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Multi-instrument multi-scale experimental damage mechanics for fibre reinforced composites

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Abstract. Reliable investigation of damage in fibre reinforced composites requires concurrent *in-* and *ex-situ* application of multiple instruments at different scale: digital image correlation, acoustic emission registration, optical/electron microscopy, C-scan, X-ray imaging and micro-computed tomography. The multi-instrument experimental mechanics allows detailed damage monitoring and inspection.

1. Introduction

The field of experimental mechanics of fibre reinforced composites is confronted with a challenge of damage observation, quantitative characterisation and interpretation not only *post-mortem*, but also during the test. Even at early stages of the specimen loading, with the average specimen deformation of a few thousand micro-strain, the composite starts experiencing cracking at the micro- and meso-scales. This involves multiple interacting damage modes at different scales, such as intra-ply and intra-yarn matrix cracking, fibre-matrix debonding, shear band formation and cracking in the matrix pockets, local and large scale delaminations and stochastic strength-controlled fibre breaking, all leading to specimen failure.

The paper presents several case studies where a multi-instrument methodology is applied to characterisation of the damage initiation and development at different scales (including matrix and fibre cracking dynamics) in composites with various thermoset and thermoplastic matrices, reinforced with glass, carbon and flax fibres. More specifically, the following cases are presented:

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- Formation of fibre break clusters observed under synchrotron radiation computed tomography (CT) [1][13];
- Micro-damage on fibre-matrix interface, observed using micro-digital image correlation (DIC) under SEM [2,3];
- Damage thresholding and identification of successive damage phases with concurrent use of acoustic emission, *in-situ* DIC and microscopy, post-mortem microscopy and X-ray inspection, in quasi-static [4-7] tensile tests, including materials with manufacturing defects [8] and fatigue loading [9];
- Delamination onset and propagation in fatigue, identified with DIC [10];
- Thermal cycling and cure-induced cracking via X-ray micro-CT [11].

The paper discusses realisations of the multi-instrument multi-scale experimental mechanics and potential future developments.

2. Formation of fibre break clusters observed under synchrotron radiation CT [1]

Synchrotron radiation CT resolves limitations inherent to standard X-ray micro-CT equipment: long acquisition times and limited space for in-situ testing rigs due to the conical geometry of the beam. Here the beam can be monochromatic and have high brilliance, which allows acquisition times for a complete CT volume of less than a second [12]. The parallel nature of the beam allows to increase the distance between specimen and scintillator, which means more space for loading rigs. Synchrotron radiation computed tomography is therefore a powerful tool for performing in situ tests. A particular case where this can be exploited is in the detection of fibre break development during testing [1, 13].

In [1], a composite laminate was strained and then held at displacement to scan the specimen. Several such steps were done at different load/strain levels to monitor how fibre breaks develop and cluster together. This study revealed new insights into the types of clusters that develop appear (see Figure 1). About 70% of the clusters were found to be co-planar (see Figure 1b) compared to 20-30% in model predictions. This discrepancy between experiments and models has triggered a new body of research trying to identify its potential causes.

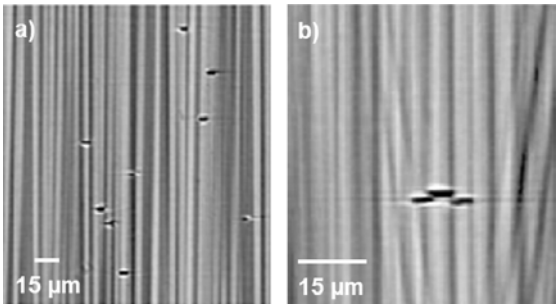


Figure 1. Two types of fibre break cluster found in the synchrotron radiation CT: (a) a diffuse cluster and (b) a co-planar cluster (reprinted from Elsevier with permission).

