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### HIGH RESOLUTION TOMOGRAPHIC IMAGING AND MODELLING OF NOTCH TIP DAMAGE IN A LAMINATED COMPOSITE

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#### Abstract

Synchrotron radiation computed tomography (SRCT) has been used to observe *in situ* damage growth and enable micromechanical damage characterization in [90/0]<sub>S</sub> carbon fibre-epoxy composite samples loaded in uniaxial tension to stresses ranging from 30% to 90% of the nominal failure stress. A 3-D finite element model has been constructed to predict crack opening displacements and shear displacements in the 0° plies resulting from thermal residual stress imposed during autoclave cure and from the application of mechanical load. Of particular interest is the demonstration of SRCT as a technique to enable direct, *in situ*, 3-D, non-destructive damage quantification to assist model development and provide model validation. In addition it has been identified that SRCT has the potential for full field analysis of strain re-distributions during damage growth.

Keywords: Carbon fibres (A), Matrix cracking (B), Thermal properties (B), Finite element analysis (FEA) (C), Synchrotron X-ray tomography.

#### **1** Introduction

Modelling the behaviour of composite materials is far from trivial due to the complex interactions of multiple damage mechanisms, including fibre-matrix

debonding, inclusion-matrix decohesion, inter-laminar delamination, intra-laminar cracking and fibre rupture [1,2]. A large number of models have been able to predict successfully the strength of laminated structures for a particular lay-up configuration or loading regime [3-6], however the inherent assumptions and simplifications within these models usually result in a lack of predictive capability when the material system, lay-up or type of loading is modified [7]. Many current models are only successful in their predictions due to empirical fitting to a particular data set. With its limited physical basis, the fitted model will not predict changes in failure processes (lay-up, load case). A homogenised damage mechanics approach to modelling requires an understanding of the interacting mechanics approach that captures the physics of fracture mechanics is computationally expensive.

This paper has two principal objectives. The primary aim is to demonstrate the potential of synchrotron radiation computed tomography (SRCT) [8] as a technique to observe the *in situ* initiation and propagation of damage in a [90/0]<sub>S</sub> carbon fibre-epoxy composite and to identify the critical damage interactions that lead to laminate failure to assist model development, calibration and validation. Particular damage parameters such as crack opening displacement (COD) and shear displacement have been quantified and used in conjunction with high resolution images of damage evolution to inform the development of a finite element (FE) model of intra-laminar matrix splitting in the 0° plies. The secondary aim is to compare a 3-D FE model with the SRCT results and to determine the extent to which the model of a single observed damage mechanism of a split in a 0° ply has useful predictive capability. In addition, the data acquired from SRCT can be compiled as a library of experimental results that can be accessed for comparison with existing and future model predictions.

#### 2 Materials and Method

Laminated plates of Hexcel HexPly® M21 carbon fibre pre-preg were laid up in a [90/0]<sub>s</sub> configuration with dimensions 300 x 300 x 1 mm and cured in an autoclave to the manufacturer's specifications at a single dwell temperature of 180°C [9]. While the cure process will initiate before this temperature with the accompanying onset of residual thermal stress, this value is considered to represent an upper bound estimate of temperature for the stress free reference state. Double edge notched specimens with the geometry shown in Figure 1 were machined from the plates using an abrasive waterjet. A comprehensive analysis of tomographic images of the samples prior to loading showed that negligible damage occurred as a result of the waterjet machining process and that all of the damage reported in this paper was a direct result of the applied loading. Aluminium tabs were bonded to the samples to allow load transfer in the screw driven load frame shown in Figure 2. The load frame was used with a calibrated load cell to determine the ultimate failure stress of the samples to be 960 MPa across the notch section.

The samples were tested at the ID19 beamline at the European Synchrotron Radiation Facility (ESRF) in Grenoble, France. The experiments consisted of *in situ* SRCT scans under two loading sequences. For the first sequence the specimens were scanned in the pre-loaded state, loaded to a particular stress and re-scanned, and finally scanned in the unloaded state. A comparison of the loaded state (combining mechanical load and residual thermal load) and the un-loaded state (purely residual load) allows the separation of mechanical and thermal loads. For the second configuration the samples were scanned in the pre-loaded state then re-scanned at incrementally increasing stresses that ranged from 30%  $\sigma_f$  to 90%  $\sigma_f$ .

