DAMAGE MECHANISMS INTO SHORT GLASS FIBRE REINFORCED THERMOPLASTIC DURING IN SITU MICROTOMOGRAPHIC TENSILE TESTS

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Abstract

Short glass fibre reinforced thermoplastics are promising materials for weight reduction of structures thanks to its very good specific mechanical properties. The current challenge is to explain and show their damage mechanisms regarding spatial configuration of the microstructure, in order to be able to predict final degradation. A compact tensile machine has been designed in order to investigate damage mechanisms during in situ microtomography test. The gage length of the specimen has been scanned at different stages of deformation, obtaining 3D-pictures of damage localisation with a resolution of $0.7\mu m$*. Experimental data confirms some of the mechanisms described in the literature but additional role of fibre failure modes are evidenced. The main point is that damage location not only depends on position around the fibre and distance with other fibres, but also of disorientation between groups of fibres and their first neighbours. Damage localisation can be explained by comparison between damage localisation and stress field induced by microstructure.*

1. Introduction

With increasing constraints of lightening in industrial fields, mechanical properties are now considered regarding material density. This trend ranks the short glass fibre polyamide 6,6 among very promising materials. Its complexity mainly comes from its microstructure. The injection process, perfectly suited for high productivity and complex shapes, induces heterogeneous distribution and orientation of fibres. Furthermore, it is well admitted that this microstructure plays an important role on damage mechanisms.

Despite significant work, there still is a lack of explanation about the link between spatial configuration of this microstructure fibres and damage mechanisms. Evolution of material process technology has to be considered. The final aim of the work is to predict fatigue life of industrial parts. This paper presents the first results concerning the use of x-ray microtomography to describe damage mechanisms in relation with microstructure.

A description of damage chronology has been made by Horst [1] and Sato [2, 3] for tensile

stresses. They highlight main damage mechanisms, in a localized region subjected to stress concentration : 1) Initiation of interfacial microfailure at the fibre tips. 2) Propagation of interfacial microfailure along fibre sides. 3) Occurrence of plastic deformation band in matrix region. 4) Crack opening and slow crack propagation. 5) Fast crack propagation.

From these descriptions, damage is associated to void apparition in the material. In accordance to the objective of the study (i.e. spatial configuration of damage with respect to fibre organisation), microtomography is the most suitable tool for 3D damage observation. As this technique is non destructive, it was possible to follow the development of damage at different levels of deformation the same part of the specimen and then, to propose a chronology of the damage evolution according to local microstructure configuration.

2. Material and methods

2.1. Microtomography

The principle of microtomography is simple: a series of N radiographs (the series is called a scan) corresponding to N angular positions of the sample with respect to the beam is recorded on a detector which is generally a CCD in modern tomographs. Those radiographs are used as input for a reconstruction software to obtain the three dimensional distribution of the linear x-ray attenuation coefficient μ within the sample. This distribution forms a 3D image which elementary unit is called a voxel and which can be viewed with appropriate imaging software. For the reconstruction step, an analytical method was used. This method is faster than algebraic ones, but requires a complete set of radiographs during the rotation and cannot deal with missing views [4]. Synchrotron sources deliver parallel x-ray beams and the analytical filtered back projection method is used to reconstruct a slice of the sample perpendicular to the rotation axis. The 3D volume is obtained by stacking the reconstructed slices.

These experiments presented in this paper were performed on ID19 beamline at the European Synchrotron Radiation Facility (Grenoble, France). A monochromatic x-ray beam was used, with a 194.77 mA intensity and a photon energy of 19 keV. A Fast Readout Low Noise (FReLoN) 14-bit CCD camera with a 2048 x 2048 pixel sensor received 2000 radiographs during rotation of the machine over 180° along vertical axis. This experimental setup was optimized to obtain a voxel edge size of 0.7 μm. The acquisition of a complete scan lasts about 9 minutes.

