DIAMETRAL COMPRESSION TESTS OF A PULTRUDED PA6 REINFORCED GLASS FIBRE COMPOSITE: A MACRO AND MICRO TRANSVERSE DAMAGE KINETIC INVESTIGATION

H.A. Cayzac[a], S. Joannès[a*], L. Laiarinandrasana[a]

[a] *Centre des Matériaux, Mines ParisTech, CNRS UMR 7633 BP 87, 91003 Evry Cedex, France*
** sebastien.joannes@mines-paristech.fr*

Keywords: microstructure, triaxiality, damage, tomography.

Abstract
The present contribution is devoted to highlight the microstructural effects on the transverse behavior of a pultruded rod. A diametral compression test is used to impose a triaxial loading within the material and follow the damage kinetic by SEM and X-tomography techniques. A visco-plastic multi-mechanisms model is implemented to simulate this microstructural behavior.

1. Introduction

Due to their high strength and low density, the use of organic composite materials for stationary or on-board gaseous storage vessels, and for gaseous transportation have recently known a growing interest. New designs are emerging with an inner polymeric core which provides hermetical sealing and a wrapped composite material enhancing mechanical properties. For pressure vessels, the fibres are wound on geodesic paths so that when the cylinders are under pressure the fibres experience tensile forces, due to a bi-axial state of stress (for thin walls), with failure being due to tensile forces in the fibre direction. Nevertheless, for thicker walls, these structures are submitted to both outer and inner pressures (up to a few hundreds of MPa) that exploit the transversal properties of the composite material. Indeed, transverse cracking plays a key role in a fatigue damage process and transverse cracks could lead to fluid leakage of pressure pipes or vessels.

The material under study is a thermoplastic polymer matrix polyamide 6 reinforced with unidirectional continuous glass fibres with a high fibre volume fraction (more than 65%). The present contribution is devoted to highlight the micromechanical transverse damage kinetic of such thermoplastic composites since they offer a macroscopic transversal ductility which is quite different from the sudden death of their thermoset counterparts. The composite studied is obtained thanks to an in-situ polymerization pultrusion process which leads to very high local fibre volume fractions up to 80%. High fibre density regions lead to highly constrained matrix which has been shown to be sensitive to hydrostatic pressure. Since this thermoplastic pultrusion process does not allow the production of wide composite plates (only thin strips or rods), the transverse experimental investigation is performed thanks to diametral compression tests (Brezilian tests).

2. Experimental techniques

2.1. Material microstructure

The material under study is provided as a 8 mm diameter rod. Transverse cross-sections of undamaged PA6GF composite were observed thanks to Scanning Electron Microscopy (SEM) techniques and X-ray microtomography. For SEM images, composite samples are embedded in a polymeric matrix and carefully polished. Figure 1 shows the microstructural images of an undamaged rod at different scales: (a) displays a 2D cross-section obtained from an X-ray microtomography scan of the rod, whereas (b) and (c) are SEM images. Figure 1 (b) clearly exhibits high microstructural variabilities. Indeed different fibre diameter can be observed and the diameter distribution was quantified on several SEM microstructure images thanks to the use of digital image processing. The average diameter size is 15.1±0.05µm. The local fibre volume fraction varies from 40% to 80%.

