ESRF	**Experiment title:** *Mapping of longitudinal fibre strain in fibre-reinforced polymer composites*	**Experiment number**: *MA-4766*
Beamline: *ID11*	**Date of experiment**: from: *03/06/2021* to: *06/06/2021*	**Date of report**: *24/12/2021*
Shifts: *9*	**Local contact(s)**: *Jonathan Wright*	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Sina AhmadvashAghbash, KU Leuven*

Matthias Bönisch, KU Leuven*

Christophe Le Bourlot, INSA de Lyon*

Eric Maire, INSA de Lyon

Yentl Swolfs, KU Leuven

Mahoor Mehdikhani, KU Leuven

Christian Breite, KU Leuven

Report:

A) Overview

Strain or stress concentrations are the key microscale feature that govern the failure of unidirectional fibre-reinforced composites and are vital in reliable failure prediction models. Despite their importance, current measurement tools have not been able to quantify them accurately because they occur locally inside the material and the strains are small. This study performs detailed XRD strain mappings on carbon fibre-reinforced composites, guided by phase contrast tomography and radiographic projections. This will yield:

- Invaluable data to validate or reject the current assumptions in failure prediction models;
- Novel mechanistic insight into local strain concentrations, which will be measured for the first time;
- Validation data for the scientific community.

B) Conducted experiments

The tested specimens are model composites with 1, 2 and 3 carbon fibre (7 µm in diameter) embedded in epoxy and carbon fibre reinforced epoxy (fibre volume fraction of 60%). Succeeding first XRD scans on initial 0.5*mm* thick specimens, the specimens were polished down to thicknesses below 0.3*mm* to reduce the inherent background from the epoxy. This preparation step was carried out with concurrent optical microscopy, making sure that the fibre(s) is still intact. The specimen of interest was then mounted on the loading rig in the 3DXRD station of ID11 and was incrementally strained (displacement-controlled) and continuously observed optically to detect possible fibre breaks. After detecting possible fibre break(s), tomography was carried out to locate and accordingly reduce the area of interest for the XRD analysis. The tomograms of the specimens have pixel size 0.6x0.6mm^2 (Marana detector). Diffraction mappings were limited to 0.3*mm* above and below the fibre break(s) with horizonal spacing of 1μm and vertical spacing of 10μm (exposure time of 3*s* and Frelon3 as detector). This procedure was then repeated at higher load levels until failure of the specimen. The displacement increments were chosen large enough (at least 10% increase per scan) to sufficiently evolve the strain levels. For two and three fibre composites, we were required to perform the experiments at 45° or 90° to avoid having multiple fibres in the beam. The test setup, including the Deben rig and drilled PMMA tubes are shown in Fig. 1. The original planning intended to perform in addition 2 and 3 fibre specimen tests using a highly-focused X-ray beam on the ID11 Nanoscope station. However, due to complexity and low likelihood of obtaining successful fibre breaks without breaking the specimens, it was not possible to carry out these additional tests within the available beam-time at the Nanoscope station.

Fig. 1. Test setup at the 3DXRD station including Deben testing rig, beam and optical microscope.

C) Status and progress of evaluation

A summary of the preliminary results will be presented in the next section. Quantitative analysis and the comparison between the different specimen types are on-going. Results from these experiments will be used to understand the mechanisms of strain recovery in a broken fibre and the strain concentration imposed to neighbouring intact fibres. The tomograms of successfully tested specimens are shown in Fig. 2.