2.2. In situ tensile machine

A displacement controlled and force measuring machine was developed. One of the key factors regarding 3D image quality (resolution) is the distance between the specimen surface and the CCD sensor. The tensile machine was designed to minimize that distance. The loading ring was made of 2 mm thickness PMMA tube in order to limitate additional attenuation by the experimental setup.

The machine was directly mounted on the rotating stage of the beam line as shown on figure 1.

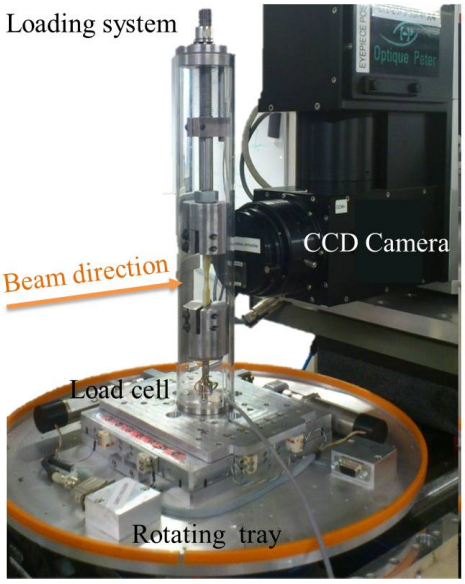


Figure 1. Compact tensile machine set up for in situ testing at ESRF ID19

2.3. Specimen

The material chosen for the study is a Technyl® A218V30, a commercial grade of polyamide 6,6 reinforced by 30wt% of short glass fibre supplied by Solvay Engineering Plastics-France. It is well known [5, 6] that relative humidity of the reinforced polyamide has strong influence on its mechanical behaviour. For this reason, specimens were conditioned at 50% of relative humidity (RH50) prior testing.

Specimens were watercut from injection molded sheets of 3.24 mm thickness. Samples were extracted following three different orientations compared to the main injection direction: 0°, 45° and 90°. Since the distribution and orientation of fibres in the sheets are heterogeneous (core-shell structure with core fibres transverse to main direction injection), extraction orientation has a first order effect on mechanical properties, as shown in table 1 for tensile tests results.

Specimen orientation	σ_R [MPa]	ε_R[%]
0°	117.5	4.1
45°	73.5	5.7
90°	64.3	6.4

Table 1. Tensile tests results according to specimen orientation of extraction

One of the limitation 3D x-ray microtomography is the limited size of observation, depending of resolution and sensor size. This constraint lead to define a particular specimen geometry: the size of the gage length had to be equivalent to observable volume with microtomography, and is illustrated given figure 2. Using this geometry ensures that the whole volume of the gage length is investigated during the test and that the damage chronology can be reconstructed.

