FIBRE ORIENTATION DISTRIBUTION IN SHORT FIBRE REINFORCED POLYMERS: A COMPARISON BETWEEN OPTICAL AND TOMOGRAPHIC METHODS

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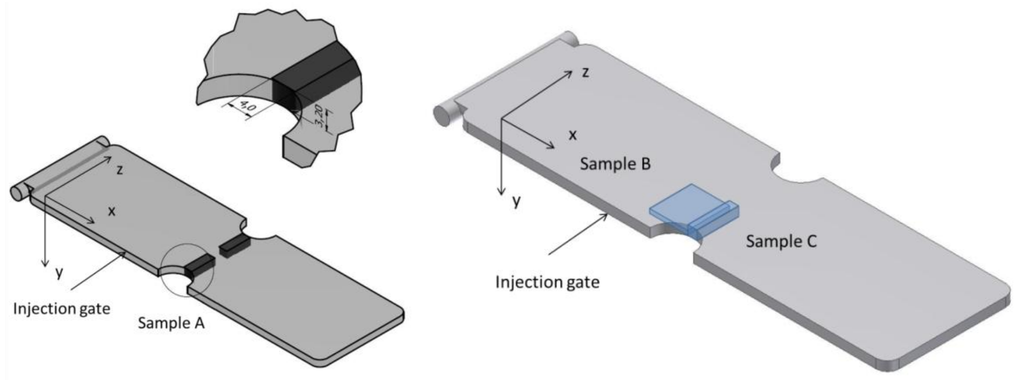
Abstract
The fibre orientation distribution in a material sample of short fibre reinforced polyamide extracted from an injection moulded notched plate was analysed using two different methods, one based on micro-computed tomography and the Mean Intercept Length concept and the other based on the classical optical section method. By the optical method, a good agreement with tomography was obtained with a longitudinal section. A poorer agreement was found with a transverse section. The two methods are in agreement provided that the most suitable section is chosen when applying the optical method, which is where the fibres lie predominantly in the section plane. Moreover, results confirm that the analysis based on the MIL concept is capable of capturing important information about fibre orientation.

1 Introduction
Knowledge of fibre orientation in injection moulded parts made of short fibre reinforced polymers (SFRP) is of great importance because fibre orientation has numerous implications on the functional (e.g. warpage after moulding) and mechanical (stiffness and strength) performances of the parts. The experimental evaluation of the fibre orientation distribution in composites can be performed by different methods [1].
For the analysis of SFRP, the most commonly employed method is based on the optical observation of the elliptical footprints left by the fibres on polished sections of a sample, from which the fibre orientation angles can be inferred [2]. When applying this method, the dependence on fibre orientation of the probability for a plane to cross a fibre has to be taken into account [3, 4]. In some cases issues arise about the accuracy of angle measurements for fibres almost perpendicular to the section plane [5], and special techniques are required to overcome the ambiguity about the sign of the orientation angle [6-10]. However, its use is widespread, thanks to the relative simplicity of the setup required, and the possibility of being automated [11].
Radiography is another possible approach to the analysis of fibre orientation [12]. It is based on the projection of X-rays, thus, it can provide only information on the orientation of the projection of fibres onto this plane. These limitations of radiography can be overcome by micro Computed Tomography (micro-CT), which is based on a series of radiographic

projections taken at different angles. Recently, a method for the analysis of the fibre distribution in samples reconstructed by micro-CT has been proposed, based on the Mean Intercept Length (MIL) concept [13, 14]. By this method it is possible to characterize the internal fibre structure by means of the components of a second order tensor, the MIL fabric tensor, and derive useful information about the preferred fibre orientation and the degree of anisotropy. This method can be applied to subsets of the reconstructed volume, called volumes of interest (VOI).
In this paper we present the comparison between the results obtained by the optical sectioning and the micro tomography method coupled with MIL analysis, applied to the same sample of short fibre reinforced polyamide extracted from an injection moulded, notched specimen, of the same type as those used to study the fatigue notch effect in a short fibre reinforced polyamide [15].

