UNDERSTANDING THE DAMAGE ACCUMULATION AND TENSILE STRENGTH IN CARBON/EPOXY COMPOSITES USING HIGH RESOLUTION COMPUTED TOMOGRAPHY

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Abstract

In situ synchrotron X-ray computed tomography is performed to investigate the fibre break accumulation mechanisms in untoughened aerospace grade carbon/epoxy coupons under quasi-static load. The technique is data-rich and allows us to analyse the fibre break accumulation mechanism from low to high strains, showing the interactions between damage and microstructure. This study provides information on the accumulation and interaction of fibre breaks in simple double-notched coupons, where groups of interacting fibre breaks (clusters) are observed and quantified. Implications for ongoing modelling efforts are discussed in terms of mechanisms and parametric calibration. Focus is given to the geometry of the singlets and duplets (groups of two broken fibres). The nearest neighbouring distances are evaluated for the cases of two intact fibres, one broken and one intact fibre and two broken fibres, showing that two fibres prefer to break in a nearest neighbouring fashion.

1. Introduction

Carbon fibre reinforced polymers (CFRPs) are a critical structural material for aerospace engineering. Current conservative design approaches lead to manufacturing and testing methods that are unnecessarily inefficient and costly. The use of well-understood, physically accurate performance simulations will allow for more efficient use of material, reducing aircraft weight and improving fuel efficiency, without compromising safety.

Fibre failure is the key mechanism controlling final failure in CFRPs, particularly under tensile loading [1,2]. High resolution *in situ* computed tomography enables identification and quantification of individual fibre break formations. This can be used to improve directly and validate predictive failure models, which as yet are still unable to account for factors controlling the onset of unstable failure [3,4]. Previous work has quantified the drastic increase in fibre breaks at high stress levels [4,5]. As such, to have a better chance to capture mechanisms behind the formation of critical interacting groups (clusters) of breaks, and the corresponding progression of clusters through the specimen, it appears logical to obtain data as close to the point of failure as possible. *In situ* synchrotron X-ray computed tomography is a unique tool to provide data to inform and validate micromechanical tensile failure models, for example, the fibre bundle model developed by Swolfs et al. [3] has been used to compare previous experimental results to the modelling predictions. Although fibre strength variation, random fibre packing and matrix micro-cracks are some of the aspects incorporated within the Swolfs model, the presence of clusters of more than three fibres was highly underestimated indicating that such models may not capture fully the relevant physical mechanisms behind fibre breaks accumulation.

As such, the current work focuses on detailed characterization of local phenomena that may contribute to cluster break formation. In particular, how aspects of local fibre packing may or may not influence this key step in the progression toward final failure.

2. Methodology

2.1. Material

A proprietary medium strength carbon fibre/epoxy resin was studied in a $(90_2/0_2)_s$ layup. Double-edge notched coupons were realised via waterjet cutting, as indicated in Wright et al. [6], see Figure 1a. Thickness of the coupons was of 1 mm. Aluminium tabs were carefully glued to ensure alignment of the sample during tensile tests in the electro-mechanical loading rig.

