A Three Dimensional Damage Characterisation of Composites
Loaded in Tension-Tension Fatigue: A Laboratory X-ray Computed
Tomography Investigation

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Abstract
This study examines how 3D X-ray computed tomography (XCT) can be employed to analyse damage in fatigue tested glass-fibre reinforced polymer (GFRP) composites. Particular attention is paid to the detection of resin microcracking by using a dye penetrant as a contrasting agent, which increased the sensitivity to crack to sub-voxel size. An innovative data processing methodology has been developed to investigate and assess the effect of reinforcement structure in terms of fibre orientation on the damage induced by fatigue. The relationship of matrix-cracking (intra-laminar) and bonding-cracking (inter-laminar cracking) to the geometry is presented and discussed.

Keywords: GFRP, composite, fatigue, X-ray tomography, staining, 3D damage characterisation, matrix-cracks, bonding-cracks.

1 Introduction
Fibre reinforced polymer composites (FRPC) are widely used in engineering applications due to their high specific strength and high specific modulus. For example, wind energy is a fast growing market worldwide where composites have gain a special attention, particularly GFRP \cite{1}. Wind turbines are subjected to constant and variable amplitude loads in service, and thus fatigue-durability of the composite is an important requirement to be investigated. Prediction of composite failure under cyclic loading would enable to the mitigation of catastrophic failure.

Due to the anisotropic nature in composite strength and stiffness they exhibit very complex failure mechanisms under fatigue as well as static loading \cite{2}. The failure mechanisms recognised under fatigue loading are; matrix cracking, delamination, fibre-matrix interfacial debonding and fibre failure.

Initial understanding of the mechanical behavior of a composite is limited to a coupon, where the specimen geometry and size may influence the damage mechanisms and the ultimate failure. Two such factors that have to be taken under consideration are free-edge \cite{3} and end effects. The magnitude of the effect from free-edge depends on the specimen width, while the magnitude of the end-effect depends on the test gauge length. Matrix-cracking may be produced at the free-edge due to large stresses at the interface. These matrix-crackings then propagate to the laminate creating delamination and ultimately cause premature composite failure. Therefore, the free-edge effect is more important for narrower specimens. As the ultimate failure of the composite initiates at the free-edge this is very important in fatigue failure. Many studies have been carried out to understand this free-edge effect \cite{3-5}. 

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A further factor governing the matrix-cracking is yarn/tow crimp within the textile preform structures. For composites with woven structures a tensile load cannot be directly transferred to the reinforcement due to the presence of yarn crimp, unlike those with unidirectional fibres. Once the structure reaches a critical load, matrix-crackings are initiated along the transverse (or weft) reinforcement (see Figure 1). These cracks then propagate as the axial tows starts to straighten up, while the transverse tows increases the crimp allowing this movement. Therefore, understanding the relationship between the crimp angle and crack propagation will inform future design modifications.

Matrix damage is the first mode of failure in continuous fibre/polymer composites, under a load, which is the weakest point of the composite [6]. This can be both matrix-cracking and fibre/matrix debonding. Fibre/matrix property mismatch results in matrix-cracks, which generally follow the fibre orientation. A typical crack followed by bonding cracks [7] is shown in Figure 1. Once a crack propagates and reaches an interface of two fibres oriented in two directions bonding-cracks (delamination) may occur. Modulus mismatch of these two fibre bundles also results in bonding-cracks/delamination.

Figure 1: The schematic diagram of connectivity of tow splitting (matrix-cracking) to bonding-cracks. b-y is the binder-yarn, w is the weft, i is the matrix-crack, and ii is the bonding-crack between binder-yarn and weft.

Overall, the complexity in the damage mechanisms has made accurate predictions for component lifetime particularly challenging. Studies have made attempts to understand the above relationships by means of optical or scanning electron microscopy (SEM) [8]. However, these techniques provide 2D information on a limited number of cross-sections that are prepared. Moreover, further damage is induced during the sample preparation, such as sectioning and polishing. Recently, XCT has been shown to provide a new means of characterising composites in 3D [9] to understand in depth, these links between structure and component failure behavior. However, the level at which the cracks can be resolved is key as to how accurately the damage can be captured and it has been shown that sub-voxel accuracy can be obtained by staining [10]. The detected damage/features and structure then can be analysed through advanced data processing [11] to correlate the damage to the reinforcement structure.

In this proof of concept study, the damage mechanisms of a tensile-fatigued 3D composite are investigated in 3D by combining XCT with a novel image processing methodology.

2 Experimental Details

2.1 Material manufacturing

A modified layer-to-layer interlock 3D woven preform (see Figure 2) of S2 glass fibre tows was infused with Araldite LY 564 resin and XB3486 hardener and cured in an oven at 80 °C for 8h.
2.2 Tension-tension fatigue tests

Tension-tension (T-T) fatigue testing was performed using an Instron 8802 testing machine. The specimen (width of 16 mm, thickness of 1.6 mm and a gauge length of 30 mm) was tested in load control at room temperature. The cyclic loading was applied in a sinusoidal waveform, with a stress ratio (the ratio of minimum stress to maximum stress in one cycle of loading) of 10, and at a frequency of 5 Hz. The fatigue stress level was 40% of the normalised ultimate tensile strength. X-ray CT was carried out on the specimen after 5k fatigue cycles.