2 Experimental

The material of the samples was a short fibre reinforced polyamide 6, containing 30% by weight of E glass fibres. The material samples used for fibre orientation analysis were extracted from injection moulded specimens, having the shape of a small plate with two symmetric, round notches of 7.5 mm radius (see Figure 1). This specimen can be injected in two different ways: through an edge gate, thus obtaining a longitudinal flow which determines a regular fibre orientation pattern, and through a point gate on one side, which causes a more irregular fibre orientation pattern, as described in [15, 16]. The specimen type studied in this work was that injected laterally, because in this configuration variations of fibre orientation across the width of the specimen's gauge are large, particularly in the core layer (the specimen displayed the common layered shell-core-shell structure).
The original aim of the study was to carry out the micro-CT and image analysis of the core layer on exactly the same sample. While there was no issue with the micro-CT of this sample, the first image analysis results indicated that the 2D plane chosen for the analysis (the YZ plane) was not optimum for reasons already discussed in the Introduction section. Therefore a second sample was analysed using the optical technique only, this time imaging the XZ plane. The first sample (sample A) was extracted from the first specimen in the proximity of the notch located on the same side as the injection gate (see Figure 1a). The sample has the shape of a prism of dimensions 3.2 x 4 x 15 mm. The other two regions, for image analysis only (and designated samples B and C, see Figure 1b) were extracted from a second specimen, belonging to the same batch as the first one. Sample C had exactly the same dimensions as sample A, and the same location. Sample B was located next to sample C, with dimensions of 15mm x 15mm).



(a) (b)

Figure 1 – (a) Position and dimensions of sample A; (b) Position and dimension of sample B and C

Sample A was first analysed by micro-CT, then observed by the optical method. The face observed at the optical microscope was parallel to the YZ plane and coincided with the outermost surface in the X direction. The other two samples (samples B and C) were analysed by the optical sectioning method. Sample B was machined in order to reach a layer located at 1.6 mm from the top surface, thus coinciding with the plate's midplane, in order to analyse the fibre orientation in the core layer. Thus, the observed face of sample B was perpendicular to that of sample A which was analysed by the optical method. The observed face of sample C was that parallel to the YZ plane, and therefore parallel to that of sample A, with an offset of approximately 2 mm towards the negative X direction. Sample C was used to check if the fibre distributions in sample A and sample B were comparable, as explained in the following sections.

3 Methods

3.1 Optical method

In the optical method, 2D polished sections are taken from the area of interest and then evaluated using an in-house image analysis facility developed at the University of Leeds [for example 11]. Each fibre that meets the 2D section is seen as an elliptical footprint and measuring the ellipticity of these images allows the two polar angles, θ and φ, that specify the orientation of each fibre (with respect to the sectioned plane) to be determined.

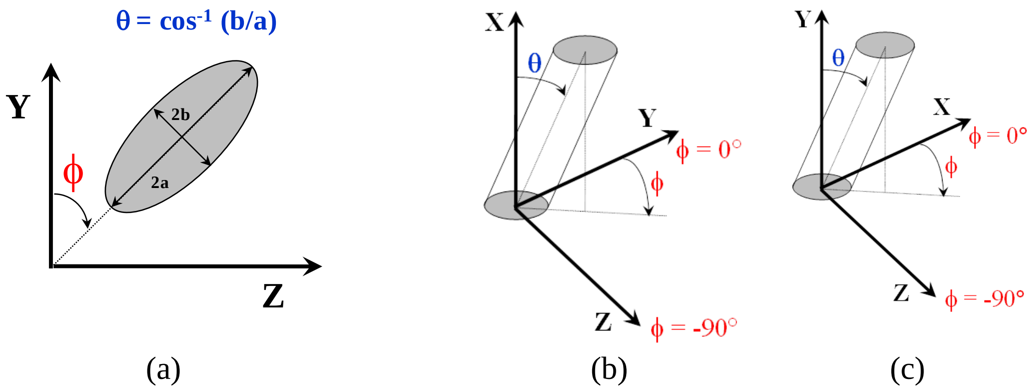


Figure 2 – (a) Definition of angles θ and φ (b) for the YZ plane (samples A and C); (c) definition of angle φ for the plane XZ (sample B)

As different samples (and section planes) were analysed in this study, the local definition of the angles θ and φ also varied. For sample A and sample C, the face to be analysed was parallel to the YZ plane. Therefore θ was defined the angle to the X axis and φ the angle formed by the projection of the fibre onto the YZ plane with the Y axis (Figure 2a,b). For sample B, the analysed face was parallel to the XZ plane, therefore θ was defined as the angle to the Y axis, whereas the angle φ was defined as the angle formed by the major axis of the elliptical footprint with the X axis (Figure 2c).

