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# Interlaminar fracture micro-mechanisms in toughened carbon fibre reinforced plastics investigated via synchrotron radiation computed tomography and laminography

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#### ABSTRACT

Synchrotron Radiation Computed Tomography (SRCT) and Synchrotron Radiation Computed Laminography (SRCL) permit 3D non-destructive evaluation of fracture micro-mechanisms at high spatial resolutions. Two types of particle-toughened Carbon Fibre Reinforced Polymer (CFRP) composites were loaded to allow crack growth in Modes I and II to be isolated and observed in standard and non-standard specimen geometries. Both materials failed in complex and distinct failure modes, showing that interlaminar fracture in these materials involves a process zone rather than a singular crack tip. The work indicates that incorporating particle/resin, fibre/interlayer and neat resin failure is essential within models for material response, since the competition between these mechanisms to provide the energetically favourable crack path influences the macro-scale toughness. The work uniquely combines the strengths of SRCT and SRCL to compare failure micro-mechanisms between two specimen geometries, whilst assessing any edge effects and providing powerful insight into the complex micro-mechanical behaviour of these materials.

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#### **1. General Introduction**

The high specific stiffness and strength of CFRPs has led to their use in aerospace applications, where a reduction in weight has a direct impact on the payload and range of the aircraft. However, composites may suffer from low velocity impact damage whilst in service that can have a significant effect on the residual mechanical properties. Such events may cause significant internal damage in the form of delaminations, which are difficult to identify from surface inspections and may reduce compressive properties by up to 60% [1]. The need to resist impact damage, and the lack of reliable predictive models to account for impact and post-impact performance, contributes to the over-engineering of composite structures and a failure to achieve the desired weight-saving and consequent performance improvement. Given that Mode I and Mode II dominated loading conditions have been identified to occur under low velocity impacts [2], modelling such Mode I and II fracture is a key first step in developing models for impact damage resistance and post impact damage tolerance.

Incorporation of secondary phase particles into the polymer matrices has been identified as an effective way of increasing the matrix toughness. Key toughening mechanisms have been identified as; crack pinning [3], crack path deflection [3,4], particle/ matrix de-bonding and subsequent void growth [5], localized shear yielding [6] and bridging of the crack surfaces by the particles [7]. However, the relevance of such mechanisms varies between specific matrix/particle combinations, where, for example, it has been shown that the size and stiffness of the particles significantly influences the ability of the crack to be pinned or to deflect the crack path appreciably [4,8]. Toughness studies have been conducted, by systematically varying particle volume fractions, and particle stiffness and size, in which orders of magnitude of improvement in bulk toughness were shown to be possible by controlling these parameters [3,9,10]. The incorporation of such matrices between the plies of a composite, called interlayering, has been shown by several authors to increase the interlaminar toughness [11–17]. However, it has been established that an improvement in bulk resin properties does not directly translate to an improvement in interlaminar toughness, and this has been assumed to be due to the constraining action from the neighbouring plies [11,12]. Furthermore, increasing interlayer toughness can be ineffective if





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the cracks propagate by avoiding the toughened interlayer [18,19], which was observed in one of the systems investigated in this work.

Damage inside a composite material can be made visible via Xray computed tomography (CT) using laboratory [20,21] and synchrotron sources [22-25] with specimen geometries adapted to the CT scanning technique. This permits in situ observations of toughening mechanisms to be made using non-invasive techniques away from free-edges under representative stress states. This has recently become feasible for planar geometries due to the development of Synchrotron Radiation Computed Laminography (SRCL) [21,26–29]. The experiments documented in this paper represent the first to capture in situ toughening mechanisms operating in Mode I interlaminar cracks in particle-toughened composite laminates, tested in both standard and non-standard geometries. They permit the direct identification of micro-mechanical features and mechanisms that affect the global Mode I fracture toughness. Mode II identification of micro-mechanisms was conducted ex situ, but the SRCL and SRCT techniques still provided invaluable information without changing the stress state on the sectioning plane that may introduce out-of-plane displacements. The controlled loading conditions and image quality permitted the identification of the sequence of local damage evolution, emphasizing the influence of local microstructural irregularity on the location and geometry of damage initiation and growth.

## 2. Methodology

#### 2.1. Materials

CFRP test coupons, provided by Cytec Engineered Materials, were manufactured from developmental particle-toughened material systems. The toughening was confined to a  $\sim$ 30 µm thick particle-toughened interlayer in each system. The primary reinforcement was a proprietary intermediate modulus carbon fibre ( $\sim$ 5.4 µm in diameter). A 16 ply (3 mm thick) uni-directional layup was prepared from pre-preg with a 40 µm thick Polytetrafluoroethylene (PTFE) insert in the mid-plane of the sample in order to control the initiation of fracture. Materials were laid up by hand and cured by the manufacturer according to a standard aerospace autoclave cure cycle. Two different particle types were investigated; with the fibre type, sizing, base resin and particle volume fraction remaining constant between the material systems. Material A (Mat. A) can be identified with the visible particles in the figures, and Material B (Mat. B) in which the constituent material density of the particles is too similar to the resin to be identifiable via CT. The composition of the materials is proprietary and is not important to the key observations and conclusions of this paper. The two systems demonstrated significantly different fracture micro-mechanisms and fracture toughness, highlighting the importance of understanding micro-mechanical behaviour to develop an effective interlayer.

## 2.2. Specimen geometries

To take full advantage of SRCT and to avoid artefacts, a cross-sectional geometry of  $3 \times 1 \text{ mm}(\text{Fig. 1}(a))$  was chosen to maximise the transmission of low energy X-rays and to provide a relatively uniform X-ray path through all angles of rotation. These specimens were 120 mm long and had a 10 mm long PTFE insert. Fracture toughness values for the narrow samples could not be obtained due to difficulties associated with their 1 mm wide geometry. British Standard geometries (BS ISO 15024:2001) were employed in the fracture toughness testing and SRCL imaging, which permits the use of laterally extended specimens that are closer to practical component and structural length-scales. The specimens (Fig. 1(b)) were



**Fig. 1.** Specimen geometries used (a) for SRCT and (b) for SRCL. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

20 mm wide, 3 mm thick and 150 mm long. There was a 50 mm long PTFE insert placed at the mid-plane. In contrast to SRCT, the sides of the specimen do not pass through the field of view during imaging, allowing the sides of the specimen to be painted with white brittle paint to discern the approximate location of the crack tip.

#### 2.3. Mode I testing

A wedge-loaded *in situ* double cantilever beam arrangement was used for both specimen geometries. The wedge was driven into the mid-plane in a displacement-controlled manner as shown in Fig. 2(a). The specimens were lightly loaded in Mode II in order to release the insert from the resin, since this was found to help the insertion of the wedge into the interlaminar region. An initial load-ing step was applied to create approximately 10 mm of Mode I crack extension prior to SRCT or SRCL testing so as to reduce any effects from the initial Mode II loading.

#### 2.4. Mode II testing

Mode II loading was conducted using a three-point bend arrangement with a 100 mm span for both the SRCT and SRCL



**Fig. 2.** Schematic of the scan locations with (a) the wedge-loaded Mode I experimental set-up and (b) the three point bend arrangement for Mode II. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

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