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In situ X-ray tomography investigation on damage mechanisms in short glass fibre reinforced thermoplastics: Effects of fibre orientation and relative humidity

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ABSTRACT

Damage mechanisms of reinforced polyamide 6,6 have been studied in 3D through *in situ* X-ray tomography tensile tests. 3D pictures of the microstructure have been taken during tensile tests to catch damage evolution in the bulk of material. The effects of relative humidity and orientation sampling are particularly investigated in this paper. Main mechanisms have been identified such as fibre failure, debonding, damage at fibre ends and matrix damage (cavitation, fibrillation, damage growth). Qualitative observations reveal that the mechanisms are very sensitive to orientation sampling and relative humidity of the specimen. A specific procedure was developed to propose a quantitative analysis of the results. This analysis shows that identified mechanisms not only have different proportions but also have different kinetics according to relative humidity and orientation sampling of the specimen.

1. Introduction

Automotive constructors having to reduce the global weight of vehicles, the need of low cost materials with a good ratio density-performances has been growing over the past ten years. Short glass fibre reinforced (SGFR) thermoplastics are good candidates but their behaviour still has to be further understood and modelled in order to be efficiently and widely used. In addition of providing good mechanical properties, SGFR thermoplastics are adapted to moulding injection process, which allows short process cycles, high dimensional precision and complex shapes, well-suited for industrial applications. However, this process induces strong anisotropy through fibres orientation: during injection, the matrix flow field drives fibres orientation.

To evaluate the anisotropy of the material, specimens are usually extracted from injection moulded sheets with different orientations compared to the main injection direction. The effect of the specimen orientation on the macroscale mechanical properties has already been widely studied in literature. For instance, it has been shown that ultimate tensile strength [1] or elastic modulus [2,3] depends on fibre orientation. Mechanical properties anisotropy is strongly linked to different damage kinetics and mechanisms depending on the specimen orientation with respect to the applied mechanical loading [4].

For reinforced polymers, damage is the main source of mechanical properties degradation and has to be precisely described and understood in order to model accurately their behaviour. The works of Sato [5,6] and Horst [7,8] opened the way of local analysis of damage initiation during tensile and fatigue testing. Using SEM fractography and acoustic emission, they observed different damage mechanisms and have elaborated qualitative scenarii for their evolution. From their results, the following main steps for tensile damage were identified: 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. However this description results from surface observations and since this material has a complex 3D microstructure, it has to be completed by volumetric observations. This approach began to be explored thanks to the improvement of observation means such as X-ray tomography. For instance, recent works of Arif et al. [9] combined X-ray tomography and *in situ* SEM bending, in order to

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understand the link between damage mechanisms at the micron scale and the degradation of macroscopic properties. With the same observation strategy, Arif et al. [10] also studied the influence of relative humidity (RH) on the damage mechanisms. This qualitative analysis demonstrated that damage mechanisms at the fibre-matrix interface are more developed when the relative humidity is higher. If it has been noted that the macroscopic properties of the polyamide 6,6 are very sensitive to conditioning [11,12], only a few study evaluate the effects of relative humidity (RH) on the damage mechanisms at the local scale and none really evaluate these effects on their proportions and on their kinetics at the local scale.

It is well admitted that the detrimental effect of water absorption on the mechanical properties of the polyamide is due to polar amide groups. In dry as moulded state, these amide groups constitute strong interactions between crystalline and amorphous phases, by the generation of hydrogen bonds. However, when the polyamide is conditioned at higher RH, amide groups of the amorphous phase interact with water molecules. Consequently, hydrogen bonds are weakened, which increases chain mobility in the amorphous phase: this is the plasticization effect. Properties of the polymer are affected by this plasticization: the glass transition temperature is reduced, as the strength and the modulus, whereas ductility is increased.

