# FIBRE ORIENTATION AT NOTCHES AND FATIGUE BEHAVIOUR OF SHORT FIBRE REINFORCED POLYAMIDE

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## **SUMMARY**

Notched specimens of short fibre reinforced polyamide were injection moulded through different gates, in order to achieve different fibre orientation patterns for the same geometry. This resulted into different fatigue behaviour. Fibre orientation pattern at notches was analyzed using a novel technique based on micro tomography.

Keywords: Fatigue, Short fibre reinforced polymers, fibre orientation, micro tomography, notches

## INTRODUCTION

Fatigue strength of injection moulded short fibre reinforced polymer components depends upon fibre orientation with reference to the acting stresses. For the case of a short glass fibre reinforced (SGFR) polyamide 6, this dependence was expressed through a relationship derived from the Tsai-Hill criterion [1], with the strong assumption that fibres were parallel to each other. This relationship was verified for off-axis uniaxial tests on specimens extracted from injection moulded plates and assuming that mean fibre orientation coincided with the plate's axis. Real parts display a more complex geometry, and their fatigue strength is influenced by other factors like weld lines [2] and notches [3,4]. No analysis of the local fibre orientation distribution was reported in these previously published papers.

In this work, the results of an experimental investigation on the combined effect of both notches and fibre orientation upon fatigue strength of injection moulded flat specimens are analyzed. Different fibre orientation patterns in notched specimens having the same geometry were obtained by changing size and position of the injection gate. A possible relationship between fatigue strength and fibre orientation is discussed on the basis of the analysis of the 3D fibre orientation distribution in proximity of notches, which was reconstructed by micro computed tomography provided by a method recently proposed by the authors of the present paper [5].

## **EXPERIMENTAL**

The material investigated is a short glass fibre reinforced polyamide-6 reinforced with 30% by weight of E-glass fibre (PA6 GF 30). Fibre nominal diameter was 10  $\mu$ m and average fibre length was 275  $\mu$ m. The specimens consisted of small plates, 3.2 mm thick, and were characterized by lateral circular blunt notches (see Figure 1). The mould was designed for injection moulding of these specimens through different injection gate locations. Type and position of the injection gates are shown in Figure 1 with blue arrows (longitudinal injection through an edge gate) and a red arrow (lateral injection). The material was conditioned prior to testing until it reached hygro-thermal equilibrium with an ambient at 23°C and 50% relative humidity.

## **Fatigue tests**

Uniaxial fatigue tests were conducted using an MTS 810 servo-hydraulic test system, with a capacity of 100 kN, equipped with a load cell of 10 kN. All tests were conducted at a room temperature of  $23^{\circ}\text{C}$ . Tension-tension (R = (min. load)/(max. load) = 0.1) fatigue tests were run in load control mode in the range of cycles to failure from  $10^{3}$  to  $10^{6}$  at a cyclic frequency of 2 Hz. Tests were interrupted at specimen separation or when the number of cycles reached  $10^{6}$  (run-outs). The nominal stress was evaluated by dividing the applied load by the reference section area (3.2 x 30 mm²). Fatigue tests were also available for standard ISO 527-2 Type 1A specimens [6] under the same test conditions and are used for evaluation of fatigue notch factors.

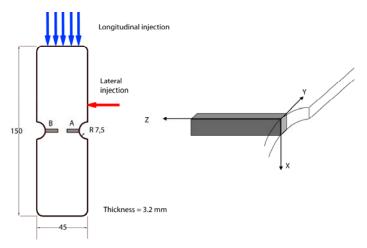


Figure 1: Specimen type and dimensions, with injection gates; shape and position of samples examined with micro-CT