Similar synchrotron CT measurements were also used in a benchmarking exercise to compare three state-of-the-art models for longitudinal strength of unidirectional composites [13]. Having such data available allowed the models not just to compare fibre break and cluster predictions against each other, but also against experiments.

A second version of this exercise is currently running within FiBreMoD - Marie Skłodowksa-Curie European Training Network. A key improvement targeted within this project and exercise is to avoid the hold at displacement scenarios during scanning. Recent advances at the TOMCAT beamline at the Swiss Light Source synchrotron make it possible to continuously acquire images at reasonably high speeds, therefore enabling realistic strain rates and avoiding the need to hold the sample at load [14]. This allows more scans to be performed on the same specimen, and to scan closer to final failure where

large amounts of fibre breaks and clusters can be observed. This methodology will improve the accuracy and reliability of the experimental results and therefore help to advance fibre break models.

Opportunities for more detailed and routine quantitative analysis of CT time series can be identified in the future, exploiting digital volume correlation methods [15], suitably adapted to address the anisotropic and relatively regular microstructure that fibre reinforced composites may present, particularly at spatial resolutions necessary for fibre-by-fibre damage analysis.

3. Micro-damage on fibre-matrix interface, observed using micro-DIC under SEM [2]

The potential of micro-scale DIC to analyse deformation in fibre reinforced composites on the micro scale, including the influence of added nano-reinforcement (carbon nanotubes, CNT) was evaluated in [2, 3].

