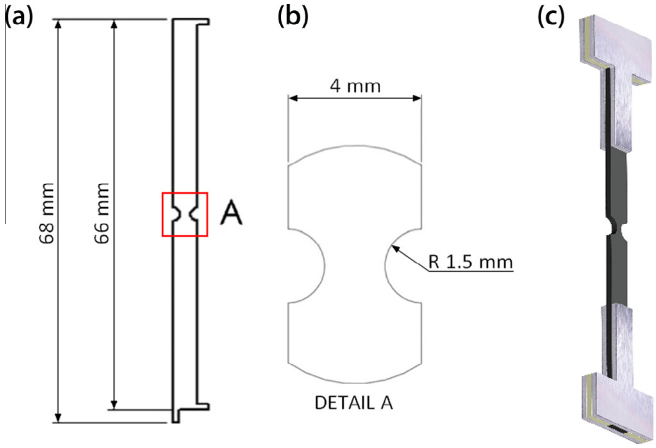

Fig. 1. *In situ* test specimen: (a) geometry and dimensions of the specimen, (b) detail of the notch, and (c) specimen with tabs bonded.

quantify damage. Comparisons are made with the results of a previous study [41] conducted on damage growth under quasi-static loading of the same material system.

2. Materials and methods

A thermoplastic particle toughened [42,43], M21/T700 carbon/epoxy prepreg (nominal volume fraction of fibre 60%), with a $[90/0]_s$ layup was investigated. The material was laid up and auto-clave cured using a standard aerospace cure cycle as specified by the supplier [44]. Double-edge notched specimens, with an overall gauge length of 66 mm and width of 4 mm, were prepared from the laminated plates via water jet cutting. Two semi-circular notches of radius 1.5 mm were introduced during the water jet cutting, leaving a nominal central cross-section between the notches of 1 mm. Aluminium tabs were attached to facilitate loading, as reported by Wright et al. [40], Fig. 1. The average ultimate tensile failure stress (σ_f) was previously measured as 960 MPa across the notched cross-section [40]. Tensile fatigue tests, with a sinusoidal waveform and load ratio of $R = 0.1$, were performed using two peak cyclic load levels, corresponding to 30% and 50% of σ_f, at a frequency of 10 Hz, up to a total of 10^4 load cycles. The number of cycles chosen is determined by the need to keep the damage, particularly the splits generated at a particular load levels within the field of view of a single SRCT scan.

Fatigue cycling was initially carried out using a standard servo-hydraulic load frame. After this fatigue loading was applied, specimens were scanned at the Swiss Light Source (on the TOMCAT-X02DA Beamline, Paul Scherrer Institut, Villigen, Switzerland). The distance between specimen and detector was set to 30 mm providing a degree of phase contrast (edge detection regime). The beam energy was 19 keV (multilayer monochromator, energy bandwidth of 2–3%). Specimens were placed in the load frame on the rotating stage, and scanned via SRCT in an unloaded and then loaded state. To ensure that the application of a tensile load in the *in situ* load frame did not cause additional damage propagation, the load chosen was 10% less than the maximum peak load used for the fatigue tests. During each tomographic scan, 1500 projections were collected on a 2560×2160 pixel detector, through a rotation of 180°. The exposure time for each radiograph was 150 ms, resulting in a total scan time of approximately 4 min. An isotropic voxel resolution of 1.5 μm was achieved; a value, which was considered reasonable to inspect damage at the level of individual fibres and toughening particles within the resin. Three-dimensional reconstruction was obtained from radiographs using an in-house code based on the GRIDREC/FFT approach [45].

Fatigue damage was detected and studied using the commercial software VG studio Max v2.1. Initially reconstructed volumes were analysed by inspecting orthogonal 2D slices. Bright fringes around the cracks resulting from phase contrast were exploited to identify cracks, particularly where these were only marginally open. Defining a region of interest around the damage and applying a semi-automated segmentation technique, based on the seed-growing algorithm [37], fatigue damage was segmented, as shown in Fig. 2; in which the bulk composite material has been set as semi-transparent and the crack volumes coloured by type. Defining the effective spatial resolution of a given CT image is a non-trivial issue: modulus transfer functions, point spread and line pair separation are well established aspects, but these do not straightforwardly take into account the specific sample geometry and X-ray path dependences that arise in computed tomography. Works by Bull et al. [46] and Guvenilir [47] note the ability to exploit the so-called 'partial volume' effect to extend the resolution of crack opening measurements to sub-voxel level. In this work a sub-voxel estimation has not been carried out, although a consistent method of estimating and comparing crack opening between samples has been adopted, with typical results illustrated in Fig. 3. Reconstructed data was analysed without additional post-processing (de-noising, edge enhancement, e.g. median filtration, anisotropic diffusion).

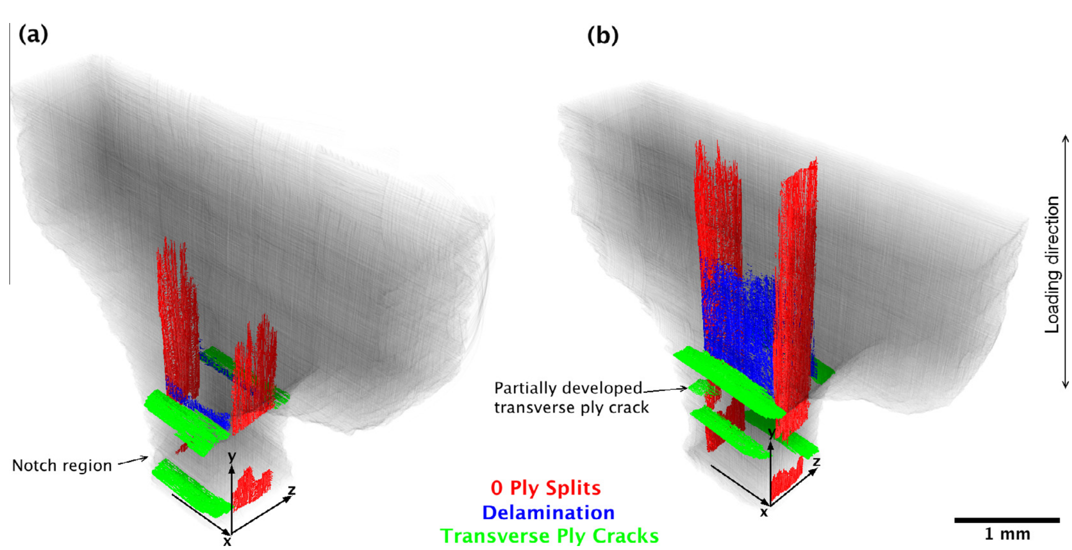

Fig. 2. Three-dimensional fatigue damage for: (a) 30% σ_f and (b) 50% σ_f, at 10^4 load cycles.