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Gaining mechanistic insight into key factors contributing to crack path transition in particle toughened carbon fibre reinforced polymer composites using 3D X-ray computed tomography

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Abstract

Composite materials are increasingly used to help in reducing the carbon footprint of transportation and upscaling renewable energy infrastructure that provides clean energy for future cities. However, the inherent susceptibility of carbon fibre reinforced polymers to impact damage results in knock-down in design and is linked to the micro-mechanistic response of the material to damage. *In situ* experimental and high-resolution imaging techniques using X-ray computed tomography (X-ray CT) have been used to gain a mechanistic understanding of the key factors controlling crack path — and hence macro-scale toughness within a composite. Multiscale Synchrotron Radiation Computed Tomography (SRCT) and lab-based micro-focus X-ray CT are used to investigate different material systems toughness response from standard Double Cantilever Beam tests. The crack transition to the weaker ply region of the composite is identified as a controlling factor across a scale of mm's, and 'trigger' regions are reported on and investigated. The 'trigger' regions were identified as gaps in the ply adjacent to the interlayer. This work feeds directly into delamination growth predictions, a better understanding of material response, and enabling informed manufacture and design, allowing for reduced material usage, longer life and more sustainable vehicles and infrastructure.

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Keywords: Transport; Renewable energy; Polymer matrix composites; Synchrotron Radiation Computed Tomography; X-ray computed tomography; Delamination; Toughness; Damage response

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1. Introduction

The use of composite parts on the Airbus A350 XWB© resulted in a fuel efficiency saving of around 25% per seat and 20% lower cash operating costs per seat compared to previous long-range competitors such as the Boeing 777 [1]. However, despite the potential benefits realised from using CFRPs in design, they remain particularly susceptible to low-velocity impact damage where subsurface delaminations can reduce the residual strength of the part by up to 60% [2]. The use of thermoplastic toughening particles (TP's) has been shown to suppress these delaminations and increase both the strength and toughness of composite materials [3,4]. However, crack initiation and propagation within particle-toughened (PT) interlayer systems are still not well understood, and previous work has shown the loss of load-carrying capacity in composites due to a crack transition away from the PT interlayer [5,6].

X-ray CT in conjunction with *in situ* loading allows for a time series sequence of crack growth to be visualised in 3D and permits further useful information to be obtained by mapping back to the undamaged state. This allows for a mechanistic understanding of damage response mechanisms which lead to the critical crack transition and subsequent decrease in fracture toughness of the composite. The Double Cantilever Beam (DCB) test is used to assess the CFRP specimens on a scale of 10'–100's mm's. *In situ* loading and imaging using both lab-based X-ray computed tomography (X-ray CT) at the University of Southampton and Synchrotron Radiation Computed Tomography (SRCT) show the damage response mechanisms and influences that correlate to the observed macro toughness.

2. Materials and specimen composition

Proprietary materials were developed, manufactured and provided by the Cytec Solvay Group. They were cured according to a standard aerospace cycle and consisted of a 26 unidirectional ply lay-up. A 40 μm Polytetrafluoroethylene (PTFE) insert was placed in the midplane at the edge of the specimen to provide a starter crack. TP's are dispersed throughout the interlayer at a 13% volume fraction. The bulk resin of the composite was reinforced with intermediate modulus carbon fibres along the length direction of the specimen. Five specimens were provided with dimensions conforming to BS ISO 15024 [7] and ASTM D5528 standards [8].

2.1. Measuring fracture toughness

The mode I fracture toughness of the composite was obtained using the DCB test method according to BS ISO 15024 [7] and ASTM D5528 standards [8]. Wedge pre-cracking was used to propagate a 'starter' crack, and a minor modification was made to the test method where "stirrups" were used to apply the load to the specimen rather than piano hinges or loading blocks. Modified beam theory was the selected data reduction method as it is reported to give the most conservative values of strain energy release rate [8]. Other methods were tested for comparison; however, no significant differences were observed.

2.2. In situ imaging and SRCT experimental technique

A mode I *in situ* wedge opening experimental rig was developed and manufactured in-house, both inspired by and using the same principles as a previous study [9]. An initial set of *in situ* experiments were carried out at the μ-VIS X-ray Imaging Centre, using the custom 450/225 kVp Hutch system, with the 225 kVp X-ray source and the 2000 × 2000 pixel flat panel detector. A series of scans were carried out at a voxel resolution of 5.2 μm's to cover a region of interest (ROI) of the central 10 mm width and 50 mm ahead of the insert. The material systems were scanned before introducing damage at 120 kVp, 11 W, an exposure time of 0.5 s, 6000 projections and ring artefact suppression enabled. The load was applied using a screw actuated wedge until the crack propagated. The region around the crack tip was then scanned. This process was repeated until the crack had propagated the length of the ROI. The scan parameters used differed by the number of projections used, 4800, and not using ring artefact suppression, reducing the required scan time from 5 h per scan to 1 h 22 min per scan, respectively, for the undamaged and damaged scans. This was to obtain a good quality reference scan allowing measurements and segmentation of images. However, such scan times are both impracticable and unnecessary for determining the crack path during material loading

