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INTERFACIAL NORMAL RESPONSE OF CARBON AND GLASS FIBRE EPOXY COMPOSITES MONITORED VIA *IN SITU* HOLOTOMOGRAPHY

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ABSTRACT

First-ply failure (FPF) concerns the point at which the weakest ply in a composite laminate reaches its theoretical limit under load. This, in most cases, means that the laminate can continue to carry the load beyond FPF. Regarding damage mechanisms, FPF is often conceptually linked with the initiation of fibre-matrix interfacial (normal) debonds that evolve into off-axis cracks and, later, delamination at ply interfaces. With the continuous advancements in non-destructive testing (NDT) methods for composite structures, the notion of FPF has become increasingly important, with many applications requiring the structure never to reach FPF. That, however, comes at a cost, with excessive safety factors put in place to withstand the uncertainties associated with composite damage and failure. In this study, we shed light on the microscale damage mechanisms in FPF of composites. To achieve this, we exploit synchrotron tomography to investigate the first damage mechanism anticipated in transverse loading of composites: fibre-matrix interfacial normal debonding. Our main objectives lie in understanding the influence of fibre type and fibre packing on interfacial normal debonding and offering a state-of-the-art experimental dataset as input or validation for numerical predictions.

To achieve these objectives, *in situ* transverse single-fibre tests were conducted for three types of specimens: (a) glass single-fibre (Advantex R25HX14), (b) carbon single-fibre (TORAYCA T700SC), and (c) carbon bundle (TORAYCA T700SC) composites. In all configurations, the fibres were embedded inside the epoxy matrix (Sicomin SR85000 resin with KTA313 hardener) and were oriented perpendicular to the load direction. The epoxy matrix was doped with 1.5 vol.% barium titanate nanoparticles to enable digital volume correlation (DVC) analysis. The particles provide a DVC speckle pattern without significantly altering the matrix's mechanical and rheological properties [1]. Each specimen was loaded step-wise, and scans were acquired at pre-determined load steps, ranging from 25% to 99% of the ultimate tensile strength.

The *in situ* tests were conducted at the ID16B beamline of the European Synchrotron Radiation Facility (ESRF), which offers holotomography. Holotomography is a phase-contrast nanoimaging technique that acquires multiple tomograms at different distances, enabling complete phase retrieval. Tomographic reconstruction is precise, especially at phase boundaries, making it an exceptional choice for studies scrutinising the fibre-matrix interface. The technique has also led to the first-ever *in situ* 3D detection of longitudinal fibre-matrix debonding and shed light on the differences in interfacial shear response between single-fibre and multi-fibre composites [2].

Figure 1a demonstrates the experimental setup, which included an in-house-developed load rig. The scan parameters were as follows: pink beam at 29.6 keV, PCOedge 4.2 CLHS detector (2048 × 2048 px^2), 10 ms exposure time, and 2505 projections. The distance between the source and the detector

was set such as to achieve a pixel size of 150 nm, which remained constant throughout the scans. The field of view imaged the fibre at the width-wise edge of the specimen where the interfacial normal debond was anticipated to initiate (Figure 1b-c).

A dedicated image analysis procedure was set up to quantify a series of damage parameters, including (a) debond depth, (b) debond opening, (c) debond angle, and (d) matrix crack propagation. The debond parameters were quantified separately for the top and bottom parts of the interface, as for both fibre types, the debond exhibited preferential growth along one of the two parts of the interface. 3D segmentations were constructed from the scans (Figure 1b-c), offering a detailed 3D view of interfacial normal debonds and matrix cracks.

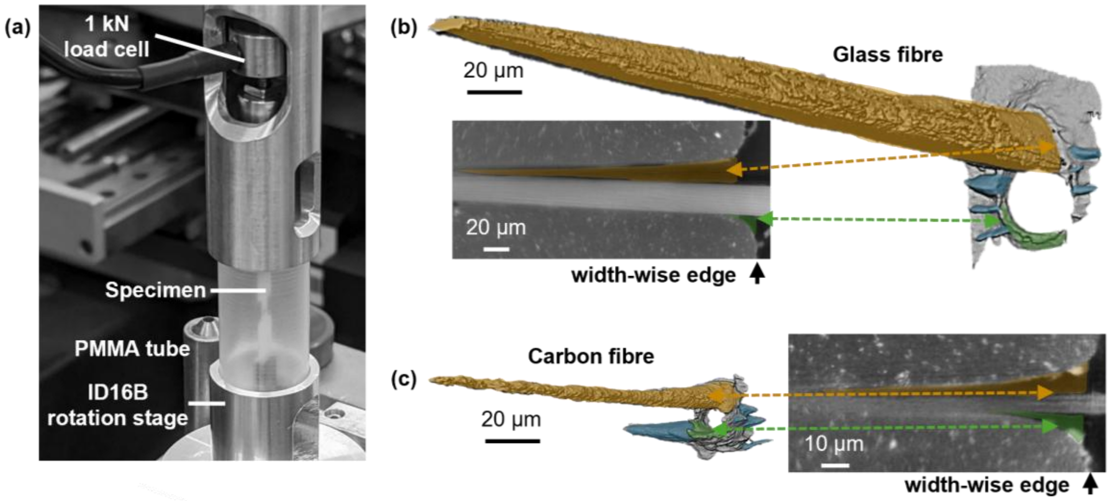

Figure 1: (a) experimental setup at the ID16B synchrotron beamline: 2D and 3D visualisation of (b) glass and (c) carbon single-fibre specimens, where different damage areas are showcased in different colours: yellow indicates the debond at the top part of the interface, green indicates the debond at the bottom part of the interface, and blue indicates the matrix cracks located at the edge of the specimen.

All specimens, irrespective of fibre type or number, exhibited the same three damage stages: (a) debond initiation at the edge, (b) slow debond propagation up to a debond depth of 2-3 fibre diameters and (c) unstable debond propagation or debond tunnelling. However, damage initiation was found to depend on fibre packing, as in the bundle specimen, earlier initiation was observed in closely packed fibres. This trend, however, was not observed for debond tunnelling, which occurred in similar global stresses for all fibres, irrespective of fibre packing. Future analysis will use digital volume correlation to quantify the local 3D strains in the same regions of interest.

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