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In situ Fibre Fracture Measurement in Carbon-Epoxy Laminates using High Resolution Computed Tomography

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Abstract

High resolution Synchrotron Radiation Computed Tomography (SRCT) has been used to capture fibre damage progression in a carbon-epoxy notched [90/0]_s laminate loaded to failure. To the authors knowledge this provides the first direct *in situ* measurement of the accumulation of fibre fractures for a high performance material under structurally relevant load conditions (*i.e.* fractures within the bulk of an essentially conventional engineering laminate). A high level of confidence is placed in the measurements, as the failure processes are viewed internally at the relevant micromechanical length-scales, as opposed to previous indirect and/or surface-based methods. Whilst fibre breaks are the dominant composite damage mechanism considered in the present work, matrix damage, such as transverse ply cracks, 0° splits and delaminations, were also seen to occur in advance of extensive fibre breaks. At loads where fibre break density levels were significant, splitting and delamination were seen to separate the central 0° ply in the near notch region from the 90° plies. Fibre breaks were initially observed in isolated locations, consistent with the stochastic nature of fibre strengths. The formation of clusters of broken fibres was observed at higher loads. The largest clusters

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observed consisted of a group of eleven breaks and a group of fourteen breaks. The large clusters were observed at the highest load, at sites with no prior breaks, indicating they occurred within a relatively narrow load range. No strong correlation was found between the location of matrix damage and fibre breaks. The data achieved has been made available online at www.materialsdatacentre.com/ for ongoing model development and validation.

Keywords: A. Carbon fibres, A. Polymer matrix composites (PMCs), B. Fracture, C. Stress transfer, Synchrotron Radiation Computed Tomography

1. Introduction

Composite materials are known to display complex failure modes, typically involving multiple interacting damage processes, occurring over various length-scales. Fibre breaks occur in the micrometre range, whereas delaminations may occur over millimetre to metre scales, depending on the structure's size and geometry. In tension, the failure of fibres is commonly a critical damage mechanism, with the strength of an axially loaded unidirectional composite being dominated by fibre strength [1].

A range of predictive models have been developed and adapted to simulate the processes that lead to ultimate failure in fibre fracture dominated situations. These generally include the statistical nature of fibre strengths [2],[3],[4],[5] considering for example a Weibull probability function [6], calculation of the stress transfer coefficient around a fibre break [7],[8],[9],[10],[11] and the evolution of critical cluster sites consisting of multiple adjacent or otherwise interacting fibre failures, prior to the onset of unstable failure [12],[1],[13],[14]. Experimental evidence to validate the underpinning assumptions of fibre failure-based strength theories is limited. Previous micromechanical analyses have exploited a variety of techniques to capture damage initiation and progression events in loaded composite components, such as optical

microscopy [15, 16], scanning electron microscopy [17] and acoustic emission [16, 1], along with sample preparation methods such as acid digestion [16]. Ultrasound inspection, such as C-scan, has also been used to detect larger damage mechanisms such as delamination [18], however the resolution capabilities prevent the detection of damage such as fibre fracture. These established experimental methods can be seen to be of restricted utility, having one or more of the following constraints: destructive, time consuming, limited spatial resolution, surface-based or mechanistic ambiguity. For example, optical and electron microscopy may offer high spatial resolution for failure event observation, but can only be carried out at surfaces, requiring either a sectioning-based *post-mortem* approach (losing temporal understanding), or *in situ* analysis under a non-representative stress state (i.e. free surface as opposed to within bulk material). Polishing and cutting techniques commonly required for microscopy can also induce artefacts which may be mistaken for features of interest.

High-resolution X-ray Computed Tomography (CT) has gained considerable interest as a powerful tool for mechanics of materials studies [19]. Advances in the last ten years now provide for relatively routine non-invasive 3D imaging in the micrometer and sub-micrometer range within solid objects. This method is gaining importance in engineering and materials science to determine the internal characteristics of materials and their behaviour [20]. In the case of fibre reinforced composite studies, CT has been used to identify material characteristics such as fibre orientation and volume fraction at the sub-ply level, and failure mechanisms, such as ply cracks [19],[21],[22],[23] and fibre fractures [14],[24].