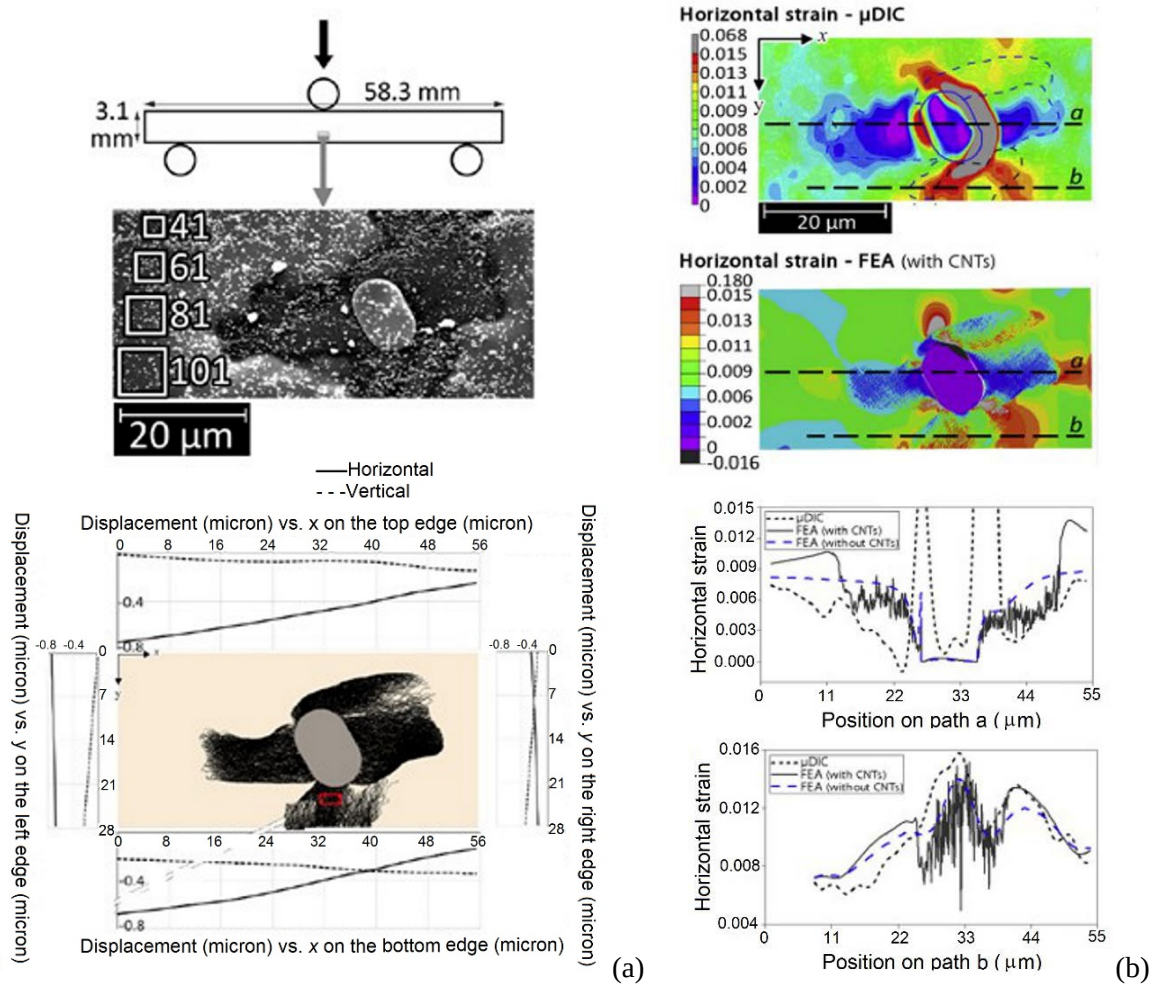


Figure 2. Micro-DIC discovering fibre debonding: (a) schematics on the specimen in three-point bending, region of interest (ROI) and FE model simulating CNTs grown on the surface of the fibre, with the DIC-measured displacements profiles on each edge of the ROI; "horizontal" and "vertical" correspond to the image orientation; (b) DIC-measured and calculated strain fields and profiles [2]

An important pre-requisite for successful DIC measurement is a high-quality random speckle pattern (produced by deposition of alumina nanoparticles), combined with optimized DIC parameters and proper microscopy settings.

Figure 2 illustrates results of the measurements. Features such as fibre, matrix, aligned CNT forest, and debonded regions at the fibre/matrix interface were detected. Openings as small as 35 nm in the debonded regions near the fibres were measured. The image correlation uncertainty in the displacement analysis was found to be below 5 nm, as estimated from the standard deviation confidence of the match. The micro-DIC results were compared with predictions of a two-scale numerical model, and a good agreement between the two was observed.

Micro-DIC measurements revealed propagation of the debond around and/or along the fibre, which manifested itself in a non-linear trend in the opening of the debonded region as function of the applied deformation.

The authors [2] concluded that the micro-DIC was found to be a promising instrument for evaluation of the effects of nano-scale reinforcements on the deformation of hierarchical materials. Further progress in mDIC is needed, particularly in the improvement of correlation methods to distinguish different phases, on which subset averaging is performed. This will allow even higher precisions in capturing deformation gradients at interfaces of materials with high mismatches in properties.

4. Concurrent use of acoustic emission, *in-situ* DIC and microscopy, post-mortem microscopy and X-ray inspection: quasi-static loading

The sequence of damage events in cross-ply and textile laminates [16] (Figure 3) suggests a presence of two thresholds of the applied load. The first, designated as ε_1, corresponds to the onset of the transverse cracking (*t*-cracks), which may not at this stage span the whole width of the specimen, being limited by the yarn crimp and/or presence of stitching in the textile reinforcement structure. The second, designated as ε_2, corresponds to, on one hand, the onset of local delaminations (*l*-cracks) and, on the other hand, to the formation of "strong" transverse cracks, which span the width of the specimen.