The same loading method was used in the SRCT experiments. SRCT was carried out at the European Synchrotron Radiation Facility (ESRF) using beamline ID 19, Grenoble, France. Using SRCT allows a resolution and image fidelity not achievable in lab-based X-ray CT, as well as scans taking only a fraction of the time. Each SRCT scan took 2 min and 30 s in the current study. A voxel size of 0.65 µm was used with a 2560 × 2160 pixel camera. Five thousand projections were used to allow for ROI padding [10] with an exposure time of 30 ms for a 180° rotation. The beam energy was 19 keV, and the propagation distance between the sample and detector was 55 mm to outline interfaces between similarly attenuating components in the material using phase-enhanced contrast in the edge-detection regime [11]. The data was reconstructed using in-house software pyHST (High Speed Tomography in python version) [12].

3. Results

3.1. DCB material toughness response

A delamination resistance curve (R-curve) is shown in Fig. 1. The SERR is the energy dissipated during fracture per unit process of a newly created surface area. The strain energy release rate (SERR) for six specimens of the same material system have been plotted. The red shaded area around the data is the 95% prediction interval for a fitted second order polynomial curve through the data. The R-curve shows an initial drop in toughness when the crack begins to propagate over a crack growth length of 5 mm, where the toughness decreases by 29%. The toughness then increases with further delamination to be larger than the initial toughness of the material before falling slightly and levelling out to become relatively flat or self-similar. Finally, at the end of crack propagation, the toughness falls below that of the critical toughness of the material at crack initiation.

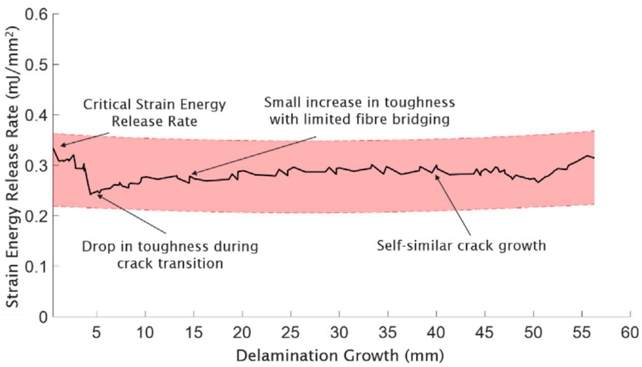

Fig. 1. Variation in strain energy release rate with delamination length during DCB test plotted with the 95% prediction interval.

3.2. Lab-based in situ X-ray CT

Fig. 2(a) shows the cross-section through a CFRP specimen. Several features are notable such as the distinct interlayer in between plies. This is formed as a result of the TP's forcing a ply separation. There are also some regions of resin pockets where the interlayer appears thicker than the regions immediately either side. The circled part of the image has been segmented and shows a gap in the ply adjacent to the interlayer. When this region was mapped to the fracture surface of the specimen, it could be seen that this was the location of a so-called 'trigger' region where the crack began a transition to the ply. Fig. 2(b) and (c) shows a plane through the crack with the crack growth direction from the bottom of the image to the top. The first step of loading is shown in Fig. 2(b), where the crack being in the interlayer is distinguished by the indistinct texture on the image, whereas the more defined 'finger-like' structures indicate the crack propagating within the ply. The same frame of the subsequent step is shown in Fig. 2(c), where most of the frame is taken up by the wake of the crack. Broken fibres are visible in the wake of the crack. At the top of the image, the crack has transitioned in some regions from the interlayer to the ply.

