Application of synchrotron radiation micro-CT to local morphological and numerical characterization of short fibre reinforced polymer composites

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1 Introduction

The behavior of materials with a complex structure strongly depends on the spatial arrangement of their components. In mechanical parts of short glass fibre reinforced polymer (SFRP) obtained by injection moulding, processing conditions produce complex orientation patterns that influence the mechanical properties of the component. For example, different fatigue (i.e. failure caused by cyclically repeated loads) behaviors can be observed when short fibre reinforced polyamide notched specimens of same geometry are injection moulded through gates whose position is varied [1]. This effect appears to be due to the different fibre orientation patterns obtained [2] and it can be interpreted in terms of different local material properties related to fibre orientation and distribution. Traditional, destructive, approaches to fibre pattern identification are affected by severe limitations. Recently, a novel technique based on microtomography 3D reconstruction of the fibre structure has been introduced for quantification of fibre pattern [3].

2 Fibre visualization

Computed microtomography with synchrotron radiation constitutes the ideal non destructive technique for fibre visualization, being able to provide high resolution images of the internal structure of a material. In particular, the source high spatial coherence available at the synchrotron facility of Elettra (Trieste) makes is possible to apply phase contrast imaging techniques that exploit also information of the phase shifts induced by the sample inhomogeneities [4]. Image contrast is then originated from interferences among parts of the wave-fronts that have experienced different phase shifts. These techniques are particularly

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effective when the samples exhibit poor intrinsic contrast due to low atomic number (i.e. the case of "soft matter") or, more in general, to low variation of absorption from point to point. Thus, phase contrast imaging techniques allow to detect even small details, such as short reinforce fibre within a polymeric matrix. Among the different approaches to phase-sensitive radiology reported in literature, PHase Contrast (PHC) radiography has the easiest implementation: the setup is the same as in conventional radiography but the detector is placed at a certain distance d from the sample. At the SYRMEP beamline, the white beam produced in one of the Elettra bending magnets, collimated by a slit system and gone through a double-crystal Si(111) monochromator, impinges the sample placed at a distance of about 23m from the source. The sample-to-detector distance d can vary from 0m to about 2m. Images taken with $d = 0$ reproduce the conventional absorption radiographs. If $d > 0$ (PHC), the X-rays free space propagation transforms phase modulation of transmitted beam into amplitude modulation when they reach the detector. According to the choice of d with respect to the size a of the feature to be identified perpendicularly, images can be directly used to extract morphological information when $d << a^2/\lambda$ (edge detection regime), where λ is the X-ray wavelength. The choice of d depends also on the detector characteristics, as it must be large enough to allow the small angular opening of the produced interference pattern to be converted into a length compatible with the detector spatial resolution. The produced diffraction pattern appears superimposed on the detector to the conventional absorption radiograph and contributes to enhance the visibility of the edges of the sample features [4, 5], such as short fibre in matrix.

3 Morphological characterization

Morphological characterization was carried out on samples extracted from specimens used in fatigue tests, at the critical locations (i.e. where fatigue cracks nucleated and propagated). Given fibre numerosity in our samples, instead of trying to isolate each single fibre and measure its geometrical properties from the 3D micro-CT reconstructions, we introduced a novel technique, quantifying fibre orientation principal directions by means of global morphological parameters including Mean Intercept Length (MIL). MIL is computed by sending a line through a 2-phase volume and by dividing its length by the number of times it crosses one of the phases. By changing the orientation of the test line, a 3D representation can be obtained. While the MIL locus for fibre networks with a discrete number of distinct fibre is a polygon [6] and therefore a fabric tensor cannot be defined [7], it was shown in [3] that the 3D MIL locus of short fibre reinforced polyamide can be approximated by an ellipsoid, so that a second order fabric tensors based on Mean Intercept Length can be used to identify local fibre orientation angles and anisotropy differences in SFRP samples. In particular, Mean Intercept Length (MIL) and the related fabric tensor were used to assess local fibre orientation variations and to interpreter fatigue tests results in the light of different fibre orientation distributions [2].

4 Numerical analysis

In order to investigate the local elastic properties of short fibre composites, strictly related to fibre orientation distribution, the apparent elastic moduli values in different sites within a SFRP sample were computed by means of a numerical model applied to the same 3D reconstructions used for morphological analysis. The micro-mechanical model implements the Cell Method [8], a numerical method based on a direct discrete formulation of physical laws that becomes particularly advantageous when discontinuities are present, since discretization and heterogeneities may have similar characteristic lengths [9, 10]. The numerical application used to assess the local elastic properties of the SFRP sample had been previously developed for the analysis of trabecular structures and was described in detail in [11] and [12]. Both morphological and numerical methods were able to capture the principal directions of anisotropy and the differences among volumes of interest. Even if the fabric tensor, normalized, eigenvalues did not show a significant correlation to the apparent elastic moduli, approximately 92% of stiffness components variance could be accounted for by changes in MIL eigenvalues once normalized values were used for elastic moduli [13]. The same numerical Cell method model was also applied to study the differences observed in fatigue behavior of SFRP notched specimens depending on injection point location. The experimental results might be attributed to different strain distributions due to variations of the local apparent elastic properties along the specimen section, and attributed to changes in fibre orientation occurring during the manufacturing process [14]. Further investigation is currently being carried on in order to quantify these effects.

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