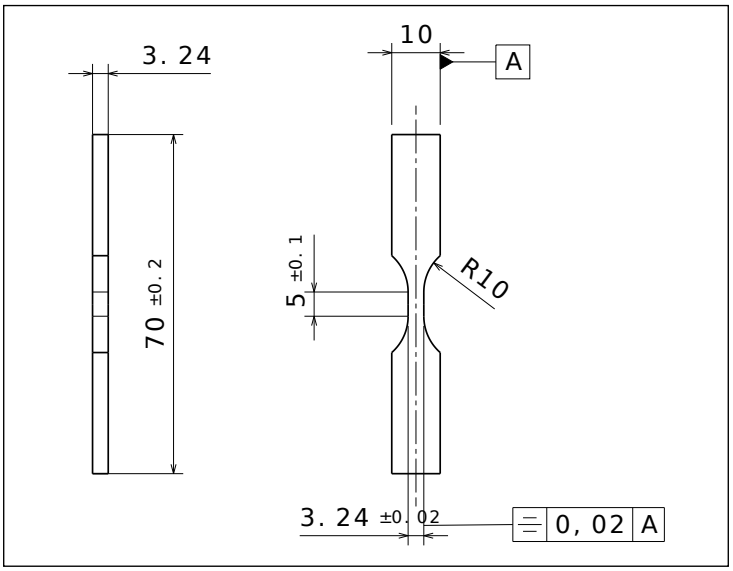


Figure 2. Specimen geometry for in situ tensile testing

3. Results and discussion

Presented results are extracted from the experiment made for 45°oriented specimen as presented in the figure 3. According to imposed displacement and measured load, first, second, third and fourth scans have respectively been pictured at strains of $\varepsilon_1 = 0\%$, $\varepsilon_2 = 0.9\%$, $\varepsilon_3 = 1.2\%$ and $\varepsilon_4 = 1.4\%$, with a strain to failure of $\varepsilon_r = 3.4\%$.

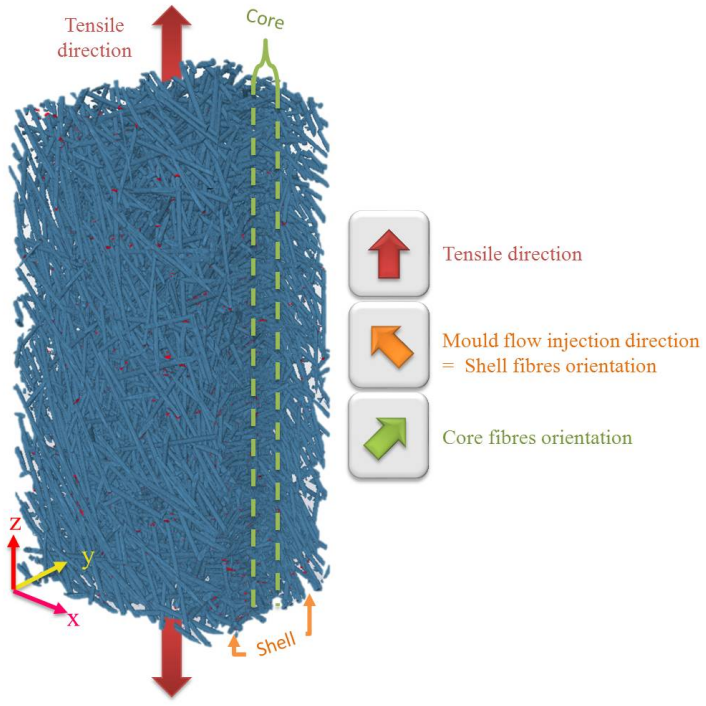
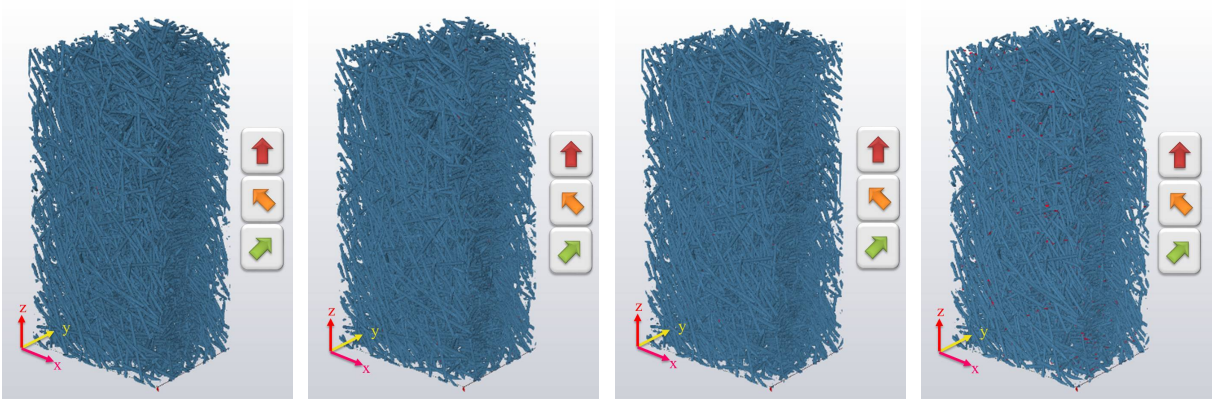


Figure 3. 3D picture obtained with microtomography. Fibres are blue, voids are red.