In this work, *in situ* microtomography tensile tests were performed to identify the damage mechanisms of short glass fibre reinforced polyamide 6,6. Specimens have been conditioned at three different relative humidity conditions: RH = 0%, 50% or 80% and extracted following three orientations compared to the main flow direction: 0°, 45° or 90°. The first part of this work was to determine the best strategy to adopt for the use of X-ray microtomography to evaluate damage mechanisms in such materials. The results of this preliminary work [4] show that this approach allows to localise damage markers, to obtain their morphology and to differentiate the observed mechanisms. A statistical study led to the evaluation of individual damage mechanisms kinetics under uniaxial tensile tests. In the present work, a similar approach has been applied to specimens with different relative humidity states to represent conditions where the material is in glassy, glass transition and rubbery states. Indeed, modifying the relative humidity shifts the glass transition temperature so that different material state can be reach while testing specimens at ambient temperature. The damage mechanisms are identified and compared in each case. The conclusions provide damage scenarii that consider the effect of the sampling orientation and the relative humidity content for the PA66GF30 composite material, providing quantitative experimental data for micro-mechanical models identification.

2. Experimental procedure

2.1. X-ray microtomography

X-ray microtomography is a non-destructive and high resolution tool based on local attenuation evaluation, which allows to take 3D pictures in the core of materials [13,14]. Its use is an opportunity to understand damage mechanisms in a large variety of materials. This method has recently been applied for SGFR thermoplastics [4,9,15,16] with different uses and interests.

Experiments presented in this work were performed on ID19 beamline at the European Synchrotron Radiation Facility (Grenoble, France). The set-up parameters for these tests were the same as in Ref. [4]: a monochromatic X-ray beam with 194.77 *mA* intensity and 19 *keV* photon energy was used. 2000 radiographs with an exposure time of 0.2 *s* have been 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 a 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 on Fig. 1. A load cell was specifically designed and an optical camera was synchronised to the data acquisition system in order to capture the specimen deformation at different loading stages. The machine maximum load capacity is 2 *kN* and the displacement is controlled with 0.35 *mm* increments.

2.3. Specimen

The studied material is a Technyl®A218V30, a commercial grade of polyamide 6,6 reinforced by 30 wt% of short glass fibre, supplied by Solvay Engineering Plastics-France. Specimens are sampled from rectangular plates with a 3.24 *mm* thickness, obtained by injection moulding. The injection moulding process leads to an heterogeneous orientation of fibres in the thickness of the plates. This structure is usually described as a superposition of distinguishable layers: the skin, the shell and the core, as illustrated in Fig. 2.

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 100 μm thick layer of randomly oriented fibres. The shell layer is the largest with a thickness of 1.4 *mm*. In this layer, the shear stress induced by the material flow orientates fibres in the mould flow direction (MFD). The core layer is 300 μm thick in the centre of the specimen. Contrary to the shell, fibres in the core are perpendicular to the MFD. This orientation is due to an

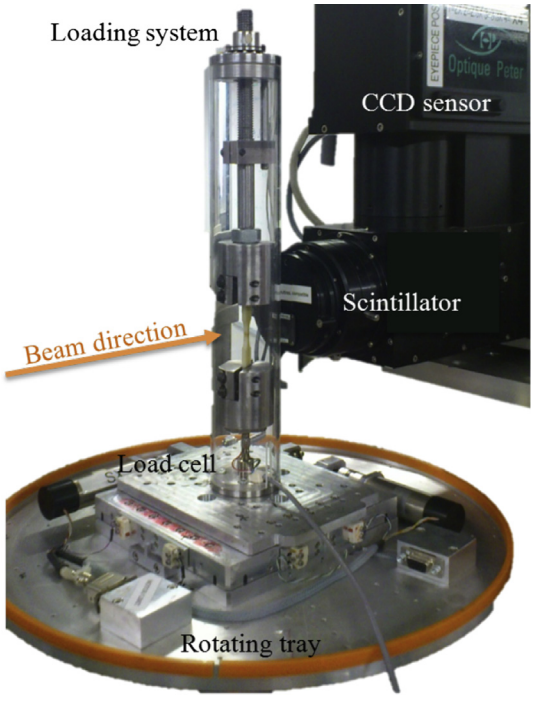

Fig. 1. Compact tensile machine set up [4] for *in situ* testing at ESRF ID19.