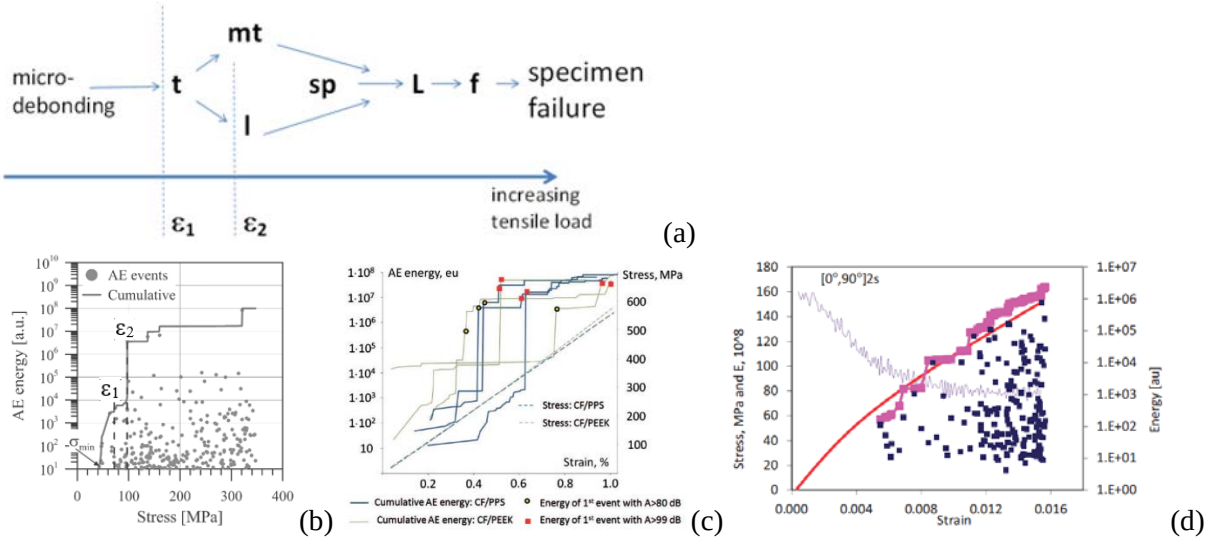


Figure 3. Damage thresholds in cross-ply and textile laminates identified with AE: (a) a sequence of damage modes (*t*: transverse cracks in yarns/plies and the matrix (*mt*); *l*: micro-delaminations; *sp*: longitudinal splitting; *L*: macro-delaminations; *f*: fibre breakage) [16]; (b) typical AE identification of the damage thresholds in a woven glass/epoxy laminate [9]; (c) damage thresholds in carbon/PPS and carbon/PEEK woven laminates [5]; (d) stress-strain, stiffness-strain and AE energy diagrams for tensile loading of flax quasi-UD woven laminates [6]

The damage thresholds can be identified using acoustic emission (AE) registration during the tensile loading, namely, a curve of cumulative energy (E) of the AE events. The damage threshold ε_1 is related to the first increase of the $E(\varepsilon)$ curve, and the damage threshold ε_2 – to the second sharp increase of this

curve. This approach was used in 2005-2018 for various textile composites; Figure 3 gives examples for such measurements for composites with carbon, glass and flax fibres with thermoset and thermoplastic matrices. The AE registration allows quantification of the influence of the reinforcement architecture, matrix ductility or fibre properties on the damage initiation behaviour of the composite.

However, use of the cumulative AE energy remains a heuristic approach for the identification of damage thresholds. To be convincing, it is better supported by direct evidence of the damage. In [3] microscope observation of the specimen edge was combined with AE for carbon/PPS woven laminates, in [17] – for carbon/epoxy woven laminates. The crack development observed in a small central portion of the specimen edge was connected to the AE events located in the same zone of the specimen. The number of new intra-yarn matrix cracks generated at a certain load level was compared to the number of acoustic event localized in the zone of observation, with a good agreement found between the number of AE events with low amplitude and low frequency and the number of observed cracks (Figure 4a-c).

The observation of cracks on the specimen edge can be enhanced with the use of DIC. In [8], the cracks were monitored via identification of peaks in the strain profile on the specimen surface in each deformation step, which is carried out semi-automatically. The method was applied to study the influence of voids on the cracking process (Figure 5).