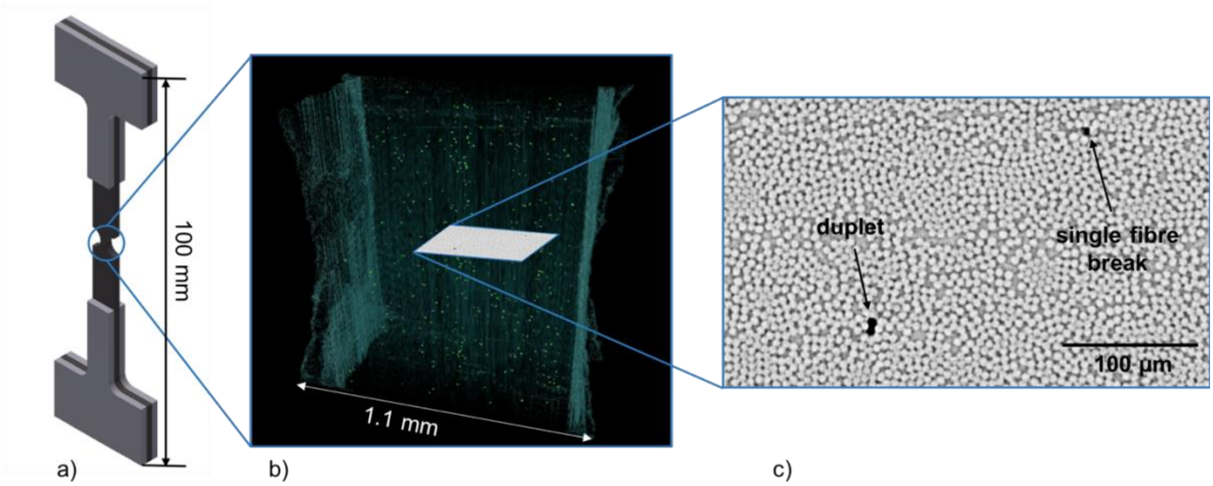

Figure 1. a) Schematic illustration of sample geometry b) 3D representation of the imaged volumes for all the scans c) features detected within the volume (single fibre breaks or group of two breaks, 'duplet').

2.2. Experimental procedure

In situ tensile tests were performed at the European Synchrotron Radiation Facility in Grenoble, France (ID19 microtomography beamline). Voxel resolution provided by the optics in this study was 0.65 µm (sufficient to observe individual fibre breaks) with a field of view of ~ 1.3 mm, see Figure 1b in which a 3D representation of the imaged volume for all the scans is shown. Exposure time was of 50 ms and the number of projections 2996, resulting in ~ 2.5 min per scan.
A modified DEBEN CT5000 tensile stage proven in previous studies ([7]) was used to carry out the tests. Crack opening due to fibre breaks was well in excess of the voxel size for all the collected volumes of scans. Samples were scanned at ~ 50% and ~ 80% of the nominal average ultimate tensile stress (UTS), with at least ten more scans being collected between ~ 80% the nominal average UTS and failure of the coupon.

2.3. Image analysis

3D volumes were analysed using ImageJ™ and MATLAB™. Automatic techniques were developed to analyse the large amount of data collected during the synchrotron test campaign. The data is first loaded in 32-bit version in ImageJ™, where all the volumes are converted to 8-bit format, cropped to smaller dimensions including the central zero degree layers only (the focus of the analysis). To accurately track specific regions of the samples during loading, a feature in the sample is recognised and indicated by the user (*e.g.* a particular pattern drawn by highly misaligned fibres seen in a top view in a resin rich

region). From this, all the scans are aligned, to ensure all measurements from a sample pertain to the same fibres at the same exact location.
Once all fibre breaks are recorded and labelled, a script catalogues singlets and multiplets, based on their morphology. Given the somewhat lower number of planar multiplets observed (~ 3 to 5% of total breaks) and their complex geometry, a final visual inspection of these sites is left to user to visually confirm the automated measurement of the number of fibres associated with a given site.
A MATLAB™ script loads the last collected volume of scans prior to failure and extracts for each fibre break site a subvolume centered of the break that includes some ten to fifteen fibres surrounding the broken one, ~ 60 µm long. Figure 2 illustrate the distribution of sub-volumes extracted from on scanned volume (transparent box). The same script also generates sub-volumes around duplet locations as well as at random <u>*intact*</u> locations, separate from break location and from each other to be reasonably uniformely distributed through the whole volume. Measurements at the intact locations are used to characterise as the 'background' microstructure of the material.