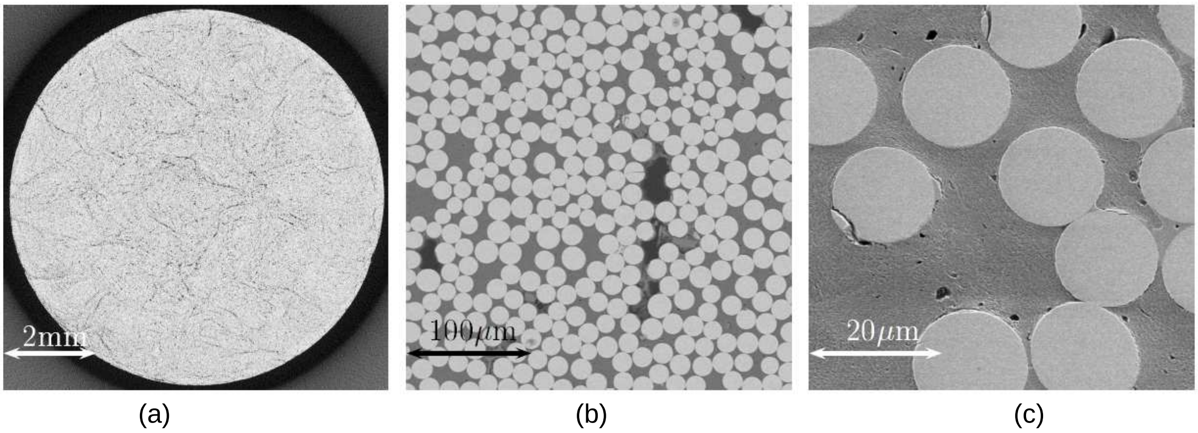
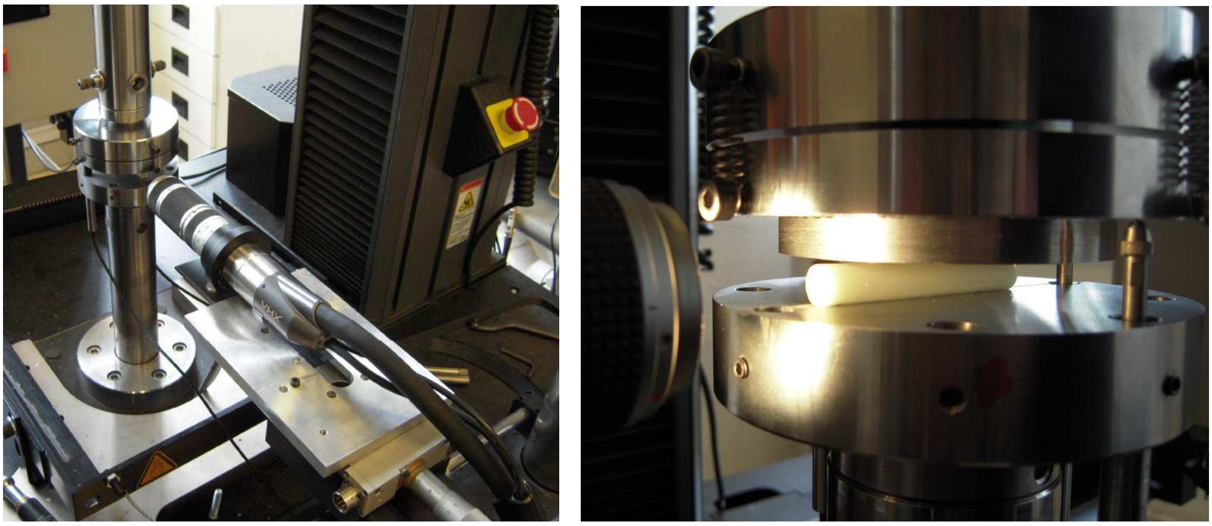


Figure 1. Microsctructural images of an undamaged composite rod: (a) full cross-section obtained by X-ray microtomography and details of the microstructure (b) and (c) obtained by SEM. Both fibre volume fraction variability and voids can be seen.

Voids can also been observed, they take place in the polymer matrix between fibres (b) and (c). Voids have various diameter sizes ranging from few µm to 100 µm. Longitudinal sections showed that “macro-voids” have “rice grain” shapes, i.e. the longitudinal size is much longer than the diametrical one. Such macro-voids are due to the pultrusion process when air bubbles get stuck between fibres. “Micro-voids” are also visible in (c) probably resulting from the polymerization process.

2.2. The diametral compression test

A mechanical testing machine (Instron 5982) was used to apply a diametral compression to the samples at a loading rate of 1 mm/min. The upper platen has a ball joint in order to fit closely to the rod surfaces. Four springs provide enough stiffness to allow a good contact on the rod for the early loading stages. A digital miscroscope (Keyence VHX 600) was used to record images of rods under load (as shown is Figure 2). A Linear Variable Differential Transformer (LVDT) was used to record the applied displacement while a load cell recorded the resulting load. The investigated test specimen length is 40 mm.