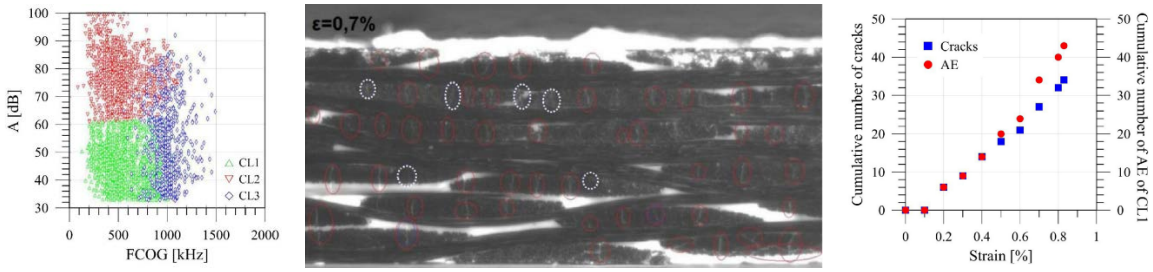


Figure 4. Concurrent use of AE and edge microscope, tension of carbon/PPS woven laminates: (a) clusters of AE events; (b) cracks observed at the specimen edge; (c) correlation between the number of cracks in an observation zone of 5 mm length and the number of AE events [4].

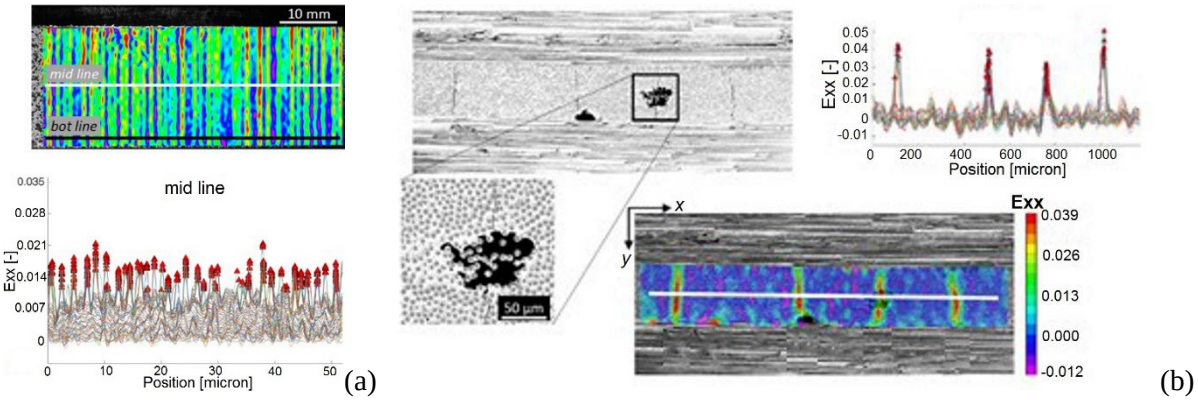


Figure 5. DIC-enhancement of the edge crack registration: (a) cracks identified as peaks on the strain profile; (b) cracks related to voids in 90° plies [8].

With the laminate structure becoming more complex, the link between the AE events signature and the damage type becomes more complex. The AE registration technique alone during tension tests is not sufficient for damage mode identification in CFRP composites. Even though clustering algorithms can recognise well-separated and dense clusters, in [6] it is found that those clusters cannot be unambiguously used to identify the type of the damage in quasi-isotropic (QI) laminates. This study

presents the necessity of multi-instrument optical observations for damage-mode identification and correlation with AE registrations: concurrent use of AE registration, optical edge observations and surface strain mapping by DIC (Figure 6).