To obtain SRCT data each specimen is rotated through 180° in equal angle increments while a 2-D radiographic projection is captured at each position through the rotation. A suitable reconstruction algorithm, in this case filtered back-projection, may then be used to create a representative tomographic volume of the sample from the sequential projections [10]. This technique is similar to conventional micro-focus X-ray computed tomography, however important differences arise in the source of the X-rays and the scale of operation. In contrast to the divergent beam emmited from a microfocus X-ray source by electron decceleration into a metal target, synchrotron X-rays are the result of electron deflection within a high energy storage ring [11]. In this context, synchrotron X-rays are not only relatively intense, but essentially parallel , monochromatic and coherent. Optical microscopy with a CCD detector is used to obtain radiographs of a sample at very high resolution. Further details of the operating mode can be found at [8,12] and a schematic of the two techniques is shown in Figure 3.

For this work the X-ray beam energy was 20 keV. Imaging conditions were set for both absorption and phase contrast (edge-detection regime) [13,14]. After the specimens were located in the load frame and attached to a precision movement stage, the samples were placed 37 mm (phase contrast conditions) from a 14 bit fast read-out, low noise (FReLoN) detector attached to a 2048 x 2048 pixel CCD camera. An isotropic voxel size of 1.4 µm was achieved using this system. During each tomographic scan 1000 radiographs were collected through the 180° rotation. The exposure time for each radiograph was 50 ms, which resulted in scan times of less than 4 minutes. The radiographs were reconstructed using ESRF in-house software and the resulting 3-D volumes were analysed using commercially available software [15] and various routines using Matlab and VTK.

#### **3 Results and Discussion**

#### **3.1 Experimental Observations**

CT reconstruction allows the volume of a given sample to be visualized as a 3-D grey-scale map [16] in which the grey-scale value correlates to the local material X-ray adsorption coefficient, depending strongly on atomic number (photoelectric regime). Two distinct peaks in the histogram of gray-scale intensity represent air (cracks, splits, voids, delaminations and the area surrounding the notch boundary) and solid material (continuous, intact fibre and epoxy). By defining thresholds around the appropriate grey-scale values, material and cracks can be segmented and separated. For low-density objects, or for regions in which two separate structures have similar densities, phase contrast can be exploited to improve edge detection.

Figure 4(a) shows a section of a typical reconstructed SRCT volume while Figure 4(b) and 4(c) show particular in-plane 2-D slices in which segmented intra-laminar damage is identified. Figure 4(b) shows damage viewed parallel with the 0° splits and Figure 4(c) shows a view parallel with the transverse ply crack.

In Figure 4(a) the volume has been partly withdrawn to the laminate centre-line to reveal one transverse ply crack in the 90° ply and two splits in the 0° ply emanating from the notch tip. In this particular material system, with a toughened epoxy matrix, delamination did not occur until a load of 70%  $\sigma_f$  was exceeded. In this figure the sample was loaded to 40%  $\sigma_f$  and as such no delaminations are present between the plies. The rough appearance of the notch is due to the abrasive water jet used to create the sample geometry.

When including samples taken to failure, three principal damage mechanisms have been observed in this work: 0° ply intra-laminar splits, 90° ply transverses ply cracks

and 90°/0° ply-interface delaminations. The three mechanisms initiate and interact in a complex manner, however as an initial analysis of the laminate damage, and since delamination does not occur in this material at loads lower than 70%  $\sigma_f$  this paper will in the first instance concentrate on intra-laminar damage in the 0° ply alone.

Quantitative information has been extracted from the CT images [10] and used to determine the accuracy of a model by comparison with its predictions. The crack opening displacement (COD) along and through the thickness of individual splits has been measured by identifying the number and position of connected voxels that have a grey-scale value corresponding to the crack. The shear displacement has been measured by identifying unique reference points in the sample microstructure and determining the relative positions of these points before and after loading. Whilst the voxel resolution of the image presented here is limited to  $1.4\mu$ m, sub-voxel interpolation may be exploited to determine edge locations to an accuracy of the order of 1/10th of this value (e.g. via the partial volume effect, taking into account the local grey scales associated with the edge detection regime).