(a) (b)

Figure 2. The diametral compression test equipment: the rod is compressed between to steel platens with a ball joint in order to fit closely to the rod surface. A digital microscope is used to get images of the rod cross-section and an LVDT extensometer records the displacement.

Figure 3 displays a normalized load versus applied displacement curve. The curve presents different stages. The first stage is the elastic part of the curve. The second stage corresponds to the beginning of the non-linear part (around an applied displacement of about 0.4 mm) up to the peak load (applied displacement of about 0.5 mm). The third stage is the tremendous decrease after the peak. The last stage is the slow decrease in the load.

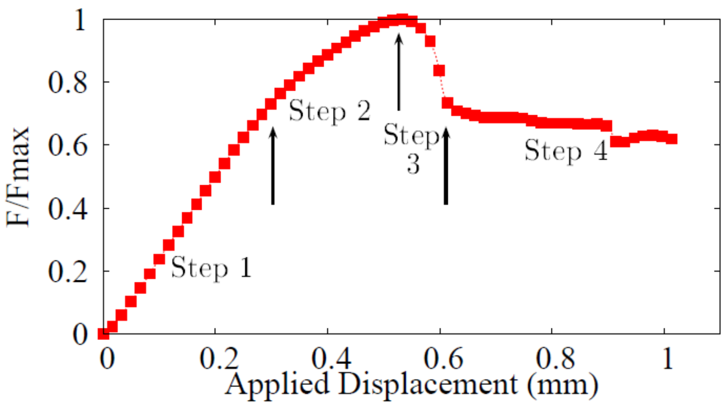


Figure 3. Normalized load versus applied displacement curve of the diametral compression test. Four stages are visible and the transverse behavior of the composite is rather ductile.

This behavior is rather ductile due to the particular properties of the thermoplastic matrix. Indeed [1] and [2] did not observed such a behavior for an epoxy matrix composite rod. Glass or carbon fibres reinforced epoxy matrix composite rods exhibited a rather fragile behavior with a load response linear up to the failure of the specimen.

Submitted to diametral compression, the composite rod stress field is composed of tensile and compression stresses. Fissuring is expected to occur in the region where tensile stress is maximum i.e. along the diameter plane.

In order to investigate these micromechanisms within the composite material, interrupted diametral compression tests coupled with micro-CT inspections have been performed. Five different interrupted tests have been carefully selected for X-ray microtomography inspections. The main idea was to be able to identify damage evolution on the non-linear part of the load/displacement curve. Figure 4 (a) displays the five applied displacements versus resulting load curves for each interrupted specimen of interest. The applied displacements have been shifted for each curve in order to clarify the graph. The experimental procedure consists of loading each specimen to a desired applied displacement "*d*" and to unload them for observation. The first specimen is loaded up to the end of the elastic part of the curve (maximum applied displacement *d*=0.31mm). The second specimen is interrupted on the first non-linear stage (*d*=0.40mm). The third rod is loaded up to the peak and is then interrupted (*d*=0.47mm). The fourth specimen is stopped at the beginning of the load drop (*d*=0.51mm). The last diametral compression test have been interrupted a little further on the load drop (*d*=0.57mm). Each unloaded samples are then ready for micro-CT inspections. The scanned area is located in the vicinity of the median cross section of each rod.