The fatigue results of the specimens having lateral notches are reported in Figure 2 as stress-life curves (also known as S–N curves or Woehler curves). It appears that fatigue strength depended on injection gate position. The Basquin equation was used to describe the relationship between maximum stress and cycles to failure:

$$\sigma_{\text{max}} = \sigma_f N^b \tag{1}$$

The values of fatigue strength exponent b and fatigue strength coefficients  $\sigma_f$  for different specimens are listed in Table 1. The values of the fatigue notch factors  $k_f$ , also

reported in Table 1, were evaluated as ratio of fatigue strength  $\sigma_W$  of the plain, unnotched, specimen over that of the notched specimens, both evaluated at  $10^6$  cycles, i.e.

$$k_f = \frac{\sigma_W (N = 10^6)_{\text{plain}}}{\sigma_W (N = 10^6)_{\text{notched}}}$$
(2)

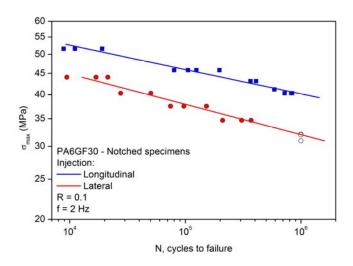


Figure 2: S-N curves for specimens with lateral notches

Table 4: Values of the fatigue strength coefficient  $\sigma_f$  and the fatigue strength exponent b of PA6 GF 30 specimens with different injection gate positions

| Injection    | $\sigma_{\!f}$ | b      | $\sigma_W (10^6 \text{ cycles})$ | $k_f$ |
|--------------|----------------|--------|----------------------------------|-------|
| ISO 527      | 101.9 MPa      | -0.048 | 52.5 MPa                         | -     |
| Longitudinal | 96.6 MPa       | -0.064 | 39.9 MPa                         | 1.32  |
| Lateral      | 88.1 MPa       | -0.073 | 32 MPa                           | 1.64  |

## Fibre orientation analysis

In order to characterize the specimens used in the tests, a detailed description of microstructure at these critical locations was performed by computed microtomography (micro-CT) with synchrotron radiation. In a previous paper [5], it was shown that the Mean Intercept Length (MIL) parameter and the related fabric tensor, computed from 3D micro-CT reconstructions, could be used to measure mean fibre orientations in samples of injection moulded polyamide. In order to investigate whether the observed variations in fatigue behaviour were related to differences in fibre orientation at notches, because of different injection point locations, samples extracted close to the specimens' notches were analyzed by means of these morphological parameters [7]. In this paper we present the analysis over a larger (11 mm from each notch root, instead of 3 mm as in Ref. [7], along z axis of Figure 1) portion of the gauge section.

Computed micro-tomographies were obtained at the SYRMEP beamline of Elettra synchrotron light source (Trieste, Italy). This beamline is equipped for high-resolution micro-CT, with a maximum achievable resolution of approximately 9 micrometers. In case of synchrotron radiation, high spatial coherence of source makes is possible to apply imaging techniques called Phase Contrast (PHC) that exploit information on phase shifts induced by the sample on radiation field. Analyses conducted at the SYRMEP beamline showed that with this PHC set-up it is possible to obtain a 3D digital reconstruction of fibre orientation pattern even in case of fibre characterized by a small diameter (11  $\mu$ m average diameter vs 9  $\mu$ m sensor resolution).

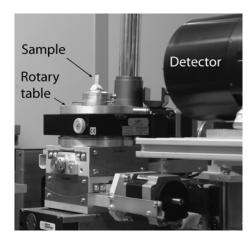


Figure 3: micro-CT experimental setup

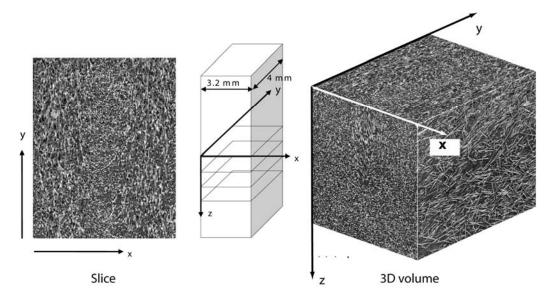


Figure 4: sample cross sections (slices) elaborated from the radiographic projections and volume reconstruction obtained by stacking the slices

The sample, visible in Figure 3, is placed on a rotary table. By rotating the sample in front of the detector from 0 to 180 degrees, a total number of 720 planar projections are acquired. From these projections, cross-sectional images (tomographies) are obtained by

a filtered back-projection algorithm. By stacking the planar tomographies, 3D reconstructions showing the internal micro-architectures were obtained, as shown in Figure 4.