2.3 X-ray computed tomography

The composite specimen was scanned at the Henry Moseley X-ray Imaging Facility using the Nikon Metrology 225/320 kV Custom Bay. The system was equipped with a 225 kV static multi-metal anode source (Cu, Mo, Ag, and W) with a minimum focal spot size of 3 μm and a PerkinElmer 2000 × 2000 pixels 16-bit amorphous silicon flat panel detector. The scanning was performed with the molybdenum target using a voltage of 70 kV and a current of 90 μA. The acquisition software was Nikon Metrology proprietary software InspectX (version XT 2.2 service pack 5.5). The data acquisition was carried out with an exposure time of 1000 ms, and no filtration. The number of projections was set to 3142 and the number of frames per projection was 1, resulting in an acquisition time of 1 hour. The test specimen was stained with a dye penetrate (containing 250g zinc iodide, 80ml distilled water, 80ml isopropyl alcohol and 1ml Kodak photoflow) in order to capture the damage with sub-voxel accuracy [10]. The samples were soaked over a period of 24 hours.

The reconstruction was performed with Nikon Metrology proprietary software CT-Pro with corrections applied to reduce beam hardening artefacts and to reduce noise. The entire volume (2000 × 2000 × 2000 voxels) was reconstructed at full resolution with a voxel size of 11.8 μm along the x, y, and z directions.

Visualisation and segmentation of the reconstructed volume was carried out using Avizo 8.0 software (FEI VSG, Burlington, MA). Out of 30 mm gauge length of the specimen, approximately 10.5 mm in length was analysed for this initial investigation. The segmentation strategy adopted was to first segment the air (outside of the composite), the cracks, the yarns and the matrix using grey level thresholds. Then a semi-manual method was used to separate the weft and the binder-yarns (see Figure 3).
The cracks were separated into 3 components; namely cracks in the binder yarn segmentation, cracks in the weft yarn segmentation and cracks in the interface between the binder and weft yarns. This was achieved by first applying the medial axis transform to give the skeleton of the cracks using the Autoskeleton module in Avizo 8 software. The points comprising the medial axis are arranged into segments which are connected together at branch points. Each point is associated with a radius which relates to the largest sphere that fits within the crack at that point. Voxels just outside each sphere surface form the local neighbourhood of the crack, which is used to determine which component of the composite the point is located within. For example, if voxels in the local neighbourhood are labelled as binder yarns, then the point is classified as belonging to cracks in the binder-yarn segmentation. However, some of the crack segments which had a majority of points classified as bonding cracks between binder-yarn and weft were found to contain short sections (e.g. a few pixels long) located within the yarns. This can occur due to imprecision in the manual segmentation of the binder yarns from the weft yarns. Therefore these short sections were reclassified as bonding cracks if they were bounded both sides by bonding cracks between binder-yarn and weft. Finally, the classification was extended to all crack voxels by assigning to each voxel the classification of the closest point on the medial axis.
To investigate the free-edge and end-edge effects, the distances of the cracks from the specimen surfaces were calculated. The specimen boundaries were extracted from the segmentation after firstly smoothing the segmentation to reduce the effects of noise by applying a morphological closing operation with a structuring element being a cube of dimension 9 voxels. The 6 surfaces of the approximately cuboid shaped specimen were then separated by first locating the edge voxels at the corners of the shape. This was carried out by cropping the reconstructed volume to a cuboid enclosing the specimen and then determining which specimen boundary voxels were closest to each of the corners of the bounding volume. The distances of the crack voxels to each specimen surface (left and right edges; top and bottom ends; and top and bottom surfaces as shown in Figure 4) were then calculated using the distance transform.

The cracks in the binder-yarn segmentation were analysed further by grouping together closely spaced cracks into damage regions. This was achieved by applying a morphological closing operation to the binder yarn bonding cracks using a cube of dimension 11 voxels. The angle between the major axis of each damage region and the top surface of the specimen (see Figure 4) was then calculated. Small damage regions (<0.016 mm$^3$) were excluded from the analysis, and the major axis vector was determined from principle component analysis of the crack coordinates in each group.

3 Results and Discussion

Staining has enhanced the detection of matrix cracks and bonding cracks (see Figure 5). In particular, Figure 5 (a), shows that without staining only relatively large isolated cracks are visible due to the limitation of the voxel size. After staining (the same specimen), the damage sensitivity has increased to reveal sub-voxel sized features allowing many more matrix-cracks and bonding-cracks to be detected as shown in Figure 5 (b). Staining also provides sufficient contrast to enable semi-automatic segmentation of the damage.

