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Damage mechanisms in short glass fibre reinforced thermoplastic during *in situ* microtomography tensile tests

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ABSTRACT

Micromechanical modelling of short fibre reinforced thermoplastics requires identification of damage mechanisms and their kinetics as a function of their microstructure. A compact tensile machine has been designed to observe damage mechanisms during *in situ* microtomography tensile tests. 3D pictures of the gage length are presented at different levels of damage, from the initial state to the failure of the specimen. Fibre failure, damage at fibre ends, debonding and damage growth in the matrix have been identified as damage mechanisms for these materials. Vicinity between crossing fibres has been pointed out as microstructural configurations driving the damage mechanisms. An analysis of the damage evolution (density, morphology and orientation) allows to establish a macroscopic failure scenario, consistent with microscopic observations.

1. Introduction

With increasing constraints of weight reduction 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, whence emerges the need to describe its behaviour. The complexity mainly comes from the microstructure of the material: the injection process, perfectly suited for high productivity and complex shapes, induces heterogeneous distribution and orientation of fibres [1,2]. Furthermore, the mechanical performance of these composites results from a combination of fibre and matrix properties and the ability to transfer stresses across the fibre–matrix interface as indicated by Thomason's works [3,4]. Micromechanical approach is an efficient way to model short fibre reinforced thermoplastics behaviour. This approach requires damage mechanisms knowledge and strain and stress thresholds values. Current experimental challenges consist in observing and localise damage initiation and its development in the reinforced polymer at the appropriate scales, i.e. micro scale. Despite significant works, there still is a lack of data about the link between microstructure and damage mechanisms. Reference work in the field results from combining scanning electron microscopy (SEM) observations and acoustic emissions analysis: a description of damage chronology has been made by Horst [5] and Sato [6,7] for tensile stresses. They highlight main damage mechanisms, in a localized region subjected to stress concentration: 1) Initiation of interfacial microfailure at the fibre ends. 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. SEM provide high resolution observations but only surface information that may not be representative of mechanisms proportions and kinetics in the bulk of the specimen. In order to reach the exact nature of bulk damage mechanisms (i.e. damage location with respect to 3D microstructure), microtomography is probably the most efficient tool [8,9]. The development of this technology over the past ten years now allows to reach resolutions (in the micron range) and acquisition times suitable for *in situ* tensile tests for 3D damage evaluation in reinforced thermoplastics [10]. In this paper *in situ* synchrotron X-ray microtomography tensile tests were performed in order to evaluate the evolution of damage in the gage length of a tensile specimen. This method was used (as detailed in Section 2) to describe qualitatively (Sections 3.2 and 3.3) and quantitatively (Section 3.4) the damage mechanisms according to the local microstructure configurations. These results allow to establish a damage scenario (exposed in Section 3.5) for short glass fibre reinforced polyamide under monotonic loading.

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2. Material and methods

This work is the result of a combination between mechanical tests management and X-ray microtomographic observations. Such an experimental set up is called *in situ* because the measure of damage is made during the test. Following paragraphs indicate parameters used for this experimental procedure.

2.1. Microtomography

X-ray computed microtomography is an observation technique based on the acquisition of a large number of X-ray radiographs obtained for different angular positions of the sample with respect to the beam. These sets of X-ray radiographs are arranged with a standard filtered back projection reconstruction algorithm to obtain the three dimensional distribution of the linear X-ray attenuation coefficient μ within the sample. This analytical method is faster than algebraic ones, but requires a complete set of radiographs during the rotation and cannot deal with missing views [11]. The elementary unit of the resulting 3D picture, is called a voxel (*volumetric pixel*).

Experiments presented in this work 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. One of the difficulties of this experimental work is to optimize these parameters to manage both quality of the signal and sample stability. Exposure time is there a key element: in one hand, it has to be short enough to limit viscoelastic relaxation of the sample. In other hand, this time has to be sufficient to allow a satisfying signal with a relatively low photon energy (19 keV in this study), to prevent material degradation. After different tested configurations, an exposure time of 0.2 s has been chosen, with a reduced number of radiographs (2000). These radiographs are received by a Fast Readout Low Noise (FReLoN) 14-bit CCD camera with 2048 × 2048 pixels, during rotation of the machine over 180° along vertical axis. This experimental set-up was optimized to obtain a voxel edge size of 0.7 μm. The acquisition of a complete scan lasts about 9 min.