3.2 Tomography coupled with Mean Intercept Length analysis

The internal structure of sample A was acquired by micro-CT scans at the SYRMEP beamline of Elettra synchrotron (Trieste, Italy). This beamline is equipped for high-resolution micro-CT, with a maximum achievable resolution of approximately 9 micrometres at the time of the measurements. The high spatial coherence of the synchrotron light source allows for enhancing the contrast at the edges of glass fibres by applying Phase Contrast (PHC) imaging techniques.

The volume of sample A was reconstructed and analysed by applying a method based on the Mean Intercept Length (MIL) concept (MIL 3D). This method has been thoroughly described in previous papers [14, 15]. In order to describe local properties of prescribed dimensions, a portion of the entire reconstructed volume is analyzed, called Volume of Interest (VOI). The method allows for defining a second order tensor, called the MIL fabric tensor, having principal components T_1, T_2 and T_3 , which are related to the fibre orientation: the first principal direction, associated to the first principal component T_1, indicates the direction of preferred fibre orientation within the observed VOI. The method can also be applied to planar images (MIL 2D) and it was applied to the same image of sample B which was analysed by the optical method, as it is shown in Figure 3, where the polar graphs of the MIL values are superimposed to the corresponding VOIs. MIL values are evaluated using Quant3D software [17].

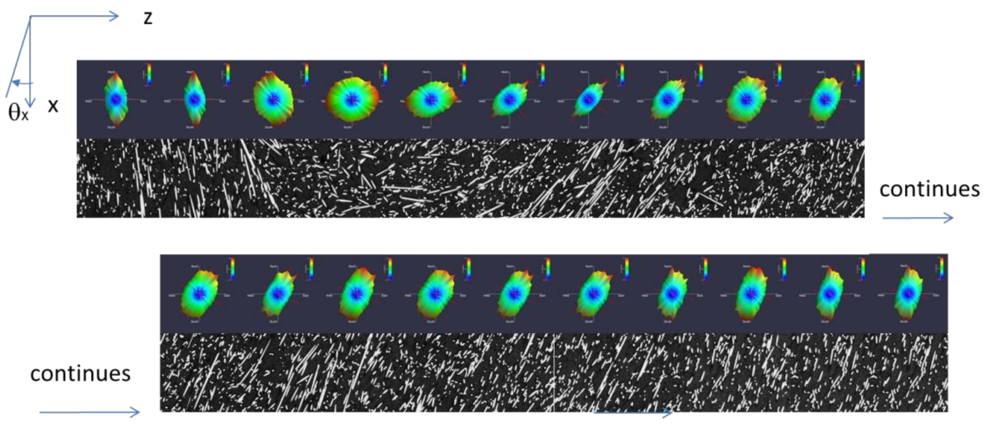


Figure 3 – MIL 2D polar graph superimposed to the corresponding VOI extracted from the image analysis of sample B

4 Results and discussion

Figure 4 shows the results of an image analysis scan of the surface of sample A (YZ plane). The results are presented as a grey scale map of the average value of the second order tensor $<\cos^2\theta_X>$, where the scanned region was divided into small cells for averaging (100 x 20 cells). A light colour shows that the fibres are preferentially aligned to the X axis in that region while a darker colour shows preferred orientation along a different axis. The notch root is located at the left of the scanned region.

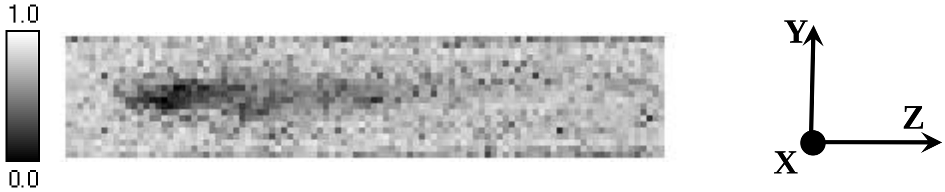


Figure 4 - Map of the value of the second order orientation tensor ($<\cos^2\theta_X>$) across sample A (YZ plane). The notch root is on the left.