In this work, high-resolution SRCT results are presented for commercial carbon/epoxy cross-ply laminates subject to *in situ* loading to failure. The

images obtained enable the major damage mechanisms to be identified and quantified for the sample scale and geometry used. With the present work particularly focused on individual fibres breaks, with micro-mechanistic behaviour then being captured in an unambiguous and mechanically representative way that has not been reported to date.

To the authors knowledge only one previous study has assessed fibre fracture for a polymer matrix composite (Aroush et al. [14]), using *in situ* SRCT of a loaded quartz/epoxy 'model' composite. Small three dimensional bundles (125 fibres) were used to determine a lower-bound prediction of the critical cluster size that results in final catastrophic failure. The present study considers a significantly larger composite volume, of approximately 15,000 fibres, of a commercial carbon/epoxy laminate.

2. Experimentation

[90/0]_s laminate plates were fabricated from Hexcel HexPly M21 T700 carbon fibre pre-preg (nominal fibre volume fraction of 60%) to the manufacturer's specifications. Double edge notched tensile samples were prepared via waterjet cutting, as reported by Wright et al. [21], see Figure 1.

A simple screw driven load frame was used to incrementally load samples to failure *in situ* with a gauge length of 66mm. The average ultimate tensile failure stress, σ_{uts} , was found to be 910MPa across the notch section (based on four specimens). Tests were carried out at the ID19 beamline at the European Synchrotron Radiation Facilities (ESRF) in Grenoble. They were scanned prior to loading and then at incrementally increasing stress levels ranging from 30% σ_{uts} to the point of final failure.

The optics used in this study provided an isotropic voxel resolution of 1.4 μm , with the sample-detector distance being set to 37mm, falling within the near-field Fresnel diffraction regime (edge detection) with a beam energy of

20kV. 2D radiographs were recorded at 1500 angular positions over 180° of rotation (parallel beam conditions) via a 2048x2048 detector system. Further details of the imaging process are provided in Wright et al. [21], whilst Figure 2 shows representative results obtained. Figure 2 a) shows a 3D image of the notched region, in which the composite material has been digitally removed to show segmented 0° splits and transverse ply cracks, b) and c) show the corresponding slices.

3-D volumes were analysed using the commercial package VG studio Max v2.1 and features of interest were identified and segmented. Semi-automatic techniques were used to analyse cracks and fibre breaks. For cracks, an approximate region of interest was drawn around the feature to be segmented, an automatic 3D seed growth tool was then used to capture more precisely the geometry and location. The 3D tool segments voxels by adding all adjoining points of a defined seed point, that are within a tolerance with respect to the mean gray value of the selection. Figure 3 shows all segmented matrix damage in three dimensions at the eight load increments up to 80% of final failure. Fibre breaks were individually detected by the distinct bright fringes generated by phase contrast. To ensure fibre breaks are not mistaken for voids or imaging artefacts, orthogonal planes were carefully inspected, as shown in Figure 4. For all load levels fibre failures were judged to be quite clear, with average crack openings well in excess of the voxel size. Average fibre break opening distances of $2.28\mu\text{m}$ were identified for loads of 64, 85 and 95% of final failure.

3. Results and Discussion

Thorough image analysis was carried out for a representative sample at multiple load steps up to 94% of the final failure load. The damage progression was analysed and all cracks and fibre breaks were identified and segmented. Figure 5, shows cracks and fibre breaks in three dimensions at the

highest load step (*i.e.* 94% of final failure), with the actual material bulk being rendered partially transparent.

Previous processing by Wright et al. [25] (Figure 3) shows that transverse ply cracks initiate by 20% of the failure load, whilst 0° splits initiate by $\sim 40\%$ and large delaminations by $\sim 60\%$. It is important to note that these cracking events occur in advance of significant fibre breaks, with Figure 6 showing a power law curve for the accumulation of fibre breaks with increasing load within the 0° ply. The data is presented as a function of the nominal fibre stress, σ_{fibre} , determined by the ply stress, σ_{ply} , and simple rule of mixtures calculation.