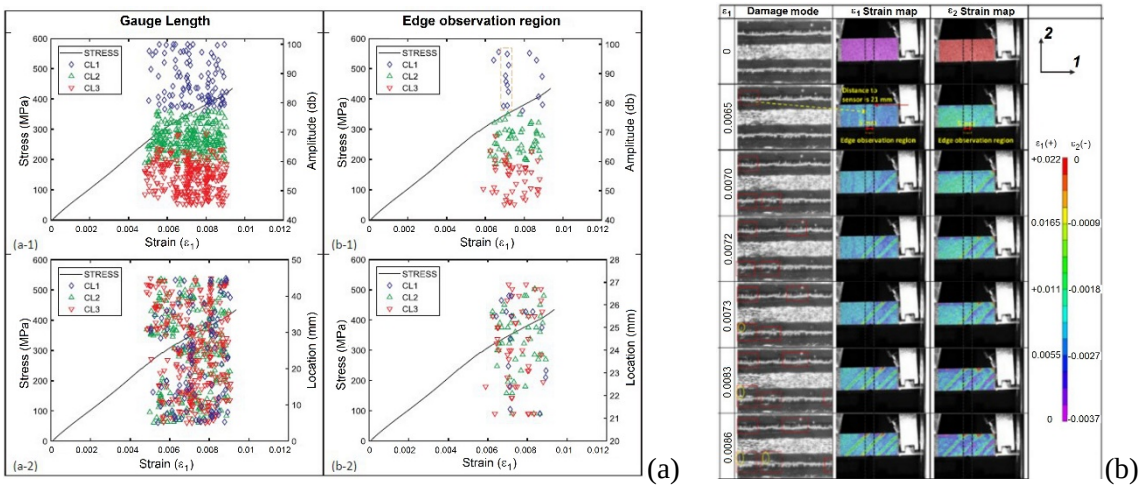


Figure 6. Concurrent use of AE (a), edge microscopy and surface DIC (b) for damage identification during tension of carbon/epoxy QI laminates [7].

5. Delamination onset and propagation in fatigue, identified with DIC [10]

Optical methods are effectively used for following tracking damage development in fatigue loading. In [9] a DIC-based method is proposed to characterize Mode I fatigue delamination onset and propagation in laminated composites. Images of the fatigue delamination on one side of the specimen were automatically recorded at a maximum displacement at specific fatigue cycles. The displacement field around a delamination crack is obtained and further processed to determine the position of the crack tip (Figure 7a). With this method the delamination length can be measured automatically in each cycle with a precision on the order of few hundreds of micrometers. The fatigue delamination onset life is then determined by detecting the increase of the delamination length, and the fatigue delamination propagation rate is calculated. The proposed method produces more conservative fatigue life measurements in comparison with the 5% compliance increase method in ASTM D6115 (Figure 7b).

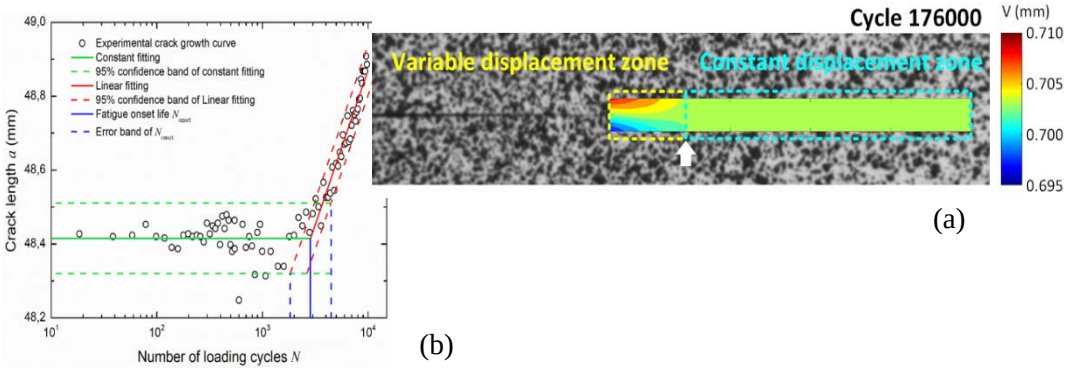


Figure 7. DIC-based method for tracking a fatigue Mode I crack: (a) detection of the crack tip position (white arrow); (b) identification of fatigue onset life N_{onset} [10].

