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Mapping fibre failure *in situ* in carbon fibre reinforced polymers by fast synchrotron X-ray computed tomography

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ABSTRACT

Fast, *in situ* synchrotron X-ray computed tomography (CT) has been used to capture damage evolution, particularly fibre failures, before final fracture (within 99.9% of the ultimate tensile stress) in cross-ply carbon fibre/epoxy coupons under continuous monotonic tensile loading for the first time. It is noteworthy that fewer than 8% of the fibres in the 0° plies have fractured at 99.9% of the failure load. The majority of fibre breaks appear as isolated events, although some instances of multiple adjacent breaks (clusters) do occur at intermediate and high stress levels. Contrary to conventional wisdom, a cluster of failed fibres always occurred in a burst as a singular failure event: clusters were never seen to accumulate additional broken fibres as load increased suggesting low-level stress concentration local to fibre breaks. Several instances of multiple fractures along individual fibres were observed, providing an estimation of the critical stress transfer length between the fibre and matrix. The factors affecting fibre failure appear to be complex, with distinct sample-to-sample variability being identified for the length-scale tested. This highlights the need for improved understanding of the mechanisms that contribute to final failure, particularly criteria controlling the arrest or otherwise of clustered fracture events.

1. Introduction

Reliable engineering predictions of carbon fibre reinforced polymer (CFRP) based on physically representative mechanisms are a key to improve CFRP structure design and material development [1]. Such an understanding may help to alleviate the time-consuming and expensive test programmes employed in the contemporary development of primary load-bearing structures. Of the characteristic failure mechanisms of CFRPs, fibre failure is widely identified as critical in unidirectional materials/plies when loaded in tension along the fibre direction. Different approaches have been exploited to predict such fibre-dominated behaviour [1], including analytical models [2–7], statistical models [8–10], and finite element models [11–13]. Such models generally incorporate some allowance for the distribution of individual fibre strengths, the local stress transfer to the fibres from the matrix adjacent to fibre breaks, and, in some cases, the concept of a critical cluster size that triggers final failure [4,14]. Despite the efforts made, contemporary models have only been partially successful in predicting experimental observations for fibre failure, which in themselves are relatively limited within the literature [7,11–13]. Scott et al. [15] have previously applied *in situ* CT to visualise the progress of fibre failure of CFRP under incremental loading, clearly showing that fibre breaks occur increasingly at higher loads and that their accumulation follows a power law curve to a good approximation. Clusters of multiple adjacent breaks were associated with high stresses, with the maximum applied stress being ~94% of the ultimate tensile stress (UTS) of the material in question [15]. These results were directly compared with models in Refs. [7,12], highlighting difficulties in predicting cluster formation at high loads [12]. Furthermore, gaps in understanding fibre fracture accumulation processes have been identified, such as the formation of coplanar and dispersed clusters, and the initiation at new locations [7]. Whilst it is evident that CT is a powerful technique for following damage accumulation using both *in situ* and *ex situ* experiments for qualitative and quantitative assessments [16]; previous *in situ* studies of fibre failure under tension have been characterised by low temporal resolution (~5 min per tomograph) with the specimen held at constant load during the acquisition of all the projections comprising a scan [15,17]. Such maintained loads

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may introduce changes in the material behaviour, particularly in the formation of clusters sustained at high loads [11] and they make difficult to capture the accumulation of fractures within clusters. In addition, given the variability of commercial CFRP coupons, important practical limitations arise in capturing damage events just prior to final failure (within 1% of UTS). Fast synchrotron radiation CT (scan time of 1 s or less) during continuous slow strain rate loading has the potential to overcome these limitations [18–20].

In this study, fast computed tomography was used for the first time to track the accumulation of fibre breaks to within 1 s of final failure, under simple ramp loading representative of standard engineering tensile tests. The combination of continuous monotonic tensile loading, and fast acquisition has allowed to : (i) avoid potential hold-at-load artefacts, (ii) capture of the sequence and location of successive fibre fracture sites at much finer load steps than reported previously, and (iii) observe the state of fracture immediately prior to final failure (to within ~0.1% of the sample-specific UTS).

2. Methodology

2.1. Material system

A commercial aerospace-grade thermoplastic particle toughened carbon/epoxy (T700/M21), Hexcel HexPly [21]), with a nominal fibre volume fraction of 60% [15] and a $[90/0]_s$ layup, was studied. Double-edge notched specimens, with a central cross-section between the notches of 1 mm, length of 66 mm and a width of 4 mm were shaped by waterjet cutting from a panel with 1 mm of thickness. Further details of the geometry and coupon dimensions are reported in Ref. [22]. Two coupons are studied in this investigation (identified here as *coupon A* and *coupon B)* to assess the reproducibility of the observed behaviours. It should be noted, that despite the small specimen size, there are more than 7000 fibres in the 0° plies between the two notch tips, with a visualised gauge length of ~2 mm over which the fibres breaks are observed (see §2.3 below).