Figure 4 presents 3D pictures of the sample, with increasing level of strain from left to right. To obtain these pictures, appropriate thresholds have been identified for fibres and for voids. Then, only these elements have been kept. In blue, we can observe fibres, with a core-shell structure. In red, identified voids used as indicators of damage into the material.

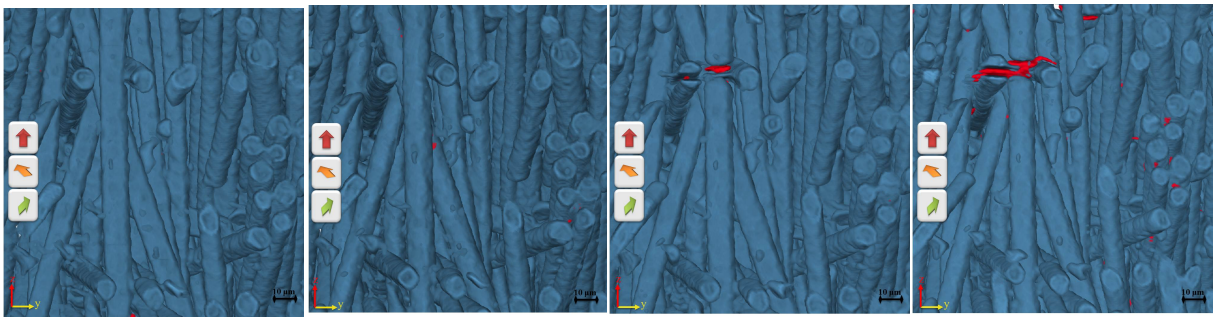


(a) $\varepsilon_1 = 0.0\%, \sigma_1 = 0MPa$ (b) $\varepsilon_2 = 0.9\%, \sigma_2 = 24MPa$ (c) $\varepsilon_3 = 1.2\%, \sigma_3 = 30MPa$ (d) $\varepsilon_4 = 1.4\%, \sigma_4 = 33MPa$

Figure 4. 3D pictures of the sample at different deformation stages of the test.

3.1. Observed damage mechanisms

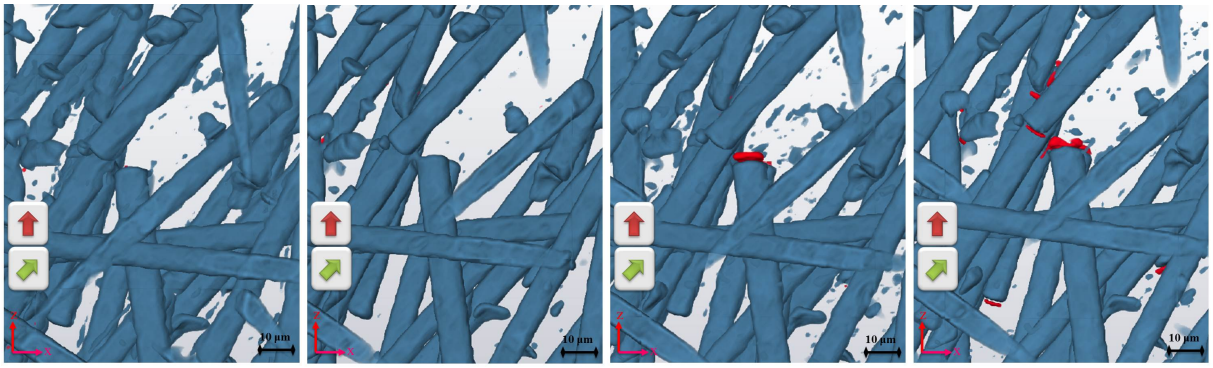
Results of the experiment confirm mechanisms described in the literature. As described by Sato [3], cohesive properties of the matrix very near to the fibre-matrix interface seem essential. Matrix cavitation and crack apparition can be illustrated by micrometric voids spheres where fibres are relatively separated. There also are star-shaped voids, which appear where two fibres are close and disoriented between them (as shown in figure 5). Then, end of fibres oriented in the same direction as stress are hardly damaged as shown in figure 6.