Beyond a fibre stress of $\sim 2800\text{MPa}$ ($\sim 70\%$ of failure), splitting and delamination have largely separated the central 0° ply in the near-notch region from the surrounding 90° plies, shown in Figure 3. Quantification of the fibre break data has been carried out within this near-notch volume of $\sim 0.7\text{mm}^3$, of length 1.8mm, containing approximately 9800mm of fibre.

3.1. Neighbouring fibres breaks

A study was carried out to estimate the ineffective length of the fibres, to enable the quantification of neighbouring fibre breaks. Measurements were taken within the bulk composite, of the distance between fibre breaks that occur along the axis of the same fibre. For the highest load case available, this was identified at five locations, where the distance along the axis of the fibre between the two breaks ranged from $335\mu\text{m}$ down to $70\mu\text{m}$ (with an average distance of $150\mu\text{m}$): the closest of the fibre breaks can be seen in Figure 7.

The spacing between breaks has been used to give an estimate of the ineffective length, *i.e.* the distance along the fibre axis from the break, where the uni-axial stress increases from zero to the far-field applied stress. Table 1 shows a comparison of ineffective lengths δ , from this data and other authors,

determined experimentally and theoretically using the following equation from Curtin [26]:

$$\delta = \left(\frac{\sigma_0 r L_0^{1/m}}{\tau} \right)^{m/(m+1)} \quad (1)$$

Where r is the radius of the fibre ($3.5\mu\text{m}$), m and σ_0 are the Weibull modulus and characteristic strength (determined from the experimental data, shown in Section 3.2), τ is the frictional sliding resistance between the fibre and matrix (81.2MPa is used from [27] in this study) and L_0 is the gauge length of the fibres. Curtin [26] notes that σ_0 is the stress required to cause one fibre failure, on average, in a fibre length L_0 : in this work an arbitrary choice of 66mm (corresponding to the sample length) has been used, it is also representative length used for single fibre tests.

Table 1 shows that theoretical values of the ineffective length are of the same order as the experimental values. The upper bound ineffective length suggested in this study ($335\mu\text{m}$) compares well with the theoretical results ($399\mu\text{m}$). However the minimum value ($70\mu\text{m}$) is apparently lower than in the other previous experimental studies and the calculated theoretical value. It was noted that the incidence of double breaks, that were observed on the same fibre, did not occur close to other fibre breaks, *i.e.* do not appear to be a result of local stress concentrations.

The formation of clusters of fibre breaks was observed and quantified, see Table 2. A cluster was considered as two or more broken fibres in neighbouring fibres, with an axial separation (*i.e.* along the fibre direction) of less than the ineffective length, using the lower bound estimate of $70\mu\text{m}$.

Figure 8 shows a histogram of the axial distance between 45 different neighbouring fibre breaks: it is seen that the majority of neighbouring breaks occur with an axial separation between 0 and $2.5\mu\text{m}$. This is further

illustrated by Figure 9, in which clusters of neighbouring fibre breaks can be seen to lie close to the same plane.

A code was written to compute the Euclidean distance between all fibre breaks. The distance from each fibre break to its nearest neighbouring fibre break (denoted the minimum distance) is then recorded, the minimum distances were arranged in a square array to avoid double counting (*i.e.* distance from break a to break b, versus the distance from break b to break a). Figure 10 shows the frequency of nearest neighbour distances at the highest load case, in which the mean value of the nearest neighbour distance is $\sim 28\mu\text{m}$. A significant proportion of fibre failure was seen to occur at separation distances of less than $\sim 8.6\mu\text{m}$, corresponding to the average inter-fibre distance for a fibre volume fraction of 60% and fibre diameter of $7\mu\text{m}$. The mean of the nearest neighbour distances was calculated at $40.4\mu\text{m}$. The R-ratio [28] approach has been used to assess the deviation of this value from the mean nearest neighbour of a volume with spatial randomness. In this approach, different types of spatial distributions are classified as $R=1$ for random, $R<1$ for clustered and $R>1$ for uniform. For the highest loaded sample a R-ratio of 0.58 was calculated, consistent with a clustered distribution. When taking into consideration only the single fibre breaks at the highest load case, a R-ratio of 0.89 was calculated, showing the singlets distribution is closer to a random pattern.