2.2. Compact tensile machine

A displacement controlled and force measuring machine was developed. One of the key factors regarding 3D image quality is the distance between the specimen and the CCD sensor. The tensile machine was designed to minimize that distance (less than 30 mm). The loading ring was made of 2 mm thickness PMMA tube in order to reduce additional attenuation by the experimental set-up. The machine was directly mounted on the rotating stage of the beam line as shown in Fig. 1. A load cell was specifically designed and an optical camera was synchronised in order to capture the specimen deformation at different loading stages. The machine capacity is 2*kN* and the displacement is controlled with 0.35 mm increments.

2.3. Specimen

The studied material is Technyl® A218V30, a commercial grade of polyamide 6,6 reinforced by 30 wt% of short glass fibre supplied by Solvay Engineering Plastics-France. In addition to an intricate behaviour, the matrix of this material shows sensitivity to its conditioning. Indeed, effect of water content on polyamide 6,6 mechanical properties has been demonstrated [12–14]. For this reason, the water content of all specimens was controlled and fixed at 50% of relative humidity (RH50). Considering the short duration of each experiment, the water content was considered as constant between the beginning and the end of the tensile tests.

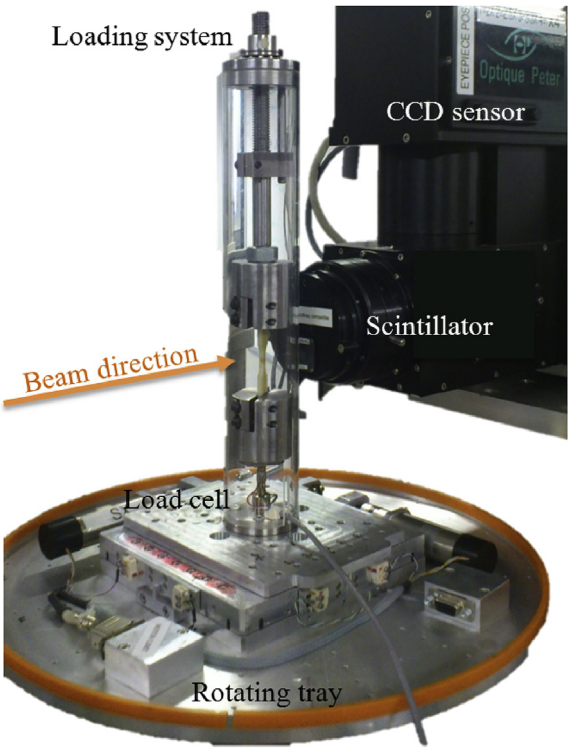

Fig. 1. Compact tensile machine set up for *in situ* testing at ESRF ID19.

The geometry of the specimen was designed to accommodate the constrains of the experimental set-up. Indeed, 3D X-ray microtomography only allows to observe relatively small volumes, depending on resolution and sensor size. The synchrotron experimental set-up described in the previous section allows to obtain a cylindrical observed zone of 1.4 mm diameter and 1.4 mm height (2048 pixels × 0.7 μm resolution). The gage length was set so that the stress state in the observed volume was homogeneous and a square section was chosen to improve microtomography quality (compared to a rectangular section). Taking into account these elements, the geometry of the specimen was chosen as presented in Fig. 2 obtained by water-cut from injected plates. This geometry allows to obtain similar tensile tests results as on full scale normalised tensile specimens (gage length of 3.24 mm × 14.1 mm × 20.0 mm) obtained by the same process.

Fig. 2 also shows the fibres arrangement in the initial obtained volume. This microstructure is composed of more than 25,000 fibres and several thousands of damage markers at each stage of the tensile *in situ* experiment, so that the representativeness of the obtained data is ensured. Fibres have a constant diameter of 10.9 μm. Their length is distributed between 20 and 800 μm, as seen in Fig. 3, with a mean length of 234.3 μm and a standard deviation of 152.1 μm.

The orientation of fibres is heterogeneous in the thickness of the specimen: a core–shell–skin structure is induced by the injection process used to form rectangular plates [15]. This structure, typically observed for injection moulded short fibres reinforced thermoplastics, is characterised by three distinguishable layers: core, shell, skin, as illustrated in Fig. 4. The skin layer is due to the thermal shock between the injected material and the mould walls. Fibres are frozen in their position and orientation forming a 50 μm