Figure 5 shows a plot of the COD derived from SRCT data in a sample loaded to 50%  $\sigma_f$ . The Y-axis represents the position through the thickness of two plies where each ply is 250µm (178 pixels) in thickness. The X-axis represents the position along the split. The grey-scale of the plot relates to the opening of the crack with darker regions indicating a larger opening displacement. The most notable feature is the area made up of two regions where the crack is pinned. This location coincides with resin rich regions where the remainder of the split length consists of echelon cracks [17].

#### **3.2 Finite Element Model**

For the initial analysis linear elastic, 20-node quadrilateral, 3-D solid continuum elements with reduced integration points were selected. The laminate has 3 planes of geometrical symmetry to allow a model of 1/8 size to be used with appropriate boundary conditions for the nodal degrees of freedom. Eight layers of elements were used to model each ply thickness. The material properties for each ply have been specified directly according to the supplier's data sheet and are listed in Table 1. The symbols have their usual meanings, and  $\alpha$  is the thermal coefficient of expansion. The  $0^{\circ}$  and  $90^{\circ}$  ply connectivity was defined by tying together the corresponding elements of the interface surface from each ply. The crack surface was modeled using separated nodes that initially occupied the same co-ordinates. The overall length of the crack was determined from the SRCT images and was calculated based on equal overall crack face area. The ligamented crack front (identified in Figure 5) has not been accounted for. A mesh refinement study was carried out to confirm that the model results converge at the selected mesh density. A mechanical load was applied by specifying a displacement of 5 mm on the upper surface of the model. The load associated with this displacement was compared to the loads applied during the experiment to calculate a conversion factor. Since the FE analysis was linear elastic this factor was applied to the model predictions of COD and shear displacement to obtain the results for comparison with the SRCT data. A thermal load induced by the residual stresses imposed during autoclave cure was also applied to the model, based on the coefficient of thermal expansion mismatch between adjacent plies during a temperature change of 180 °C to 25 °C. Figure 6 shows the mesh geometry representing the experimental samples that were imaged using the in situ SRCT test conditions.

To obtain COD and shear displacement results, the nodal displacements of the mesh were recorded after loading. For COD, the relative movement along the X-axis of each

node pair that represents the split was determined along the length of the split for each of the eight elements through the thickness of the laminate. The shear displacement was calculated as the relative motion of the node pairs along the Y-axis. Post-processing of the model returned separate results for mechanical load and thermal load for comparison with the corresponding experimental results derived from the loaded and un-loaded SRCT data.

For model validation the COD and shear displacements calculated from the SRCT data was compared to the model predictions. Figure 7 illustrates the locations around the split at which the calculations and predictions were taken. A COD plot was calculated from segmented SRCT images. The 3-D nature of the technique and analysis allows COD to be plotted as a function of position through the thickness of the two 0° plies and of position along the split length. The FE model assumes laminate symmetry and therefore consists of just one 0° ply. To facilitate an equivalent comparison with the experiment the COD data from SRCT analysis was mirrored and averaged across the two 0° plies. Figure 7(b) shows four locations along the length of the split. Each location is labeled according to the fraction of the split length and a plot of COD vs. position through the 0° ply thickness has been generated for each location. The location "4/5" is closest to the crack tip and the data is shown graphically in Figures 7 and 8. Figure 7(c) shows four additional, separate locations through the thickness of a 0° ply. Each location is labeled according to the fraction of the ply thickness and a plot of COD vs. position along the split length has been generated for each location. The location "1/5" is closest to the 0°/90° ply interface and the data is shown graphically in Figure 9. The dotted centre-line shows the boundary between the two 0° plies.