Direct analysis of each single fibre appearing in the 3D reconstructions was hardly suitable because of small dimensions and great number of fibre. A global approach, based on the Mean Intercept Length (MIL) concept, was therefore preferred, as described in [5]. The Mean Intercept Length (MIL) is a parameter commonly used in biomechanics, which is defined as the average distance between the two phases of a structure, computed by sending a planar grid of length L through a 3D image volume and by dividing L by the number of times the grid intercepts fibre-to-matrix interfaces (matrix-to-fibre interfaces are neglected). Therefore, higher values of MIL are to be found in the preferred direction of fibre orientation.

When this measure is repeated over a large number of 3D angles, MIL values can be plotted on a polar graph as a function of orientation angles. This is the MIL value locus which, for two-phase materials, can be approximated by an ellipsoid or, equivalently, by the second order positive defined MIL fabric tensor. The MIL fabric tensor eigenvalues (T1, T2 and T3, in descending order) are assumed to correspond to the principal directions of fibre orientation and its eigenvectors constitute a measure of fibre orientation pattern shape in the principal directions. Anisotropy of the sample can be thus globally evaluated, avoiding analysis of each fibre, for example by means of the Index of Anisotropy, defined as  $IA = 1 - T_3/T_1$ , which gives IA =0 for total isotropy and IA = 1 for total anisotropy.

#### **RESULTS AND DISCUSSION**

In specimens having the same 3.2 mm thickness and injected together with the specimens used in this work using the same mould [1], a layered structure was found, formed by a shell layer, where fibres tend to align to melt polymer flow because of shear flow induced by high viscosity, and a core layer, where, mainly due to extensional flow, fibres tend to align perpendicularly to melt polymer flow. A similar structure was expected in the notched specimens and therefore volumes were extracted from both layers and analysed.

The procedure we set for the analysis of samples is described schematically in Figure 5. Each reconstructed sample was divided into cubic volumes of 80 x 80 x 80 voxel<sup>3</sup>, that constituted the Volumes of Interest (VOIs) for the morphological analysis. MIL parameters were evaluated using Quant3D software [8]. In Figure 5, a tomographic image of the core layer at point A of the specimen injected longitudinally is shown. In order to make fibres visible, the images cover only 320 pixel in the z direction, which correspond to approximately 3 mm, but analysis was performed covering approximately 11 mm along the z direction (specimen gauge section is 30 mm wide, thus microCT analysis cover 22 mm over 30 mm, excluding the central part of the specimen). A representation of fibre spatial distribution is given by the MIL diagrams, which are reported below the reconstructed image in correspondence with the centre of each cubic volume. Both x-z and y-z views are displayed. Form the diagrams, mean fibre orientation can be inferred, assuming that it coincides with the first principal direction of the MIL fabric tensor (first eigenvector). From eigenvectors, mean fibre

orientation angles can be evaluated. This procedure was repeated for both samples, indicated as A and B in Figure 1, for two specimens: one injected longitudinally and one laterally

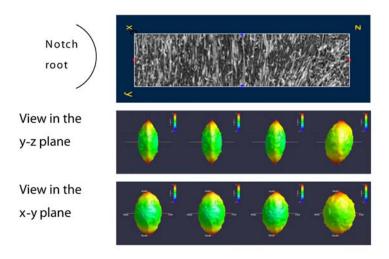


Figure 5: Longitudinal injection, point A in Fig. 1, core layer: micro-CT reconstruction and MIL graphic representation