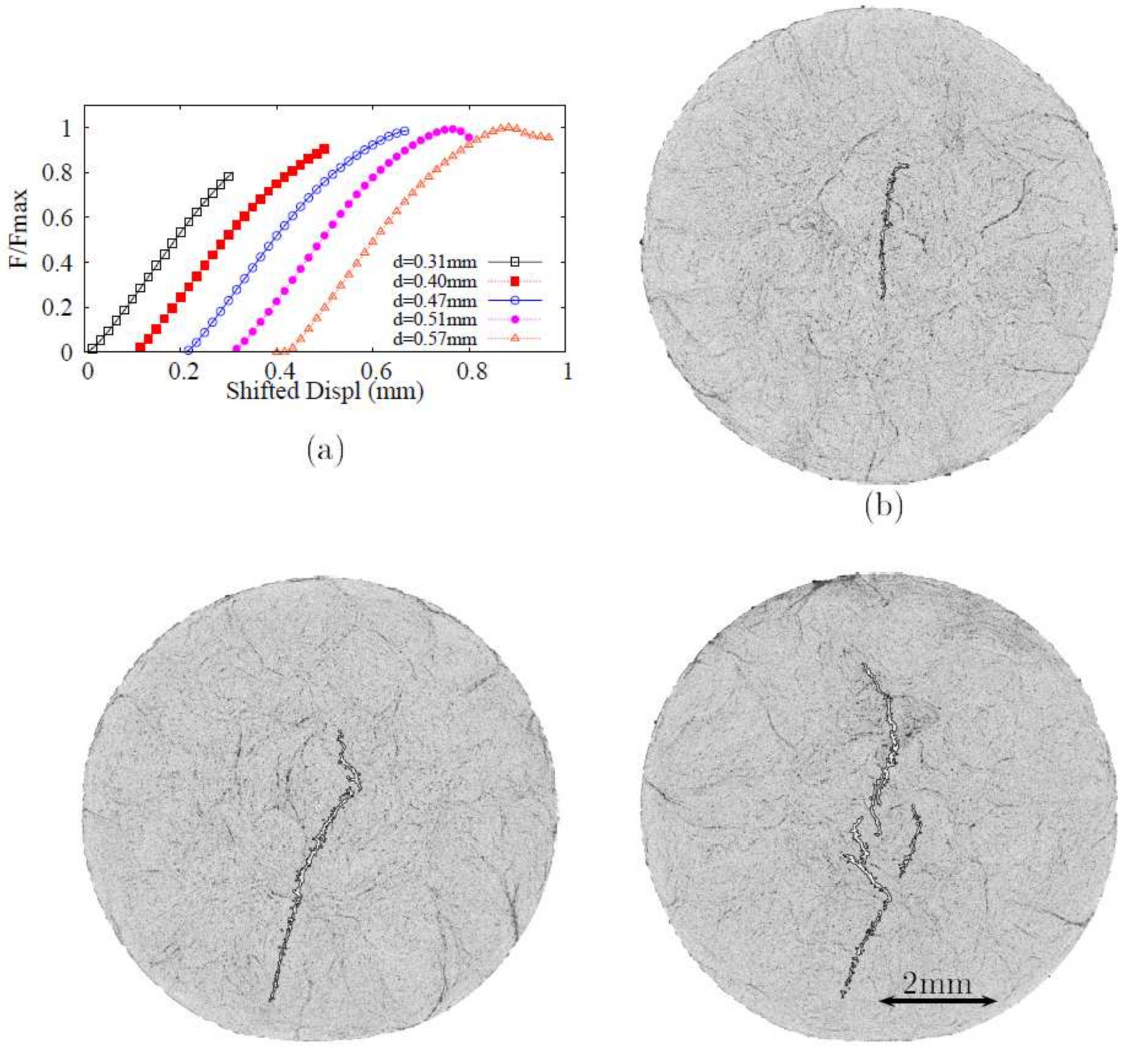


Figure 4. (a) Normalized load versus applied displacement curve of the five interrupted diametral compression tests (the applied displacements have been shifted for each curve in order to clarify the graph). (b) From top to bottom and left to right, X-ray microtomography scanned areas located in the vicinity of the median cross section of each rod for *d*=0.47mm, 0.51mm and 0.57mm. The material clearly exhibits several cracks in the diametrical plane of the composite material which are parallel to the loading axis.

The material clearly exhibits several cracks in the diametrical plane of the composite material. These cracks are parallel to the loading axis. The use of X-ray tomography techniques allowed examinations of cracks propagations. The damage initiates within the polymer and not at the fibre/matrix interface. A multi-cracks initiation can be observed and the main crack always propagates through the matrix starting from macro voids. Figure 5 clearly shows the two sides of a transverse crack observed by SEM. A strong cohesion is observed between the fibres and the polyamide matrix, this characterized a cohesive failure within the polymer.

