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In situ synchrotron tomographic evaluation of the effect of toughening strategies on fatigue micromechanisms in carbon fibre reinforced polymers

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Micromechanisms of fatigue failure in double-notch cross-ply carbon/epoxy coupons have been investigated using synchrotron radiation computed tomography (SRCT). The fatigue behaviour of toughened and untoughened matrices has been compared, highlighting similarities and differences of damage modes in terms of fatigue initiation and propagation. Results show that damage does not propagate evenly for the toughened systems: the presence of resin rich regions constrains crack propagation, which is shown to suppress damage growth. In contrast, untoughened material is characterized by more uniform crack progression. The presence of toughening particles in the resin system favours complex local crack geometries, such as crack deflection and the formation of bridging ligaments along the crack wake due to particle debonding events. A distinctive aspect of fatigue loading is identified in the degradation of crack bridging ligaments associated with increasing number of cycles.

1. Introduction

Carbon fibre reinforced polymer (CFRP) composites are widely recognised as a key lightweight structural technology, underpinning major engineering applications such as current and next generation airframes [1]. Whilst often driven by primary properties such as specific strength and stiffness, factors such as resistance to fatigue and corrosion are critical in a number of contexts. Previous studies have focused on assessing fatigue damage mechanisms and the link to fatigue life using various investigation techniques [2–8]. However, overly conservative and empirical design methods predominate in engineering practice. Thermoset composites may exhibit high strength and stiffness [9], fundamental requirements in structural design, but achieving adequate damage resistance has often proved more challenging [10]. In fifty years since the original development of carbon fibres, there has been a consistent emphasis placed on enhancing composite toughness, particularly under impact loading conditions. Improving the fracture toughness of thermoset resins has been the focus of numerous studies, which have explored a range of solutions such as the introduction of rubber particles [11–14], thermoplastic particles [15–17], or high stiffness filler particles [18,19]; including variations in particle dimensions from the nanometer to the micrometer scale [20]. To date, several approaches to toughening have been developed to a sufficient level of maturity that they are employed in service in CFRPs for aerospace applications; however, the underpinning micromechanisms of failure associated with the modified microstructures have not been widely investigated. Studies on toughening micromechanisms in composite laminates are relatively limited [10,21–24], and the consequence of fatigue loading on the effectiveness of toughening strategies in particular has been largely neglected.

High resolution X-ray computed tomography of composites has emerged as a powerful tool to assess damage micromechanisms for small coupons subjected to quasi-static [25], impact [10,23], and fatigue loading [26]. A resolution on the order of 1 μm has been shown to be appropriate for capturing both primary damage events (i.e. ply cracking), along with key local events, as the decohesion of toughening particles or fibres, facilitating correlation with primary microstructural features such as the ply structure and the presence of resin-rich and fibre-rich regions.

The present work represents the first use of *in situ* synchrotron radiation computed tomography (SRCT) imaging to understand the micromechanisms operating in toughened and untoughened composite systems subjected to fatigue loading. The comparison of damage initiation/propagation for three different material configurations provides insights as to the effect of fatigue on shielding

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micromechanisms in toughened composite systems. The particular focus of this work is to determine the evolution of the crack tip process zone and the corresponding damage events at the length-scale of individual microstructural features (e.g. toughening particles, and fibres).

2. Materials and methodology

2.1. Material systems

Three carbon/epoxy material systems, all produced by the Hexcel Corporation, have been assessed in the present work: two toughened systems; one containing thermoplastic toughening particles (T700/M21), the other utilizing a homogeneous, intrinsically toughened matrix (IM7/8552), and an untoughened system (IM7/3501-6). All systems contain intermediate modulus carbon fibres. A cross-ply lay-up was used, with a $[90/0]_s$ stacking sequence for the particle-toughened system, and a $[90/0]_{2s}$ lay-up for the other two systems. The nominal overall laminate thickness was maintained at ~1 mm for all three systems. Materials were laid up and autoclave cured as flat plates using a standard aerospace cure cycle [27–29]. Rectangular coupons containing two semi-circular notches of radius 1.5 mm were introduced by water jet cutting, leaving a nominal central cross-section between the notches of 1 mm. The geometry and the dimensions of the specimens used are shown in Fig. 1. A coupon length of 34 mm was selected with the requirement to fit the specimen into the compact/portable fatigue loading device to perform *in situ* experiments. The average ultimate tensile failure strength (UTS) for each material system has been evaluated in previous studies [30], and is calculated based on the failure load and the net cross-sectional area between the notches. Values reported are: 918 MPa for the particle-toughened system, 1419 MPa for the untoughened, and 1271 MPa for the intrinsically toughened system [30]. The consistent tendency for the specimens to split and delaminate extensively from the notch before the final ultimate failure event is considered to justify the use of the net section to calculate the nominal UTS.