(a) $\varepsilon_1 = 0.0\%, \sigma_1 = 0MPa$ (b) $\varepsilon_2 = 0.9\%, \sigma_2 = 24MPa$ (c) $\varepsilon_3 = 1.2\%, \sigma_3 = 30MPa$ (d) $\varepsilon_4 = 1.4\%, \sigma_4 = 33MPa$

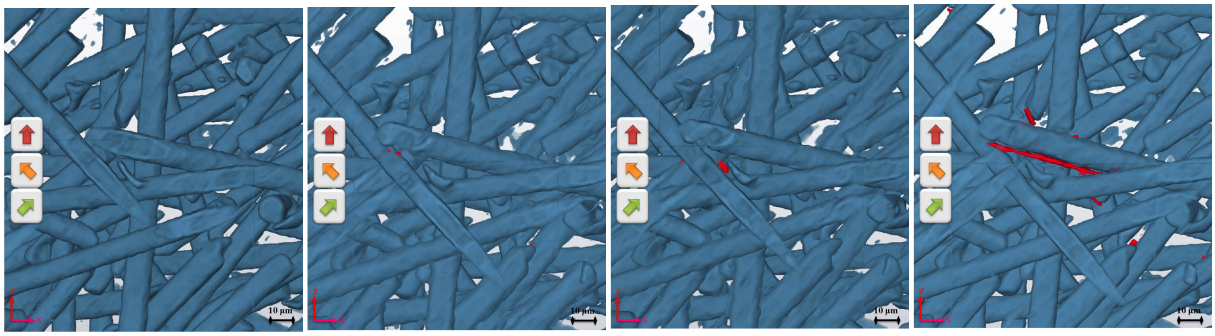
Figure 5. Broken fibre from the *yz* side, near the core-shell transition

However, mechanism descriptions of literature are questionable. The debonding along fibre sides is not consistently introduced by debonding at fibre ends (as visible in figure 7). Debonding along fibre sides highly depends on local configuration around the fibre and not only on its orientation. Indeed, debonding along fibre sides not only occurs when fibre is oriented transversely to macroscopic stress direction, but mostly when fibre is surrounded by fibres with different orientation. Furthermore, one mechanism which is non mentioned in tensile damage



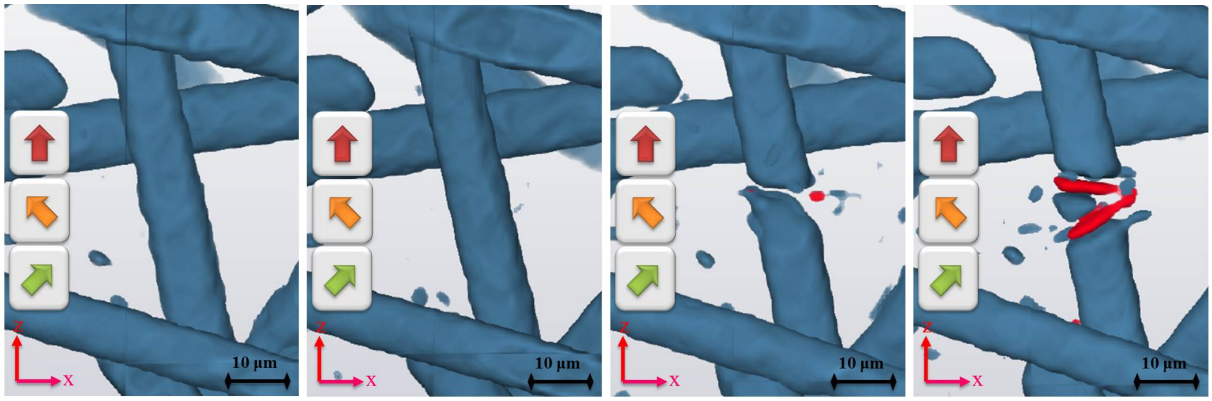
(a) $\varepsilon_1 = 0.0\%, \sigma_1 = 0MPa$ (b) $\varepsilon_2 = 0.9\%, \sigma_2 = 24MPa$ (c) $\varepsilon_3 = 1.2\%, \sigma_3 = 30MPa$ (d) $\varepsilon_4 = 1.4\%, \sigma_4 = 33MPa$

