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FATIGUE DAMAGE MECHANISMS IN SHORT FIBRE REINFORCED THERMOPLASTICS OBSERVED BY X-RAY MICROTOMOGRAPHY

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Summary: Short glass fibre reinforced thermoplastics have been observed by X-ray microtomography during mechanical fatigue testing. 3D pictures of the gage length of the specimens have been obtained for different levels of damage. Numerous damage markers (fibre failure, matrix damage, interface debonding) have been observed, described according to the microstructure and quantified to obtain the kinetic for each mechanism during the material life.

1. Introduction

Short glass fibre reinforced thermoplastics are promising materials to be used in the automotive field. Their low density against very good mechanical properties can contribute to the vehicles mass reduction, key element to reach objectives of gas emission decrease. However, the associated process, injection moulding, induces a complex microstructure and the link between this microstructure and fatigue mechanisms has not been clearly shown yet. The current challenge is to explain these mechanisms regarding spatial configuration of the microstructure, in order to be able to predict the material final degradation and optimize structures. First observations by SEM fractography of polyamide 6,6 reinforced by 30wt% of short glass fibre reveal several damage mechanisms such as matrix cavitation, fibre-matrix debonding and fibre failure. X-ray microtomography is then used for volumetric observations of a microstructure at different stages of damage evolution, since this 3D high resolution tool is non-destructive. This in situ testing allows to elucidate chronology and extent of each mechanism according to the local microstructure

2. Experimental method

A machine was developed to combine fatigue testing and high resolution X-ray microtomography measurements. The machine has been designed to be compact, in order to minimize the distance between the specimen and the sensor. To maximize the X-ray beam signal, the machine had to present an homogeneous path around the specimen with a minimum attenuation of the X-ray beam. This function is ensured by a 2 mm thickness PMMA tube at the level of the specimen gage length. The machine was directly mounted on the rotating stage of the beam line. A load sensor measures the load applied to the specimen. All tests were performed under load control, with a maximal capacity of 1.5kN.

These experimental sets-up has been used on Psyché beamline at the SOLEIL synchrotron (Saclay, France). The set-up parameters used for the experiments are described below. The filtered X-ray beam (2 mrad mirror, 0.5 mm aluminium and 0.25 mm silver) had an energy of about 26 keV. The CMOS detector with 6.5 micron pixels, was associated to a x10 objective, leading to a voxel edge size of 0.65 µm, on the acquired image. Specimen-sensor distance was 35 mm. 1500 projections were recorded per scan during a 180 degrees rotation of around 5 min, with 31 references pictures before and after scanning.

The studied material is a Technyl®A218V30, a commercial grade of polyamide 6,6 reinforced by 30wt% of short glass fibre, supplied by Solvay Engineering Plastics-France. Specimens are sampled from rectangular plates with a 3.24mm thickness, obtained by injection moulding.

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3. Results

Six damage mechanisms have been observed in the bulk of the specimen: fibre failure, damage at fibre ends, debonding, cavitation, crazing and microcrack. A description of these mechanisms is proposed, including their initiation, their growth and their interactions.

The **cavitation** mechanism concerns small (inferior to 5µm diameter) spherical damage markers. These damage markers seem to appear soon in the fatigue life, nevertheless, this mechanism was not observed in such proportions during monotonic tensile loading at the same load level. When detected, they appear in the shape of spheres of 1 to 5 µm diameter. Their size is quite stable from one scan to another, meaning that they do not grow significantly during the fatigue lifetime. However, it should be noticed that their number significantly increase with fatigue lifetime. From our observations, cavitation occurs either in fibre free zones or very close to the fibre-matrix interface.

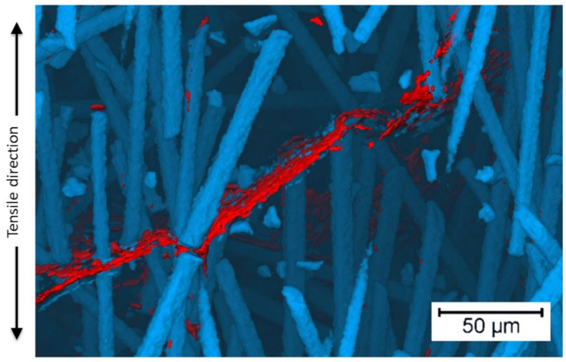
The apparition of **damage at fibre ends** (also called fibre tips) has frequently been observed. This damage mechanism has also been observed for monotonic tensile testing and is explained by the lack of bounding between fibre ends and matrix (due to the process) and the geometry of fibres, turning ends of fibre to stress concentrators. If these damage markers initiate at the first cycle in the same way as in quasi-static solicitation, they evolve very differently during the fatigue lifetime. Indeed, instead of growing in the matrix as for tensile loading by ductile damage growth, each damaged zone tends to initiate a microcrack in the matrix, transversally to the macroscopic tensile direction.

F**ibre failures** have been observed. Even though fibres are relatively short, they can break in two or more parts, in a transverse direction to the macroscopic loading. A crossing with close neighbouring fibres has systematically been observed around broken fibres. This mechanism happens during all the fatigue lifetime of the specimen the cyclic solicitation, with high strain lead to local strain redistributions. A fibre failure can early happen due to the local stress concentration (fibres crossing). The stress release in the matrix leads to a very high local strain and successively, the failure of neighbouring fibres. It is worth noting that when a fibre breaks, contrary to observations made for monotonic testing, the damage does not grow spherically but grows in the form of a microcrack transversal to the macroscopic loading.

Debonding concerns damage at the fibre-matrix interface. It mainly happens along fibres oriented transversally to the macroscopic load. This mechanism can be initiated by damage at fibre ends but, is mostly due to shredding of the interfacial matrix, or by a confinement effect between neighbouring fibres. It appears at an advanced stage of the fatigue lifetime and can contribute to crack propagation.

Fibrillated crazes are damaged zones of the matrix, where there are still fibrils between the two parts of the damage marker. They are initiated by high deformation of the matrix and observed close to the fibre-matrix interface or between damage zones of the matrix. This phenomenon appears at the end of the fatigue life, most of the time where cavitation was developed.

Microcracks larger than a few hundred microns result from growth or coalescence of damage markers in highly damaged zones. They have only been observed in advanced steps of the fatigue lifetime (cf figure 1). These microcracks appear in zones with high density of damage markers. The propagation rate and the crack path are highly dependent on the local microstructure.



Micro-crack going through a fibre – Fatigue: N/N_R= 95%

Figure 1: X-ray microtomographic observation of a microcrack due to fatigue

This study presents the first results by *in situ* microtomographic fatigue tests on reinforced thermoplastics. The high resolution allows to consider many aspects of the microstructure and to link it with the damage mechanisms.