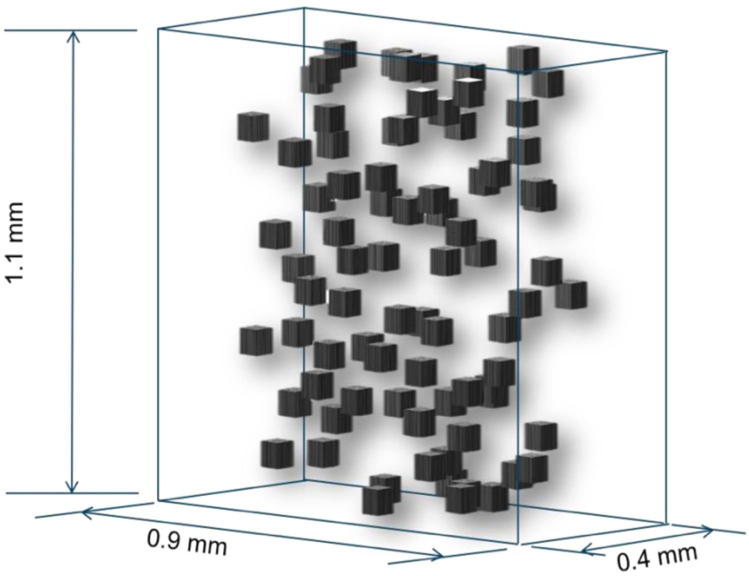

Figure 2. Sub-volumes extracted from the SRCT main scans with dimensions of the scanned region for one of the two analysed coupons. A script developed on MATLAB randomly locates the centres of the sub-volumes which do not contain any fibre break locations, and which have enough volume-to-volume distance to ensure the whole scanned volume is considered.

Within the sub-volumes, the approach used to detect the nearest-neighbouring distance is shown in Figure 3. Centres of the fibres are detected by image enhancement and extracting local maxima. Centres are detected for multiple slices to identify the position of the fibres on a plane below the failure plane. Distances between the fibre centres are then evaluated for each sub-volume.

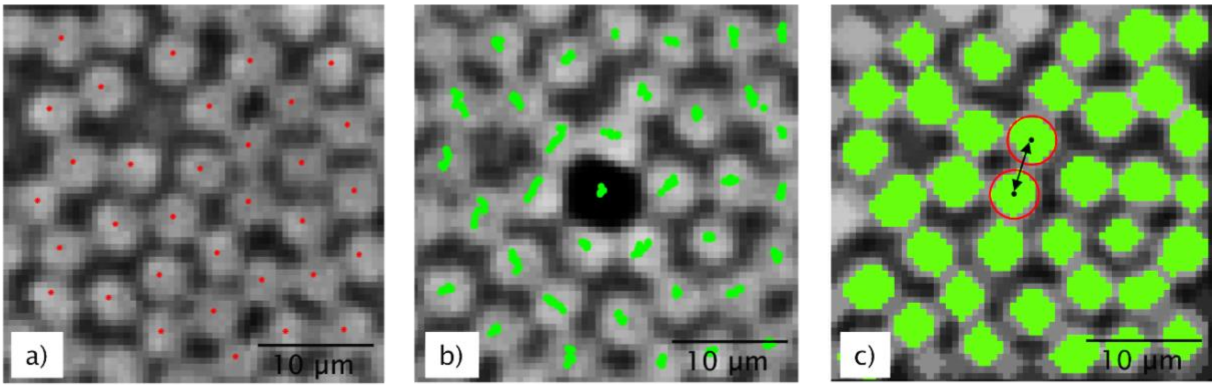

Figure 3. a) Centres of the fibres are detected a few microns below a specific failure point, b) centres of the fibres are to identify the associated broken fibre and the immediately surrounding fibres locations, c) fibres segmented on same plane as a) and the nearest-neighbour of the broken fibre is obtained.

3. Results and discussion

Image analysis is shown here for two representative coupons made of the same composite material, extracted from a same as-manufactured panel, for which damage progression was analysed and all fibre breaks identified.

Within a volume of ~ 0.5 mm^3, the quantification of fibre breaks on approximately 8900 fibres has been carried out. Two different break clusters patterns have been observed. Figure 4a-b indicates planar clusters and diffuse clusters. In this study, focus is given to the planar clusters, which were considered as groups of fibre breaks having a distance between each other equals to zero or less than a fibre diameter, where diffuse clusters are those with an axial distance wider than that but less than the ineffective length [3,4].