Figure 6. Damaged matrix at the end of fibres with same orientation as tensile direction, in the core



(a) $\varepsilon_1 = 0.0\%, \sigma_1 = 0MPa$ (b) $\varepsilon_2 = 0.9\%, \sigma_2 = 24MPa$ (c) $\varepsilon_3 = 1.2\%, \sigma_3 = 30MPa$ (d) $\varepsilon_4 = 1.4\%, \sigma_4 = 33MPa$

Figure 7. Damaged matrix between fibres with different orientations, at the transition between shell and core

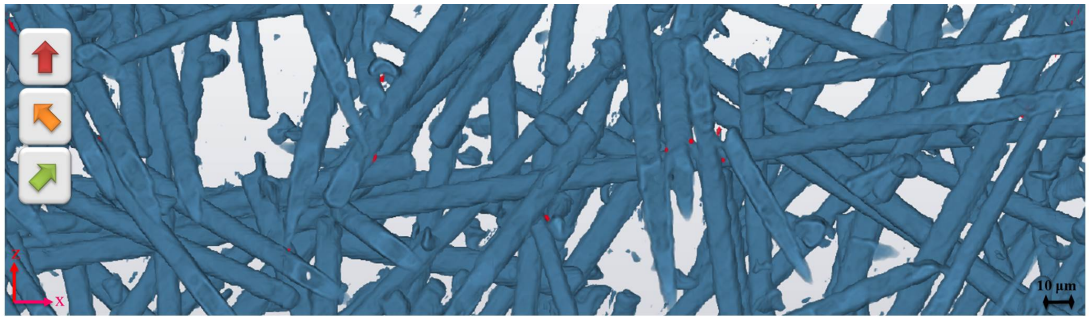


(a) $\varepsilon_1 = 0.0\%, \sigma_1 = 0MPa$ (b) $\varepsilon_2 = 0.9\%, \sigma_2 = 24MPa$ (c) $\varepsilon_3 = 1.2\%, \sigma_3 = 30MPa$ (d) $\varepsilon_4 = 1.4\%, \sigma_4 = 33MPa$

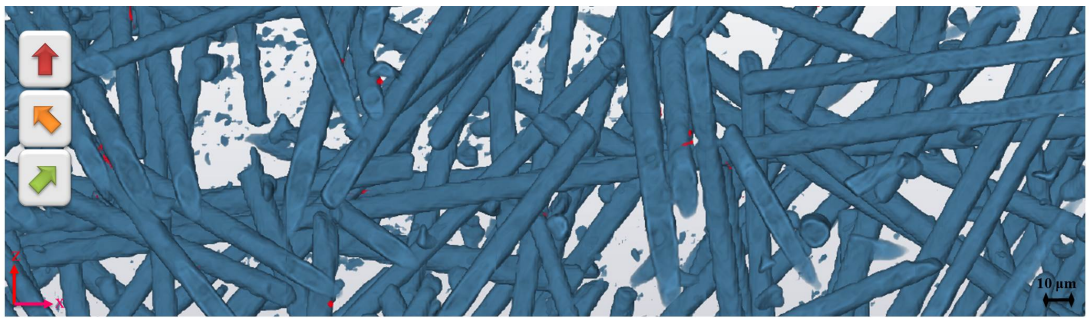
Figure 8. Rupture of a fibre oriented in the same direction as tensile strength

mechanisms for this material, has been observed and seems to contribute to failure mechanism. As a matter of fact, significant fibre breakage is largely detected, transverse to macroscopic stress direction. This mechanism is present, whether the fibre and the main stress direction are collinear (figure 8) or perpendicular (figure 9). The fact that this mechanism has not been considered yet can be explained by two points. First, the previous descriptions were limited to surface observations, where fibre failure can hardly be evidenced. Secondly, sizing or other