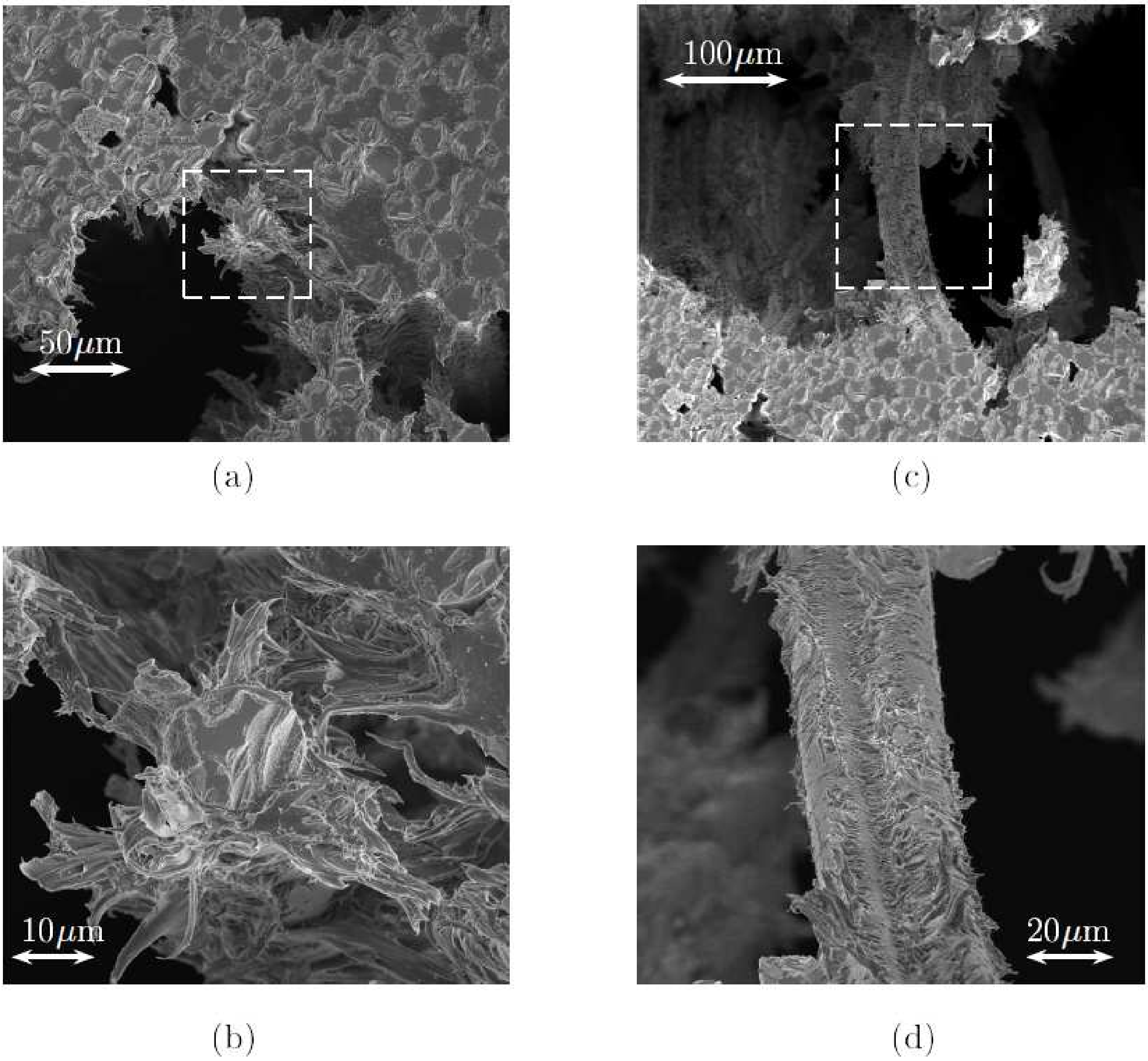


Figure 5. Post-mortem observations of a transverse crack.