2.2. Fatigue tests and SRCT scan procedure

Tensile fatigue tests with a peak load of 50% of the nominal UTS were performed using a load ratio of $R = 0.1$. Fatigue cycling was initially carried out at a frequency of 5 Hz using a standard servo-hydraulic load frame to apply 700 load cycles. After the pre-cycling had been applied, each specimen was placed in the *in situ* load frame, a load less than the maximum peak load (~90% of peak) was applied to open the crack, and the coupon was scanned. Following these initial scans, the *in situ* load frame was used to apply an additional 100 cycles, and then the specimens were imaged again. For the particular micromechanistic focus of this work, the high resolution used and the full-field imaging afforded by SRCT allowed detailed observations to be made with relatively few cycles applied. Scans were conducted at the Swiss Light Source (SLS), TOMCAT-X02DA Beamline, Paul Scherrer Institute, Switzerland. The specimens were placed at a distance of ~22 mm from the detector to allow a degree of phase contrast to be obtained. This facilitates the identification of cracks with small opening displacements. During each tomographic scan, 1500 projections were collected through the rotation of 180°. The beam energy was 19 keV, and an isotropic voxel resolution of 0.69 μm was used. Three-dimensional reconstruction was obtained from radiographs using an in-house (SLS) code based on the GRIDREC/FFT approach [31]. No post-processing, such as filtering, de-noising and edge enhancement were applied to the final scanned volumes. The region of interest for all scans was the notch zone; in particular, due to the field of view (~1.4 mm) associated with this resolution, growth from one notch tip was monitored in detail (rather than both notches), see Fig. 1(c).

2.3. Damage analysis

Micromechanical analysis of damage initiation and propagation has been conducted considering corresponding 2D slices (of the same region of interest) between the two cyclic conditions for each material system. Volume scans of each coupon were obtained for the two cyclic counts to facilitate the comparison.

Damage segmentation allows the influence of microstructure on the crack growth to be understood. Segmentation was performed using the VG Studio MAX v2.1 package via a "seed-growing" algorithm. A detailed sub-voxel interpolation has not been carried out in this case. As such, the error associated with the segmentation process is on the order of the voxel resolution (±0.69 μm). However, for the purposes of this work, the segmentation method utilised was consistent between specimens, and did not substantively affect the mechanistic assessment of crack morphology and microstructural interactions. Segmented damage

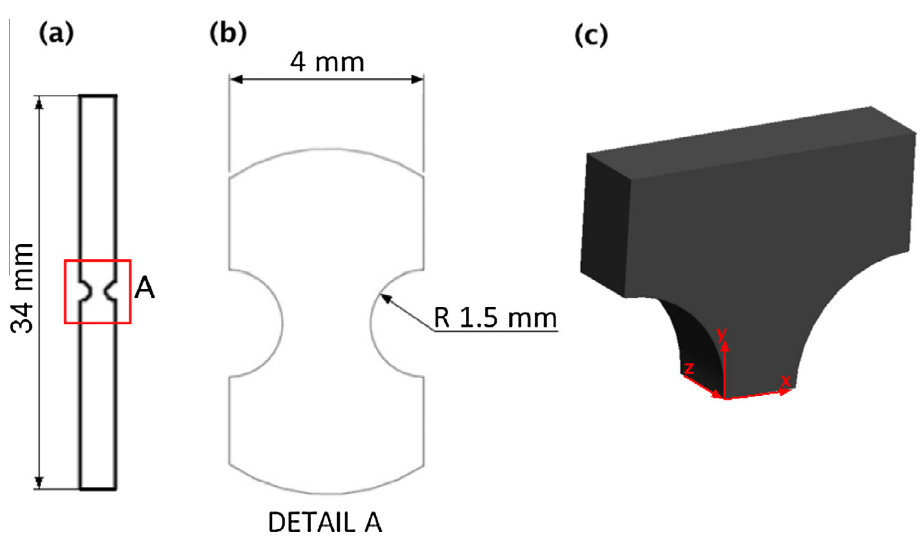

Fig. 1. Specimen used for *in situ* fatigue tests: geometry and dimensions (a), (b) of the notch region, (c) schematic of the region considered during the scan, and reference coordinate system used.