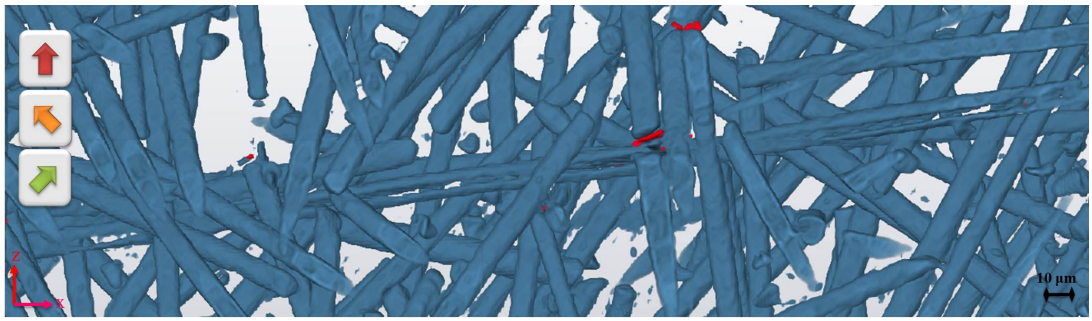
process improvements could have changed microscopic behaviour near the interface between fibre and matrix.



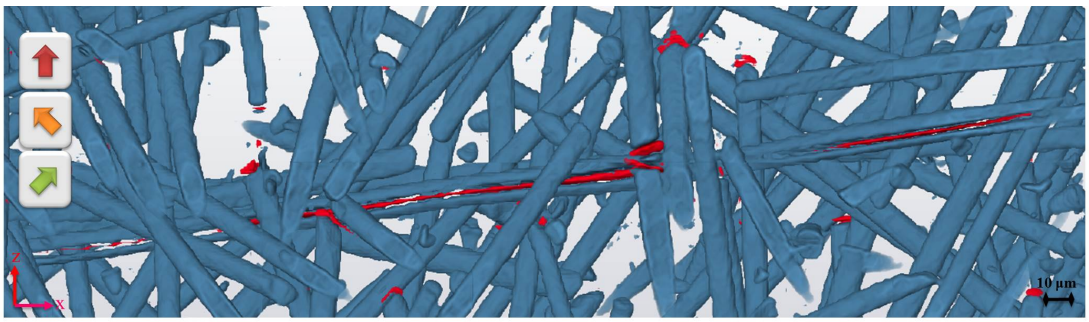
(a) $\varepsilon_1 = 0.0\%, \sigma_1 = 0MPa$



(b) $\varepsilon_2 = 0.9\%, \sigma_2 = 24MPa$



(c) $\varepsilon_3 = 1.2\%, \sigma_3 = 30MPa$



(d) $\varepsilon_4 = 1.4\%, \sigma_4 = 33MPa$

Figure 9. A fibre oriented transversely to tensile direction breaks following its length

4. Conclusion

First objective was to link damage mechanisms with spatial configuration of microstructure. To do so, an adapted tensile machine (size and load ring) has been designed and used to microtomographic in situ testing. 0°, 45° and 90° extracted samples have been extracted and scanned at four different stages of strain during tensile tests. Results of these experiments clearly show damage mechanisms, presented in their spatial configuration. It is then possible to see that damage takes place where there are strain field singularities. These particular points are due to disorientation between neighbouring fibres and tensile stress direction.

Similar analyses are conducted to study fatigue mechanisms with this method (observation of the damage mechanisms during in situ fatigue tests with microtomography). Our intention is to identify local damage kinetics and define a local fatigue criterion in order to be able to predict life of SGFRPA66 components.

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