Figure 4c-f shows example clusters, where it was clearly seen that a cluster could arise in locally high volume fraction regions, but also in more complex 'mixed' volume fraction regions, where break clustering may follow a local line of near fibres. The statistical analysis of near- and nearest-neighbour distances is then required to discern local microstructural influence.

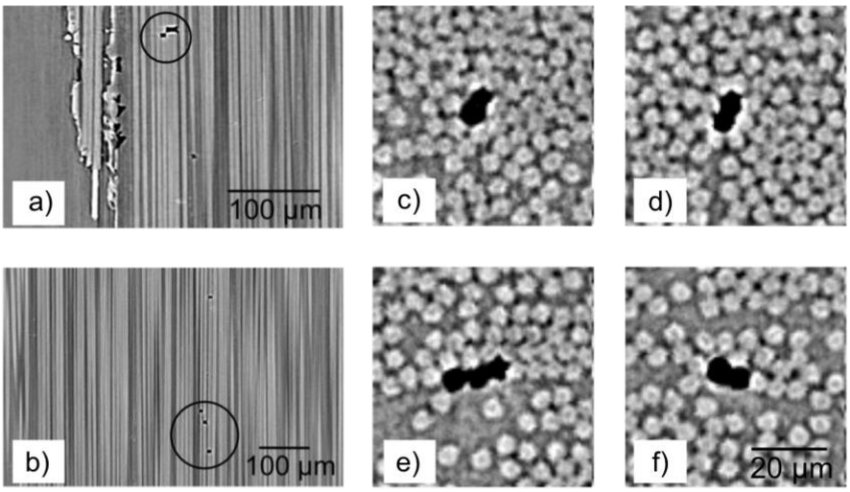

Figure 4. a) Example of a planar cluster b) diffuse cluster c-d) fibre break clusters in high volume fraction region e-f) in 'mixed' volume fraction region.

Fibre breaks accumulated mostly in the form of singlets, which kept increasing in number until the final failure. New fibre breaks appeared in new locations when increasing the load, rather than accumulating around pre-existing breaks. Figure 5 shows an example of the very few observed cases of the growth of a cluster between: a) shows the load step at ~79% coupon UTS and b) ~94% of the coupon UTS, in which a single fibre is seen becoming a duplet. Figure 5b also shows that the broken fibres are twisted and close to a resin rich region. However, only two cases have been observed of this phenomenon: overwhelmingly, break clusters do not grow under progressive loading, as reported in [3–5].

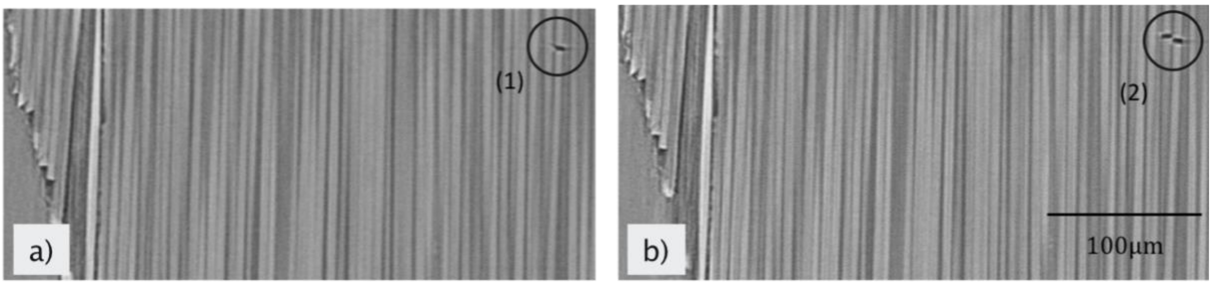

Figure 5. Highlighted in black circles, a) singlet at ~79% coupon UTS observed becoming a duplet in b) at ~94 coupon UTS. b) also shows broken fibres to be twisted and close to a resin rich region.

Fibre breaks do appear to occur in closely spaced neighbours. Measurement of fibre-to-fibre separation within planar broken pairs was found to be ~ 1.1 fibre diameters, i.e. matrix separation of the order of ~ 10% fibre diameter. The only case of a larger nearest-neighbouring distance detected between broken pairs is shown in Figure 6 where a distance of 1.65 fibre diameters has been recorded.