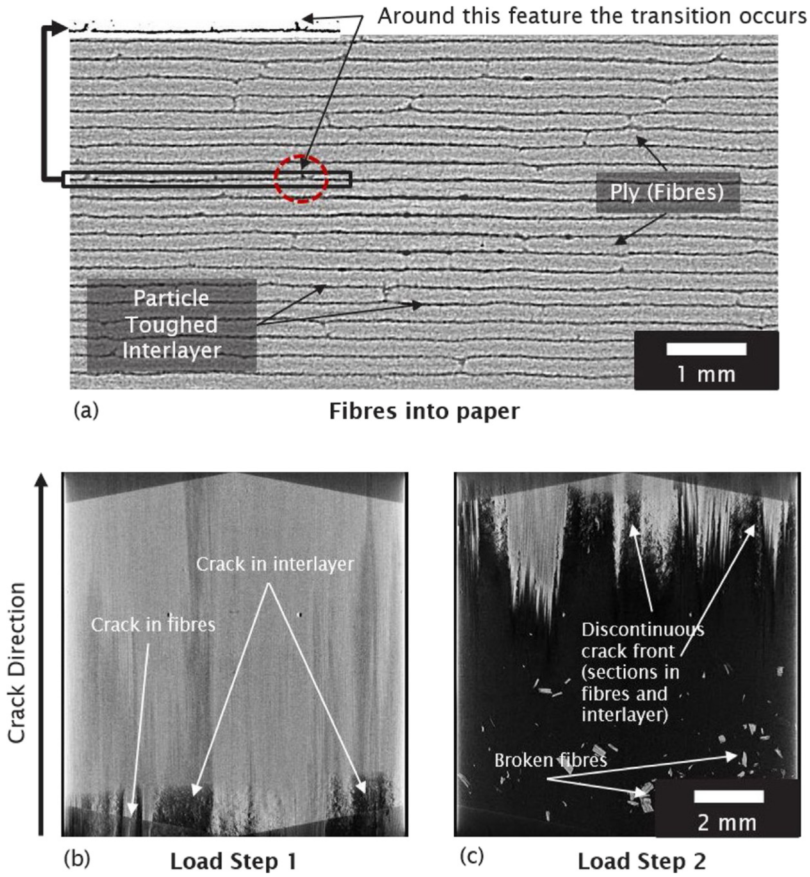

Fig. 2. (a). Cross-section through CFRP sample with the fibres into the screen showing variation in interlayer thickness and fibre tow gaps. (b). Step 1 of the in situ load increment shows the crack in different layers of the composite. (c). Step 2 of the load increment in the same frame. The wake of the crack can be visualised, showing the transition of the crack to the ply.

3.3. SRCT in situ CT

The first load step during an *in situ* SRCT experiment is shown in Fig. 3(a). The fibres in the ply layers can be clearly distinguished, as can the resin-rich interlayer regions between them. The bright edge around the crack is caused by phase-enhanced contrast, and the resulting fringe allows for a clear distinction of the crack boundaries. In the first load step, the crack remains within the PT interlayer and along the interlayer-ply interface. Fig. 3(b) shows the subsequent step in the experiment near the new crack tip further into the specimen. The crack can be seen to have transitioned to the ply in the central region of the image. The transition corresponded to a transition through the ply boundary in the width direction of the image and has bifurcated towards the RHS of the image.

4. Conclusions

Composite materials such as CFRPs are playing a continuing role in increasing the efficiency of transportation and renewable energy infrastructure. Advanced non-destructive multiscale imaging techniques are key enablers of mechanistic understanding of the factors causing knock-down on composite structures. The variation in the toughness response of the material shows the sensitivities to the identified crack path through high resolution *in situ* CT, where the toughness response decreases as the crack transitions to the ply. Variations and distortions in the interlayer and ply boundaries appear to contribute to crack transition as well as a drop in the macro toughness of the composite. It can also be seen that a transitioned crack tends to propagate several fibres deep into the ply, making the transition back to the interlayer difficult. A deeper understanding of the mechanisms leading to crack path

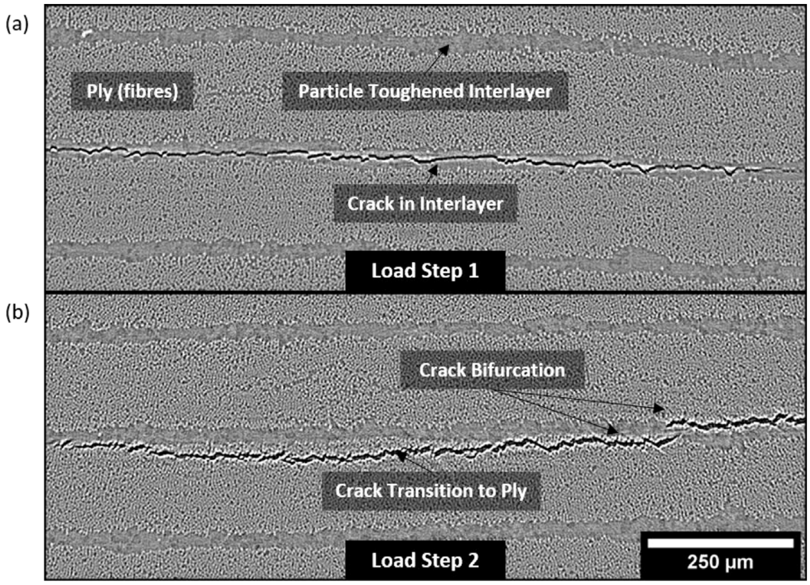

Fig. 3. (a) The first load step in the SRCT experiment. The crack can be seen to be in the interlayer for this section of the sample. (b) In the second load step and along the same section of the specimen, the crack has partly transitioned to the ply.

transition, and therefore a reduction in toughness behaviour, will contribute to optimising composite manufacture and design. Therefore, reducing knock-down factors by retaining the crack within the particle toughened interlayer, such that it is not too weak as to reduce the toughness of the system but not so tough that the crack transitions to a weaker fibre interface in the ply. These advances, in turn, will have the potential to allow for less conservative design approaches, allowing for reduced material usage and more sustainable vehicles and infrastructures.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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