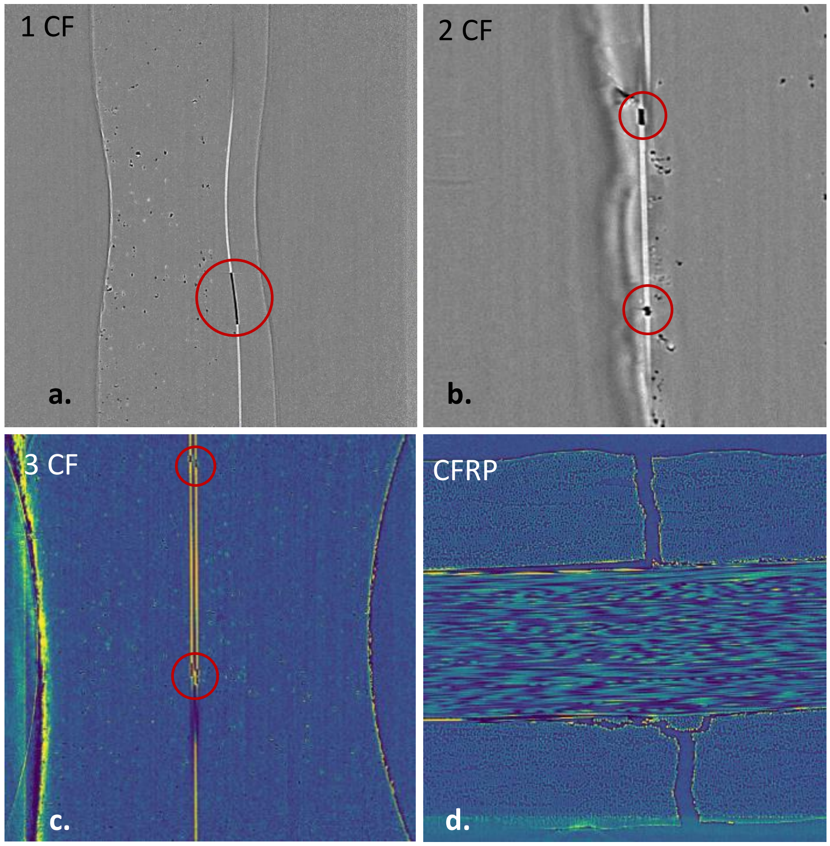

Fig. 2. Computed tomography results of successful specimens: (a) single-fibre (b) two-fibre, (c) three-fibre and (d) CFRP specimens (the fibre break(s) are encircled in red).

D) Preliminary results

The general analysis steps that are required to initiate the preliminary analysis include: calibrating and transforming the detector images into polar space (q, azimuth), defining ROIs for carbon fibre peaks (see Fig. 2), averaging the intensity in ROIs along azimuth, removing the background and extracting the peak position by fitting the peak profile. Finally, the acquired peak position is converted from q (nm^{-1}) to d (nm) and the strain is calculated from d and reference d_0 for pure carbon fibre at zero load. A first short and fast analysis of the single-fibre composite specimen has been carried out. By assuming a zero strain where the fibre is broken, this first analysis indicates a maximum 1.5% strain 500μm away on each side of the fibre (see Fig. 3). However, this result has to be taken with a lot of precautions, as the analysis requires further improvement and refinement. Possible additional necessary steps include correcting for peak overlap, fluctuations in beam current, etc.

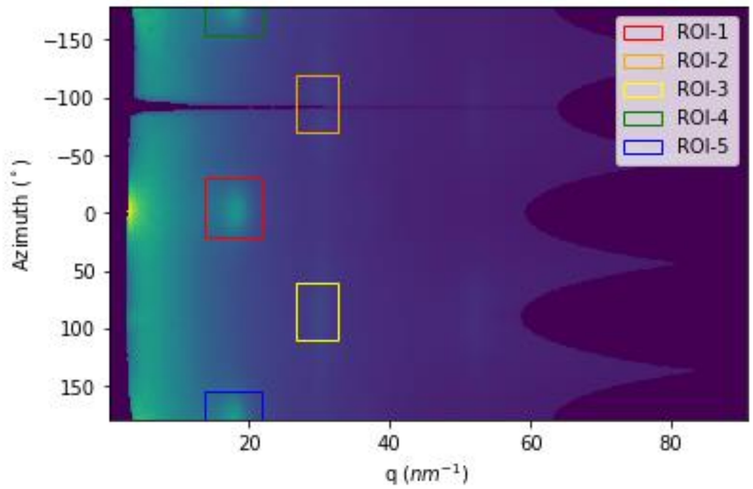

Fig. 2. Selected regions of interests for transverse strain: ROI-1, ROI-4 & ROI-5; and for longitudinal strain: ROI-2 and ROI-3.

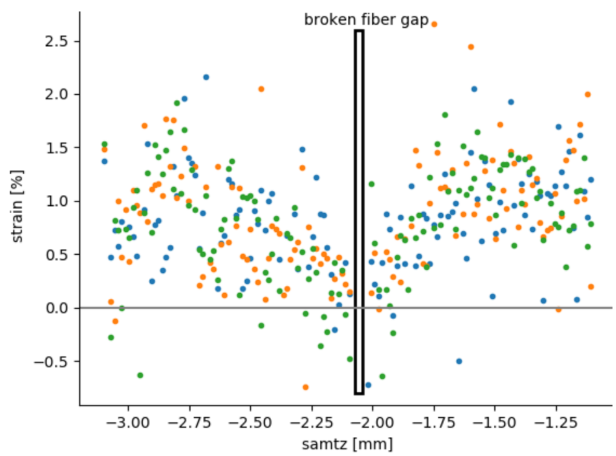

Fig. 3. Longitudinal strain evolution along a broken fibre for a single-fibre composite for 2 different analysed peaks.