3. Understanding and modeling the transverse damage kinetic

3.1 Triaxial state of stress

Recent studies on bulk polymer materials have shown that stress triaxiality ratio (hydrostatic pressure) have a strong influence on damage mechanisms [3][4][5]. Whitening of polymers was related to void growth leading to a significant volume change in the polymeric material [6]. It was reported that the hydrostatic pressure played a major role on the damage growth mechanisms. SEM analysis [3] assessed with recent X-tomography analysis on interrupted tensile notched specimens tests have showed the microstructural evolution of polymer PA6 or PA11 in deformation. In addition the damage distribution (voids nucleation and growth) were spatially and chronologically quantified.

Based on those observations, a viscoplastic multi-mechanism based model was implemented in an in-house Finite Element Model (FEM) code in order to simulate the thermoplastic semi-crystalline polymer mechanical response submitted to complex triaxial loadings (due to the fibre packing). A damage based model incorporating the porosity as a damage variable was set up to simulate the damage mechanisms. The present constitutive model is able to distinguish the location of maximum damage depending on the hydrostatic pressure. A Representative Volume Element (RVE) was investigated in order to study the complex failure of such a composite material. This RVE takes into account both local fibre volume fraction and fibre diameter distributions. Defects can be taken into account by including a distribution in the initial damage variable value. Computations were then carried out to better understand the influence of hydrostatic pressure on the transverse behavior of polyamide reinforced composite materials. The simulation is built on a 2 scales analysis: the compression test is simulated in order to compute the state of stress within the composite rod by using a phenomenological non-linear behavior. The maximum stress is obtained for the center of the rod which is then loaded at the micro scale (Figure 6). A real microstructure mesh is used to observe the damage multi-crack initiation and the crack propagation through the matrix. Due to the fibre packing, the stress triaxiality is very high between fibres and leads to a diffuse damage initiation (figure 7).

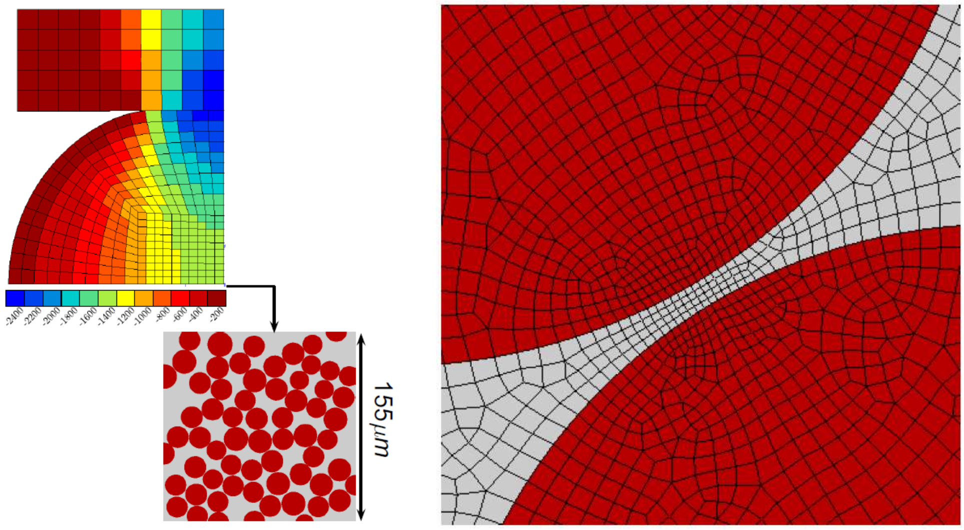


Figure 6. A two scales analysis is performed to account for the damage kinetic within the composite material.

Conclusion

Microstructural variability of pultruded glass fibres reinforced polyamide was quantified thanks to SEM image analysis. A great interest was given to the local fibre volume fraction distribution. In situ damage evolution within rods subjected to diametral compression tests was characterized with X-ray tomography technique. The damage initiates between packed fibres due to the high triaxiality stress state. This multi-crack mechanism is then followed by damage propagation through the polymer voids leading to crack branching. A constitutive damage based model was investigated in order to simulate the polymeric matrix behavior and account for those observed damage mechanisms at the microscale. The local fibre volume fraction has a strong influence on the hydrostatic pressure and thus on the damage initiation.