The two largest adjacent fibre clusters, observed at the highest load step of 94% of failure, consisted of a group of fourteen breaks, as shown in Figure 11, and a group of eleven breaks. The accumulation of breaks with load was not observed for most large clusters (5 neighbouring breaks or more): the previous load step (88% of failure) showed no breaks at the same locations. One larger cluster (of 6 neighbouring breaks) was seen to accumulate from a 1plet (at

88% of failure) into the 6plet (at 94% of failure). Phoenix and Beyerlein [29] gives a comprehensive review of shear lag models and discusses the static and dynamic overloads of Hedgepeth [9]. The dynamic overload produces significant increases in the stress concentration factor (SCF) on intact fibres neighbouring a fibre break, for instance for a 2D planar array of parallel fibres with one fibre break, the dynamic SCF is 1.53 versus 1.333 in the static case. This increased SCF in the dynamic case will then increase the probability of neighbouring fibres to break, and potentially could be the cause of an isolated break to cascade into a sequence of breaks, as observed here. No correlation was evident between the location of cracks in the 90° plies and fibre breaks in the 0° ply, indicating that transverse ply cracks are not a significant influence on fibre failure in this instance. This is contrary to the previous findings of Wright et al. [19], in which a correlation was found in an untoughened system between fibre breaks in the 0° ply of a [90/+45/-45/0]_s notched laminate at the -45°/0° interface, where a concatenated series of breaks aligned with the intralamina damage in the -45° ply was clearly observed. No dominant correlation of fibre failure locations and other microstructural features, *e.g.* voids and resin rich regions, has been found, however, quantitative assessment is ongoing.

3.2. Comparison to previous work

Figure 12 shows a Weibull plot of the fibre break data shown previously in Figure 6, in which the cumulative distribution function is given as:

$$P_f = 1 - \exp(-(\sigma/\sigma_0)^m) \quad (2)$$

Where P_f is the probability of failure, defined as the number of fibre breaks/ the total number of fibres, σ_0 is the characteristic stress and m is the Weibull modulus. Least squares has been used to fit the plot in Figure 8, giving an

estimate of σ_0 of 6.25GPa and m of 8.44, based on the lower tail of the distribution. Table 1 shows a comparison of Weibull parameters, σ_0 and m , reported for carbon/epoxy determined in other studies.

Figure 13 shows a comparison of previous experimental studies which measured fibre break numbers with increasing load. The extent to which differences in fibre break accumulation varies with material or test method cannot be separated straight forwardly. In the first instance it can be seen that trends are of a similar order. However it is evident that the 2D laminate approach of Rosen [2] (*in situ* loading of a glass fibre single layer thickness composite) and 2D optical microscopy method of Marston et al. [15] show a higher break density than the other methods. The higher break densities observed in the 2D laminate of Rosen [2] may be explained by the higher load transferred to the neighbouring fibres (due to fewer neighbouring fibres in 2D laminates versus 3D laminates). The results from the optical microscopy method of Marston et al. [15] may in part be explained by the destructive methodology (sectioning) inducing more fibre breaks or artefacts. Although a direct comparison between previous experimental methods can not be made it is conclusive that the large scatter in fibre break accumulation illustrates the need for methods which can provide unambiguous measurements of fibre break density. This is particularly shown by Pickering et al. [16], for which there is distinct differences in analyses carried out for the same fibre and composite layup using the two different methods of microscopy and AE. The SRCT based methods used in the present study enable each individual fibre break within the whole sample volume to be located and quantified. The high resolution imaging enables considerable confidence to be placed within the results.

4. Conclusions

CT and subsequent image analysis used in this study have enabled the first detailed quantification of the accumulation of broken fibres in a representative 3D volume of a carbon/epoxy laminate. It has been found that failure of fibres is the dominant damage mechanism controlling tensile fracture stress for the sample geometry tested.