2.2. In situ monotonic tensile loading experiments

Uninterrupted rising tensile loading was applied *in situ* upon the CT beamline while the coupons were continuously scanned. Tensile tests were performed using a compact electro-mechanical rig developed by INSA-Lyon [20], specifically designed to be stable at high rotational speeds. The strain rate applied was $\sim 3 \times 10^{-4}$/s. The UTS registered was 1400 MPa for coupon A and 1280 MPa for coupon B. These values are higher than that reported in previous studies (960 MPa [17]), which was based on an average of 10 specimens (coefficient of variation of 0.03 [23]). This difference might be mainly related to the smaller cross-section at the notch used in previous studies, reported as 0.7 mm width between notches in Ref. [12]. Final failure was seen to occur at the notch in a catastrophic manner for both coupons considered.

2.3. Fast CT acquisition procedure

Experiments were performed at the Swiss Light Source on the TOMCAT-X02DA Beamline, Paul Scherrer Institut, Villigen, Switzerland. The beam energy used was 20 keV and the distance between the specimen and the detector was set to provide a degree of phase contrast to facilitate the visualisation of small crack-like features [24], such as fibre breaks [25]. The exposure time was set to 2 ms and 500 projections were collected for each tomograph, resulting in 1 tomograph per second. The voxel size was 1.1 μm corresponding to a field of view of ~2.2 × 2.2 mm, sufficient to image the notch region shown in Fig. 1. Coupons were initially scanned in the unloaded condition to confirm that no significant damage was introduced during the manufacture. All cutting damage was confined to the near-surface, and as such it was readily excluded from subsequent internal damage evolution under load. The specimens were then mounted in the rig and loaded in tension. The random access memory (RAM) storage capability of the camera limited the data acquisition to roughly twelve consecutive scans (6000 projections) of the entire region of interest (2016 × 2016 pixels). In continuous mode the image data was overwritten when the storage capability of the camera was reached. The above settings were also used to obtain information at low and intermediate loads, the difference being that when the scan was stopped only the last volume of the sequence (of 12) was considered and saved. This approach avoids having interruptions during the tensile test to perform intermediate scans in the '*start and stop*' mode. At high loads continuous acquisition was used until final failure of the specimen, giving 10 continuous scans immediately prior to failure for coupon A, and 9 for coupon B. When coupon failure occurred the acquisition was manually stopped and the data downloaded from the camera. The percentage of the sample-specific UTS associated with these final continuous scans was in range of 99.4%–99.9%. Two types of reconstruction have been considered, based on: (i) conventional X-ray attenuation (edge enhanced in this case by acquisition in the near-field Fresnel regime) and (ii) phase retrieval, via the Paganin method [26]. In house GRIDREC/FFT code was employed in both cases [27].

2.4. Image analysis

Previous work has established that a voxel size of ~1 μm allows individual fibre breaks to be detected in carbon fibre composites [25]. Fast tomography is characterised by a short exposure time and fewer projections per scan, with consequent compromises in signal to noise ratio and effective spatial resolution compared to that which is achievable using conventional settings [25]. Fig. 2 illustrates the same cross-section parallel to the load direction using conventional absorption (Fig. 2(a)), and Paganin phase reconstruction (Fig. 2(b)), without applying any image post processing. Paganin reconstruction enhances the contrast between damage and material (fibres and matrix), but compromises sharpness cf. Fig. 2(a). The Paganin reconstruction was seen to be less suited to distinguish small features such as single fibre breaks, but it facilitated the segmentation of more open cracks, such as matrix failure (see split in Fig. 2). As such, the analysis of fibre breaks here was conducted using absorption-based reconstructions, while the Paganin results were used for 3D rendering of the different damage modes before final failure, as shown in Fig. 1. Reconstructed volumes were filtered by a median filter, ensuring a degree of edge preservation. An example of the improvements obtained by applying the median filter is provided in Fig. 2(c) for the same cross-section shown in Fig. 2(a)–(b). For each coupon the volume of interest for successive scans was first registered using ImageJ to facilitate the correlation of failure sites between subsequent load increments. VGStudio Max 2.1 was used to allow the fibre breaks to be detected and manually counted. The numbers of fibre breaks reported are based on three independent counts for each volume.

3. Results

3.1. Progression of the overall damage in the notch region

Damage modes in the notch region have been described previously for tensile loads up to 94% of the UTS [15]. Here the higher