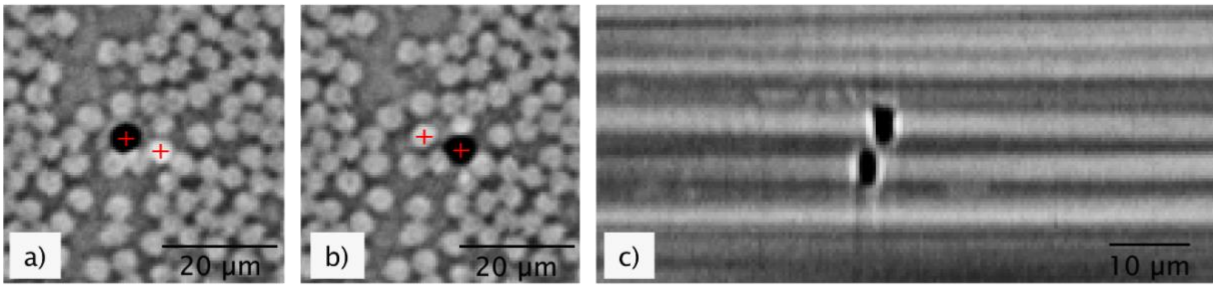

Figure 6. Example of a duplet with the largest distance occurring between the two fibres involved in the failure: a) and b) in a top view of the fibres, at ~ 3 μm of distance and c) lateral view of the duplet.

Load shedding behaviour would intuitively indicate that clustering should indeed occur most readily in between close neighbours (i.e. load magnification at an intact fibre will be greater when the fibre is closer to a break). In a well packed region, it may however be expected that the load shed by a single break would be more uniformly distributed between many neighbours, whilst (in the extreme) a fibre break with just one close neighbour fibre would lead to disproportionate loading of the neighbour due to the limited load capability of surrounding matrix.
The data collected for the tested coupons cut from a same CFRP panel has been combined and in Figure 7 the histograms of the nearest neighbouring fibre distance are shown. In the same figure, normal distribution fits are overlayed. For the singlet and the intact locations, ~ 600 values have been collected, whilst for the duplets, due to the low percentage of appearance, a sample of twentyfive site is analysed.

For the three cases, the mean nearest neighbouring distance value is ~ 1.1 fibre diameter, with a very few cases of fibres having their nearest neighbour farther than 1.5 fibre diameter, meaning that locations with more isolated fibres are not common but the great majority of fibres tend to have a very close fibre even in lower packed regions.
For a same fibre volume fraction (which in this study is of 50%), same fibre diameter and a 2D hexagonal packing the value for the nearest neighbouring distance would have been of 1.35 fibre diameter, higher than the value observed in this study. This connects to what reported by Swolfs et al. in [8]: compared to square and hexagonal packings, random fibre packings can lead to much higher maximal stress concentration factors, as these packings have fibres closer to the broken fibre.
As it can be deducted from the graph, planar duplets tend to prefer closely adjacent fibres to form. A two-sample Kolmogorov-Smirnov (KS) test has been used to compare the three distributions of data, being a nonparametric method to compare samples, sensitive to differences in location and shape of the samples cumulative distribution functions [9]. The two distributions of singlets and intact locations have been compared showing no evidence of difference between the two distributions. The KS test has been performed for the case of duplets-singlets and duplets-intact fibres, indicating the samples are drawn from a same distribution. As such, no statistical difference can be observed between the analysed cases. However, as part of the future work, a higher number of observations for planar duplets will be collected and analysed to provide stronger evidence of the phenomenon.