Figure 5: The enhancement of the cracks due to staining (xz-orthoslices) (a) without staining (b) with staining.

The 3D woven composite in this study showed bonding-cracks connected to matrix-cracks (see Figure 6 (a)) and bonding-cracks between binder-yarns. The initially fully segmented damage was separated into three categories; matrix-cracks in the weft, bonding-cracks between binder-yarns interface and bonding-cracks between binder-yarn and weft interface as shown in Figure 6 (b). The main matrix-cracking are presented in the weft, which is transverse to the test direction. The pulling action due to the tensile-fatigue load caused splitting of the weft, where either matrix-cracking or fibre-matrix debonding occurred. Bonding-cracks (delamination) mainly occurred where the surfaces of two fibre bundle are in contact. This phenomenon has taken place at two distinct locations; at binder-yarn/weft (ii in Figure 6 (a)) and binder-yarn/binder-yarn interfaces (iii in Figure 6 (a)).
Figure 6: Damage presented in the 3D woven composite, after 5k fatigue cycles. (a) yz-orthoslice; \( b \)-\( y \) is the binder-yarn, \( w \) is the weft, \( i \) is the matrix-crack, \( ii \) is the bonding-crack between binder-yarn and weft, and \( iii \) is the bonding-crack between binder-yarns (b) volume rendering of the separated damage relative to the binder-yarn.

The volume rendering of these matrix-cracks in Figure 7 (a) shows that the cracks in the weft follow the fibre direction (weft), which is transverse or perpendicular to the axis of testing. The distribution of these weft matrix-cracks is shown in Figure 7 (b), as measured relative to the end of the specimen (see Figure 4), which is parallel to the matrix-cracks and the wefts. The peaks in the graph are occured in three distinct groups, which correspond to the weft groups in the structure shown in Figure 4. It should be noted that the length of the tested specimen used for the image analysis was approximately 10.5 mm out of the full test gauge length was 30 mm. A thorough investigation of the influence of end-effects on crack distribution would therefore require the complete specimen to be analysed. However, these results indicated that such an analysis is possible using the methodology presented in Section 2.

Figure 7: Distribution of matrix-cracks in the weft, after 5k fatigue cycles; (a) volume rendering of the matrix-cracks relative to the binder-yarn (b) damage volume as a function of the \( y \)-distance from an end of the specimen.

The volume rendering of the separated bonding-cracks located between binder-yarn and weft are shown in Figure 8 (a). Although continuity would be expected in the cracks (as for delamination of 2D
woven or unidirectional composites) a discontinuity can be observed. This would not have been detected through 2D analysis such as optical microscopy or SEM. The damage distribution presented in Figure 8 (b) is approximately symmetrical across the specimen width (16 mm) from one edge to the other. The highest maxima are closer to the specimen edges, which suggest that there is an edge-effect which influences the damage mechanisms of the fatigue tested specimen.

Figure 8: Distribution of bonding-cracks between binder-yarn and weft, after 5k fatigue cycles; (a) volume rendering of the bonding-cracks relative to the binder-yarn (b) damage volume as a function of the x-distance from an edge of the specimen.

The volume rendering of the separated cracks located in the binder-yarns segment is shown in Figure 9 (a). All the cracks having a local neighbourhood of voxels in the binder-yarns have the appearance of bonding-cracks with little evidence for matrix-cracking in the binder-yarns. The presented bonding-cracks showed a similarity to delamination in 2D woven or unidirectional composites. Due to the effect of the modified layer-to-layer interlock 3D woven structure (Figure 2) the binder-yarn has a higher crimp. Also the modification has created interfaces between binder-yarns especially towards the top and the bottom surfaces. During the tensile-fatigue tests, it seems that the attempt of reducing the crimp in the yarn has caused these bonding-cracks. The distribution of this damage volume through the specimen thickness as presented in Figure 9 (b) shows three distinct peaks. This suggests that at three levels through the thickness there are binder-yarn/binder-yarn interfaces. The average angle between the major axis of each of these bonding-crack regions and the top surface of the specimen (see Figure 4) is 12±4 °, which also follows the binder-yarns.

Figure 9: Distribution of bonding-cracks between binder-yarns, after 5k fatigue cycles; (a) volume rendering of the bonding-cracks relative to the binder-yarn (b) damage volume as a function of the z-distance from a surface of the specimen.
4 Concluding remarks
For the first time, X-ray computed tomography was employed to assess tensile-fatigue tested modified layer-to-layer interlock 3D woven composite. The post-tested damage was assessed in 3D, which facilitates the understanding of the damage relative to the structure of the reinforcement. A novel image processing methodology was employed to separate the segmented damage. After the separation of this damage three major categories were observed; matrix-cracking in the weft, bonding-cracks between binder-yarn and weft interfaces, and bonding-cracks between binder-yarns interfaces. This created the possibility of assessing the distribution of different mode of failures relative to the specimen boundaries. Future work will focus on establishing the accuracy of the quantitative assessment through statistical analysis.

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References