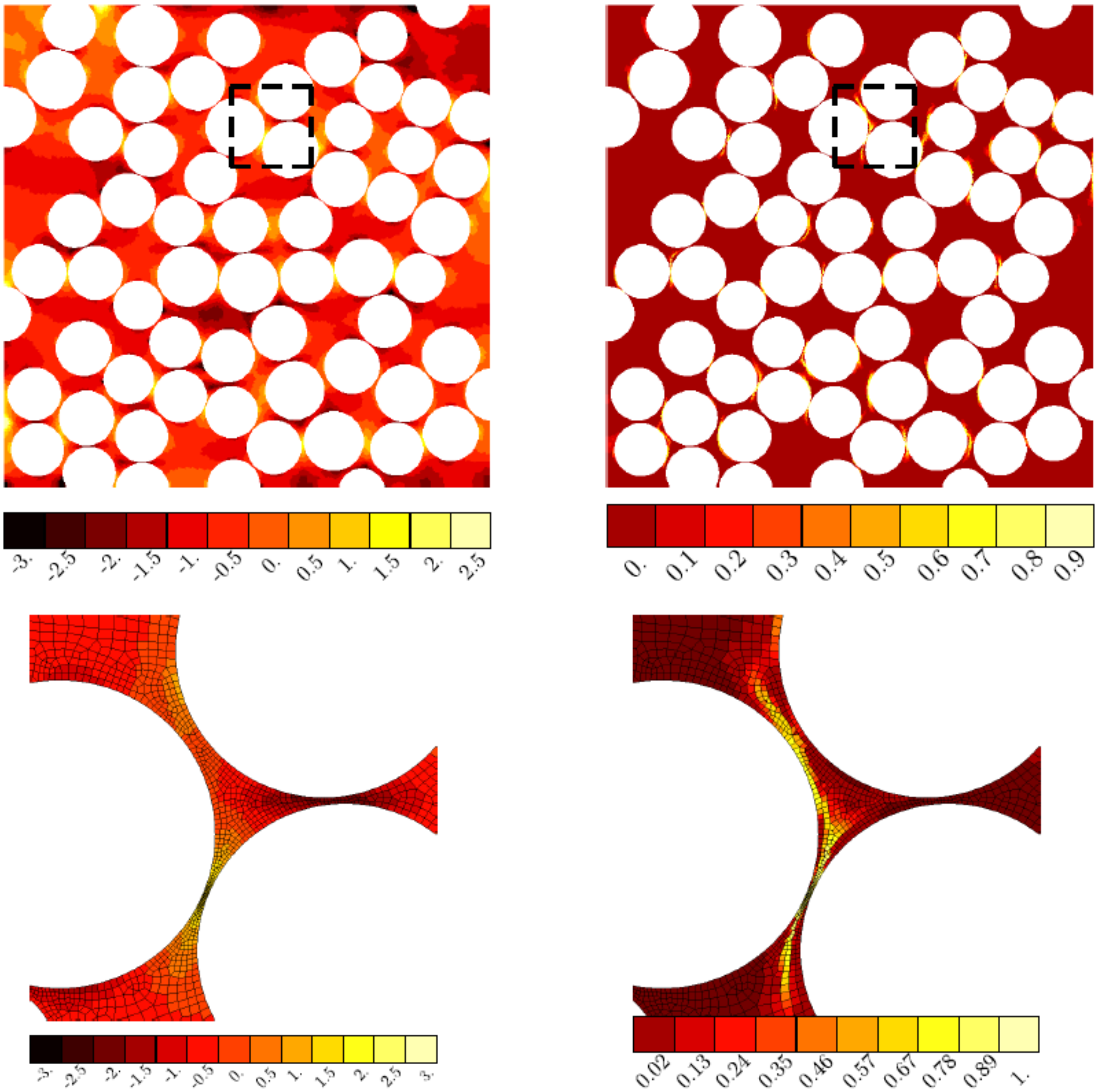


Figure 7. Left: triaxility within the microstructure. Right: damage initiation and propagation.

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