6. Thermal cycling and cure-induced cracking via X-ray micro-CT [11]

The thermal cycling behaviour of fibre reinforced composite can be effectively studied using X-ray micro-CT. The key difficulty is an effective automated recognition of the cracks.

In [11] a carbon fibre/epoxy matrix composite material reinforced with a non-crimp 3D orthogonal woven preform was investigated. The aim was to characterize the damage mechanisms – i.e. matrix cracking – induced by thermal cycling. Qualitative and quantitative descriptions of the morphology and the evolution of cracks with thermal cycling are carried out through the analysis of μCT scans of samples at different cycle numbers. Through suitable image processing, which is of general and wide application, qualitative and quantitative descriptions of the initial porosity, of morphology and evolution of matrix cracking up to 1400 thermal cycles were performed (Figure 8a).

In addition, since the specimens have a certain level of porosity due to the infusion process, a complete description of this defect is carried out, and its influence on the damage mechanisms induced by thermal cycling is analysed. At first sight, the presence of pores seems to facilitate the propagation of cracks, pores acting as “attractors” when they are in the vicinity of the crack path (Figure 8b).

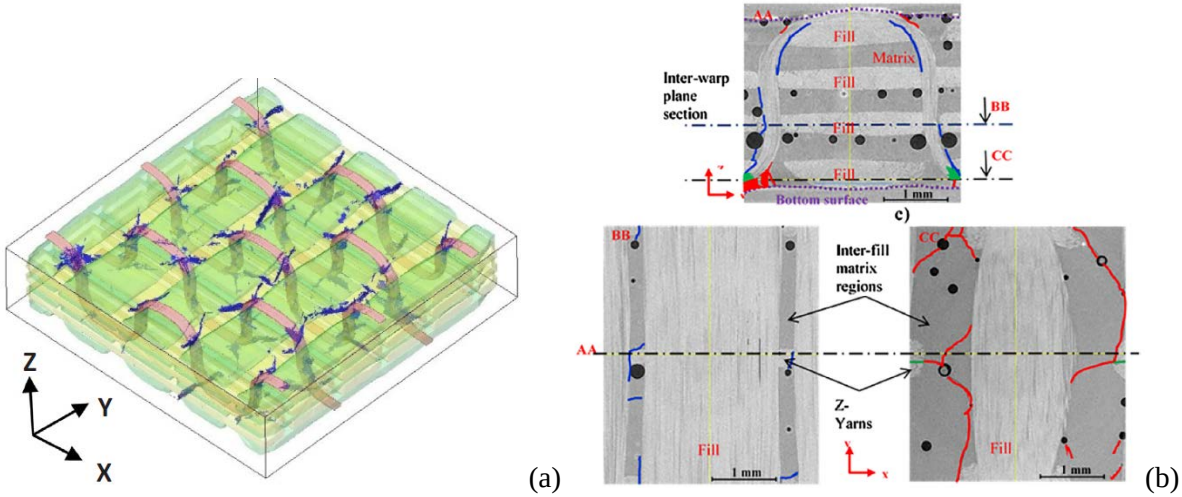


Figure 8. Thermal fatigue of a 3D woven carbon/epoxy composites in the presence of voids: (a) cracks identified using micro-CT; (b) details of the crack morphology [11]

7. Conclusion

Comprehensive methods of the multi-instrument experimental damage mechanics in fibre reinforced composites involve concurrent application of multiple in- and ex-situ instruments at the scale of the specimen and at the micro-scale: digital image correlation, acoustic emission registration, optical/electron microscopy, C-scan and X-ray imaging and micro-computed tomography.

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