#### **3.3 Model Performance**

A comparison of the model results against the measured experimental results for COD is shown in Figures 8, 9 and 10 for a specimen loaded to 60%  $\sigma_f$ . In Figure 8 and 9 the opening displacement is plotted against the position through the thickness of the 0° plies for four positions along the length of the split. In Figure 10 the opening displacement is plotted against the length of the split for four equal incremental positions through the thickness of the two 0° plies. Figure 8 shows the results in the loaded state (a combination of mechanical and thermal stress). Figure 9 shows the results in an unloaded state where the thermal residual stress acts alone. Figure 10 shows the results of the difference between the loaded and unloaded states (purely mechanical load). Figures 8(a), 9(a) and 10(a) show the experimental data and Figures 8(b), 9(b) and 10(b) display the model data.

The data in Figure 10(b) starts at 500  $\mu$ m as the split does not initiate from the same location through the ply thickness. The experimental data was measured from the location closest to the notch at which the split was present through the entire ply thickness.

The COD data presented in Figures 8 and 9 show that there is reasonable agreement between the experimental and model results. As expected the crack is more open at the root than at the tip and exhibits pinned behaviour with reduced opening as the crack approaches the  $0^{\circ}/90^{\circ}$  interface. The graphs of experimental and model results show similar trends both through the thickness of the laminate and along the length of the split despite the immediate simplifications in the idealized model compared to the observed experimental data. As identified in Figure 5, the experimental crack does not have a single simple length value to which the model can be compared. In addition there are many microstructural features that have not been accounted for in the model, most notably the bridging and crack pinning along the length of the split, which occur at

resin rich regions. Matrix-toughening particles have also been identified in these resin rich regions throughout the plies. The uneven trend of the experimental results is due to the non-straight-fronted crack propagation.

The crack opening displacement in the unloaded state is shown in Figure 9 and is due to the thermal residual stress. These graphs show that the contribution to COD is significant and as such thermal effects must be included when modelling laminate damage.

Subtraction of the residual opening from the loaded case allows the COD from mechanical loading alone to be extracted. This is shown in Figure 10 in which COD is plotted along the length of the split for four positions through the laminate thickness. The experimental results and model predictions show good correlation both in terms of the overall trend and the absolute values.

The experimental shear displacement data was obtained by identifying particular features, such as particles, within the microstructure at similar positions either side of the notch tip (from which the split initiates) and tracking their motion before and after loading. The shear displacement,  $\delta_y$ , is defined as the difference of travel in the y-direction between a pair of features either side of the split and is illustrated schematically in Figure 11. In this diagram *notch side* refers to the area closest to the free edge of the sample, while *bulk side* refers to the area closest to the centre of the sample.

The shear displacement data is shown in Figure 12 where it can be seen that the model displacements vary substantially from the measured experimental results. In addition to the above discussion of experimental and FE data, further contributions to their comparison can be identified in the effect of the de-bonding of rubber toughening

particles that have been introduced within the matrix (Figure 13) and transverse ply cracks in the 90° ply. The bridging/ pinning behaviour of the resin toughened regions has not been accounted for in the model. The experimental results show that the COD does not vary significantly through the thickness of the 0° ply. SRCT images also show that there is a matrix rich region at the interface between the 0° and 90° plies. The reduced compliance of the matrix with respect to the averaged 0° lamina properties may allow the stress gradient at the 0°/90° ply interface to act mostly across this region alone. The model does not incorporate this region at present and uses element tying to attach the plies. This method effectively pins the split at the interface and does not allow for deformation.

It is evident that, having seen the true three dimensional behaviour of cracking in the CT data, the FE modelling of a simple straight crack front is missing important micromechanical detail. Future work is underway to develop more detailed finite element models that include the observed micromechanical detail and an analysis of the sensitivity of the model to resin material properties and toughening processes. The loads applied in the study presented here were below the threshold for initiation of delamination: future modelling work aims to determine the effect of inter-ply decohesion on displacement fields and energy release rate during damage growth as the load approaches the laminate failure stress using physically based cohesive elements calibrated from the SRCT data. Notwithstanding the need for more refined models it is evident that the initial modelling presented herein makes predictions for the internal crack openings and displacements under load experimentally which are of the correct order. A key point to highlight is that with the internal full field measurements afforded by the SRCT data, uniquely detailed model initialisation and validation may be achieved.