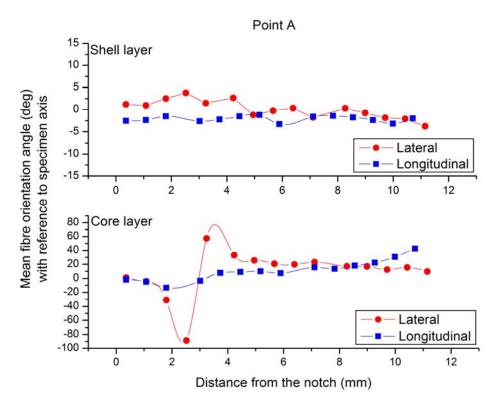


Figure 6: comparison of values of mean fibre orientation angle with respect to the specimen's longitudinal axis for specimens injected longitudinally and laterally; point A of Figure 1

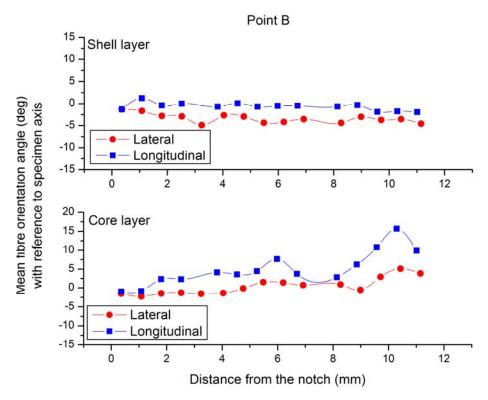


Figure 7: comparison of values of mean fibre orientation angle with respect to the specimen's longitudinal axis for specimens injected longitudinally and laterally; point B of Figure 1

Values of the angle between the y axis and the projection of the first eigenvalue  $T_1$  on the y-z plane, i.e. values of the mean fibre orientation angle with respect to the specimen's longitudinal axis in the y-z plane, are reported for both the core and shell layer as a function of z. Comparison of these values at the same location in specimens injected laterally and longitudinally are reported in Figure 6 and Figure 7, for location A and location B, respectively.

These observations evidenced that in the shell layer of both point A and B, mean fibre orientation direction is mainly aligned with the specimen axis up to a distance of 11 mm form each notch root, irrespective of the injection mode (lateral or longitudinal). In case of lateral injection, deviations from this preferred direction appear only in the core layer and only at distance comprised between 0 and 4 mm from the notch root.

Mean fibre orientation alone is not sufficient to characterize the local properties of the material. Mechanical properties (i.e. stiffness and strength) also depend on the dispersion of the fibre orientations around the mean value, i.e. the index of anisotropy, IA. The variation of IA with distance from the notch root is reported in the graphs of Figure 8 and 9 for location A and location B, respectively. It appears that values of IA in the shell layer are uniform over most part of the samples; moreover, fibre orientation distribution tends to be planar in this layer. Conversely, in the core layer there is a larger scatter of AI values, with values close to 0 at some locations. This indicates that fibres are dispersed almost randomly, with several fibres having mean orientation along the x axis, i.e. perpendicular to the mid plane of the specimens.

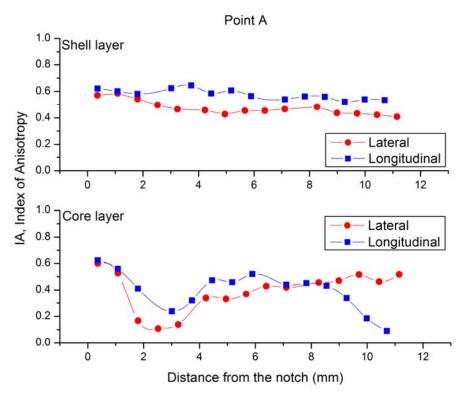


Figure 8 - comparison of values of the index of anisotropy for specimens injected longitudinally and laterally; point A of Figure