The incidence of fibre break clusters gives evidence of the role of load sharing in the progression to failure, indicating that the failure of fibres can not be described by the Weibull probability function alone. At lower load levels the fibre breaks appear singly and in small clusters (2 neighbouring breaks). At higher load levels larger clusters are formed, in which eight clusters of four breaks or more were found within the volume at the highest load step of 94% of failure: the larger clusters appear to occur within a narrow stress range, as opposed to accumulating with increasing load. The spatial distribution of all fibre breaks were found to occur in a clustered pattern, however when considering only single fibre breaks, the distribution is closer to that of a random pattern.

The data analysed in this work is available online [30] to facilitate more extensive exploration of the data. We particularly encourage the modelling community to exploit this opportunity to validate and compare the capabilities of available models.

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	Fibre	L_o mm	Type	Weibull modulus	σ_o MPa	Exp. δ	Theory δ
Scott <i>et al.</i>	T700G	66	Notched laminate	8.44	5054	(min)70 (ave)150	399
Deng <i>et al.</i> [27]	T700S	50	Single fibre	4	5800	620 ¹	638
Beyerlein & Phoenix [31]	AS4	10	Single fibre	4.8	4493	-	-
			4 fibre micro- composite	LT 15 UP 10	LT 3890 UT 4000	-	LT 90 ² UT 120 ²
Marston <i>et al.</i> [15]	M40B	20 fibre	Single	6.9	2864	-	-
Pickering <i>et al.</i> [16]	Tenax HTA	130	Tow 6K	15	1790	200 ³	295
		150	Tow 12K	7.37	3240 - 3480	-	-

Table 1: Comparison of estimated Weibull parameters and ineffective lengths with previous work.(LT = lower tail, UT = upper tail)

1. Single fibre fragmentation 2. Calculated in [31] 3. laser Raman spectroscopy

% of Ultimate Failure	28	64	70	80	85	88	94
1plet	1	5	12	22	92	100	144
2plet		3	5	6	7	18	29
3plet					3	1	11
4plet						1	3
5plet							1
6plet							1
7plet							1
11plet							1
14plet							1

Table 2: Increasing cluster formation with load. Where an N-plet consists of N, number of adjacent fibres breaks with a maximum axial separation of $70\mu\text{m}$

Figure 1: a) Notched sample geometry b) dimensions of the sample c) complete sample arrangement, with adhesively bonded aluminium end tabs. [21]

Figure 2: Representative SRCT imaging of matrix damage [21]: (a) 3D image of notched region, bulk of the composite digitally removed to reveal the damage (in red). (b) 2D slice showing a 0° ply split, and (c) transverse ply crack.

Figure 3: Extraction and segmentation of matrix damage: *in situ* loading from 20% to 80% of failure load, composite has been made partially transparent. Transverse ply cracks in the 90° plies, 0° splits extending from the notch, and delamination occurring at $90/0$ interface

Figure 4: a) Axial and b) longitudinal views of the same fibre break.

Figure 5: 3D segmented damage in sample at 94% of failure, in which the composite is rendered partially transparent.

Figure 6: Accumulation of fibre breaks with increasing load. Error bars of average error (in%) from three manual recounts of the data.

Figure 7: Example of fibre breaks occurring along the axis of the same fibre.

Figure 8: Measured axial separations between neighbouring fibre breaks, with images below illustrating these axial separations

Figure 9: 2D slices of examples of closely co-planar adjacent fibre failures, (a) cluster of 5 fibre breaks, (b) cluster of 14 breaks, (c) cluster of 3 breaks.

Figure 10: Distribution of the nearest neighbour distances between fibre breaks

Figure 11: Largest cluster of 14 fibre breaks, (a) and (c) 2D slices at different planes (b) 3D image, (part of composite made transparent to reveal cluster)

Figure 12: Weibull plot of fibre breaks versus fibre stress.

Figure 13: Comparison of accumulation of fibre breaks in loaded composites with existing methods

Highlights

- Notched $[90/0]_s$ carbon epoxy laminates loaded *in situ* to failure
- Synchrotron radiation computed tomography (SRCT) used to capture damage progression
- Non destructive method allows all fibre breaks and matrix cracks to be identified
- Accumulation of fibre breaks with load analysed
- Providing data for which existing models can be informed and validated