#### **4** Conclusions

The three-dimensional nature of damage onset and growth has been observed, allowing the extent of intra-lamina damage to be quantified with a confidence not possible using conventional 2-D imaging methods. Using microstructural features it has been possible to calculate local displacement fields around the intra-laminar 0° splits. In addition the CT data provides information on crack opening displacements along the splits. This information has been used to inform the development of an initial finite element analysis model of the intra-lamina damage. The quantitative data for strains and COD allows the model to be validated for the loading conditions of interest. The 'data rich' mechanics approach that SRCT offers is a valuable tool in developing and validating finite element models. Micron-scale damage can be resolved readily and full field, internal 3-D data is made available for model comparison.

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Figure 1: (a) Geometry of composite samples, (b) detail of notch section of samples, and (c) illustration of sample with bonded aluminium tabs.

Figure 2: (a) Semi-transparent schematic of *in situ* load frame, and (b) annotated cross-section of schematic.

Figure 3: Schematics of the operation of computed tomography (CT). (a) Divergent cone beam micro-focus X-ray CT, and (b) generation of a high intensity, low divergence synchrotron X-ray beam at the European Synchrotron Radiation Facility showing SRCT experiment set-up.

Figure 4: (a) Tomography image of intra-laminar damage in a  $[90/0]_S$  laminate at 40%  $\sigma_f$ . The bulk of the composite has been withdrawn to reveal the damage, comprising two 0° splits and a transverse ply crack, (b) 2-D slice through the volume in the X-Z plane showing the 0° splits, and (c) 2-D slice through the volume in the Y-Z plane showing the transverse ply crack (half laminate thickness shown). Note that no delamination is present at this load.

Figure 5: Greyscale plot of the crack opening displacement for a 0° split.

Figure 6: Element mesh geometry representing the experiment sample. View of the deformed mesh around the split at the  $0^{\circ}/0^{\circ}$  ply interface (symmetry conditions) under load.

Figure 7: (a) Segmented tomographic volume of notch region of sample identifying particular region of damage (0° split) to be quantified, (b) COD plot of 0° split showing the four positions along the split length at which a comparison is made with model data, and (c) 2-D radiographic projection of 0° split showing the four positions through the laminate thickness at which a comparison is made with model data.

Figure 8: Crack opening displacement through the ply thickness for multiple positions along the split length from the split root towards the tip. (a) Experimental data mirrored and averaged across two 0° plies, and (b) model data for one 0° ply (by symmetry).

Figure 9: Crack opening displacement through the ply thickness for multiple positions along the split length from the split root towards the tip. (a) Experimental data mirrored and averaged across two 0° plies and (b) model data for one 0° ply (by symmetry).

Figure 10: Crack opening displacement along the split length for four positions through the ply thickness. (a) Experimental data and (b) model data. Positions are distinguished by fraction of the depth into the 0° ply thickness from the 90°/0° interface towards the  $0^{\circ}/0^{\circ}$  interface.

Figure 11: (a) Illustration of the shear stress acting between the split flanks in the 0° plies, (b) position of microstructure features either side of the split identified for

tracking in the loaded and unloaded stated, and (c) Schematic of definition of shear displacement, d<sub>y</sub>.

Figure 12: Shear displacements along the split length for 4 positions through the ply thickness.

Figure 13: 2-D slice through the volume in a 0° ply showing de-bonding of rubber toughening particles within the split next to the notch.

Young's modulus E (GPa)	Poisson's ratio v	Shear modulus G (GPa)	Thermal expansivity α (10 <sup>-6</sup> K <sup>-1</sup> )
$\begin{array}{c} E_{11} = 148.0 \\ E_{22} = 7.8 \\ E_{33} = 7.8 \end{array}$	$v_{12} = 0.35$ $v_{13} = 0.35$ $v_{23} = 0.35$	$G_{12} = 3.8$ $G_{13} = 3.8$ $G_{23} = 2.9$	$\alpha_{11} = -1.0 \times 10^{-3}$ $\alpha_{22} = 34.0$ $\alpha_{33} = 34.0$

Table 1: Material property constants used to define carbon fibre-epoxy composite in the fibre direction for finite element modelling.





a)







y-position (µm)























150 µm