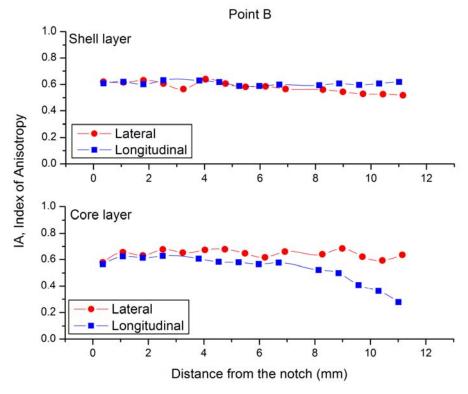


Figure 9 - comparison of values of the index of anisotropy for specimens injected longitudinally and laterally; point B of Figure

This effect is evident from comparison of the shape of the MIL tensor ellipsoids of volumes extracted from the shell and the core layer, respectively, for the same location z = 2.5 mm at point A of the specimen injected laterally. From the shape of the ellipsoid of the shell layer, it may be inferred that fibres are aligned with the y axis, with low scatter, whereas fibres in the core layer are dispersed almost randomly, as indicated by the almost spherical shape of the ellipsoid.

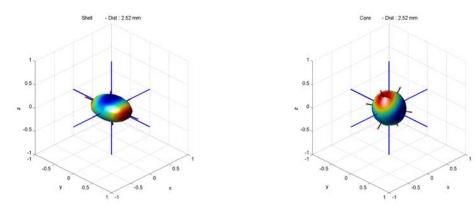


Figure 10 - MIL tensor ellipsoids of volumes extracted at z = 2.5 mm, point A, from the shell (left) and the core (right) layer, respectively (lateral injection)

It appears that a model based only on mean fibre orientation is not sufficient to explain the observed differences in fatigue behaviour. At the notch roots, fibres appear to be aligned with the specimen axis, irrespective of the type of injection, probably because of the aligning effect by shear flow induced by the lateral walls of the mould. Thus, to relate fatigue to fibre orientation, straightforward use of the modified Tsai-Hill relationship derived in Ref. [1] is not possible. Instead, the observed differences could be explained in terms of different stress concentration at notched as a result of different global stress distribution, which in turn reflects differences in the fibre distribution patter in the whole specimen. Moreover it appeared that other parameters than mean fibre orientation, like the index of anisotropy, should be taken into account to fully characterized the material. Thus, in order to predict fatigue behaviour of the specimens it seems necessary to model the entire specimen, e.g. by finite element modelling, taking into account the effect of fibre orientation distribution on local stiffness and strength of the material. Commercial software exist, allowing for simulating injection moulding ad deriving local properties of the elements of a finite element model on the basis of the predicted fibre orientation. Although a relationship between the MIL fabric tensor and the fibre orientation tensor used in these software packages has not been established, the reconstruction of the microstructure by micro CT and the analysis method based on the MIL concept could be used to verify predicted fibre orientation distributions.

## **CONCLUDING REMARKS**

Fatigue of SGFR polyamide notched specimens and its dependence upon type and position of injection gate was investigated by analyzing local fibre microstructure with micro-CT and applying the MIL concept. The following conclusions can be drawn:

- 1. with the exception of the core layer, fibre orientation in proximity of notches is not significantly influenced by changing injection gate position, thus preventing from easy interpretation of results of fatigue tests in terms of local fibre orientation at notches; instead the full stress pattern, and consequently the full fibre orientation distribution need to be taken into account;
- 2. in order to model the stress field by finite elements, it is necessary to include information about fibre orientation and to derive the relationships between local fibre distribution and local elastic constants, e.g. by an intermediate analysis step simulating injection moulding. In this context, experimental measure of fibre orientation by MIL applied to micro-CT could serve as benchmark tool to experimentally validate accuracy of numerical predictions of fibre orientation angles by software simulation.

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