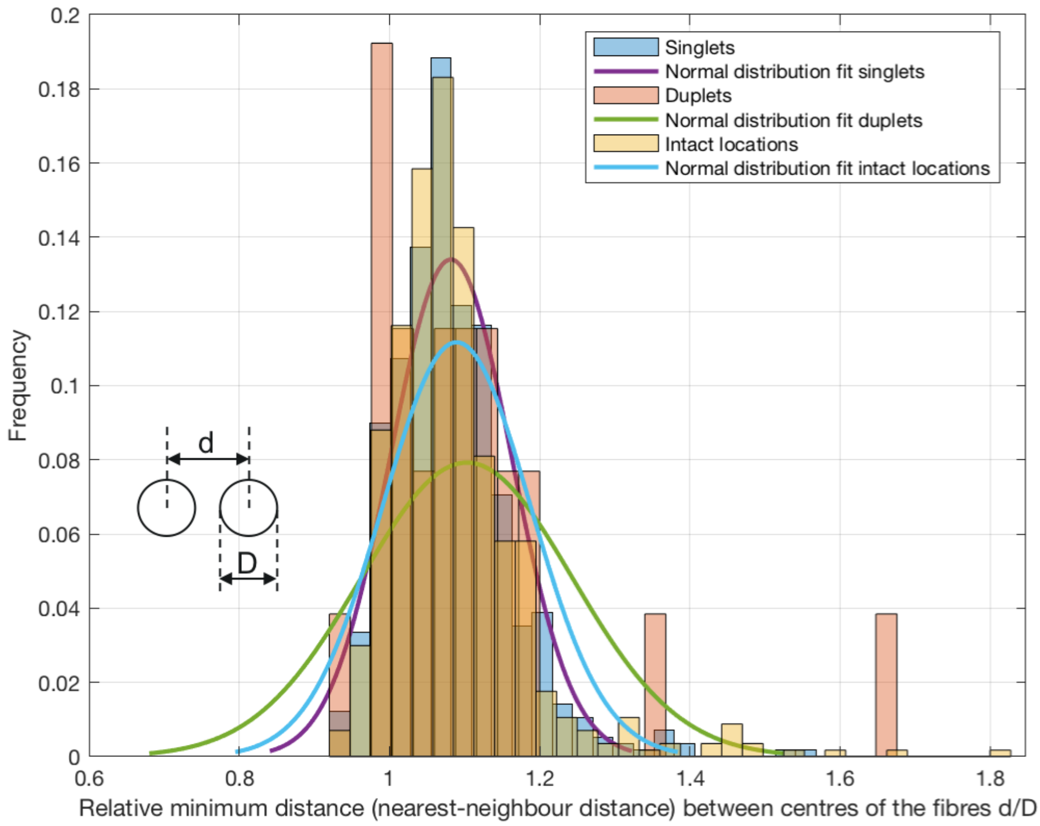

Figure 7. Histogram of the relative distances between two broken fibres recorded a fibre diameter below the failure plane (duplets); relative distances between a broken fibre to its nearest neighbouring intact fibre (singlets); closest distance between random intact fibres (intact locations). Values are obtained from two coupons with the same material specification, tested at the same conditions.

3. Conclusions

In situ X-ray computed tomography is used to follow the damage progression in CFRP coupons with stacking sequence $(90_2/0_2)_s$. A quantification of fibre breaks accumulation has been performed showing the rapid progression of appearance of single fibre breaks prior to failure, according to previous studies [3–5]. Planar cluster accumulation does not show cluster growth prior to failure, after the formation of the first clusters when ~ 90% coupon UTS is reached. A nearest neighbouring distance analysis has been carried out, showing great majority of fibres having their closest fibre at a distance of ~ 1.1 fibre diameter for both intact and singlets locations. This value is higher if compared to what obtained with hexagonal packings of fibres with same volume fraction and fibre diameter, meaning that random fibre packings should be preferred for an accurate representation of the stress redistribution after fibre failure. A similar distance evaluation has been performed for the duplet case, for which the distance between the two broken fibres is evaluated. From the amount of duplets observed in this study, it is seen that fibres involved in a duplet prefer to break in a nearest neighbouring fashion, being the nearest neighbouring distances for duplets, singlets and intact locations, drawn from the same distribution, confirmed by a two-sample Kolmogorov-Smirnov test [9] performed on the three groups of data.

Acknowledgments

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