The results show that in general the fibres are aligned in the flow (X) direction, but that there is a centrally located core region (close to the notch root) with preferred orientation along the Z axis.

To enable comparison with the micro-CT analysis the values of the angle θ (angle to the X axis) were determined along the sample length. The most interesting region of the sample is along the centre so a portion of the surface corresponding to the core layer was selected (between 40% and 60% of the sample thickness) and subdivided into smaller areas. The most frequent value of θ within each area, θ_{max}, is plotted in Figure 5 as a function of the distance from the notch root of the centre of each area. For comparison, results obtained by the MIL 3D (θ_{MIL}, angle between the first principal eigenvalue T_1 of the MIL fabric tensor) are superposted. In this case the agreement between the absolute values of θ_{MIL} and θ_{max} is not perfect although the peak positions are similar.
These differences can be explained by considering that by the MIL method, the whole volume is analysed, whereas by the optical method only the surface is analysed, and that the accuracy of measurement of angle θ by the optical method is known to be less accurate when fibres are almost perpendicular to the plane over which fibre footprints are analysed. These two explanations were then investigated by analysing a section of sample B parallel to the XZ plane by both the optical and the MIL method.

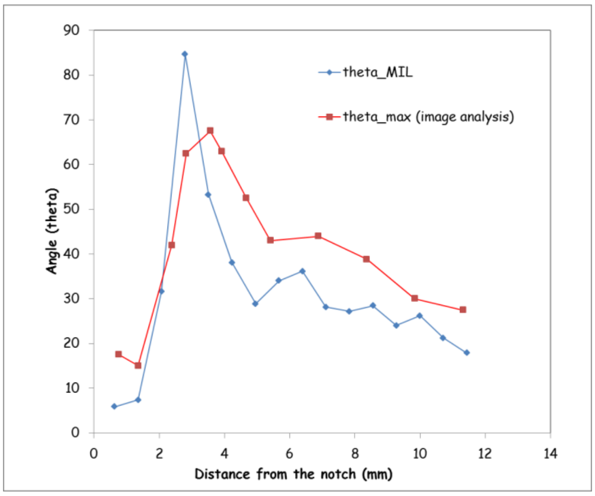


Figure 5 – A comparison between values of the angle theta evaluated by MIL and by the optical method in sample A (YZ plane).

Before proceeding to the analysis of sample B in the XZ plane, it was necessary to establish if fibre orientation distributions in sample B and sample A were similar (in the YZ plane). This was done through sample C, which has a face in common with sample B (YZ plane). First, the fibre orientation distributions on the same surface of sample A and sample C, as obtained by the optical method, were compared in terms of values of $\langle\cos^2\theta_X\rangle$, which are reported in Figure 6. It clearly appears that the two samples have very similar orientation patterns, although they are extracted from two different specimens. Therefore also sample B, which has a surface in common with sample C, can be considered homogeneous with sample A, thus allowing for comparison of fibre orientation distributions of the two methods.

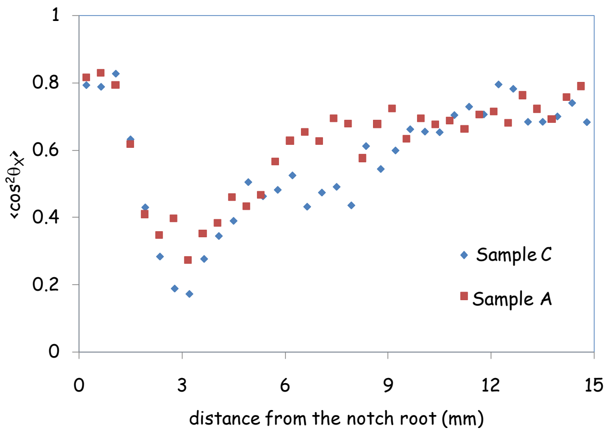


Figure 6 – comparison of fibre orientation between sample A and sample C

Moreover, before proceeding with sample B, the values of the angle θ_x evaluated by direct analysis of the surface of sample B (MIL 2D) and those of the same angle, evaluated as the projection onto the XZ plane of the first principal direction of the MIL 3D fabric tensor in sample A are compared in Figure 7. Although obtained on two different samples, the values are in good agreement.

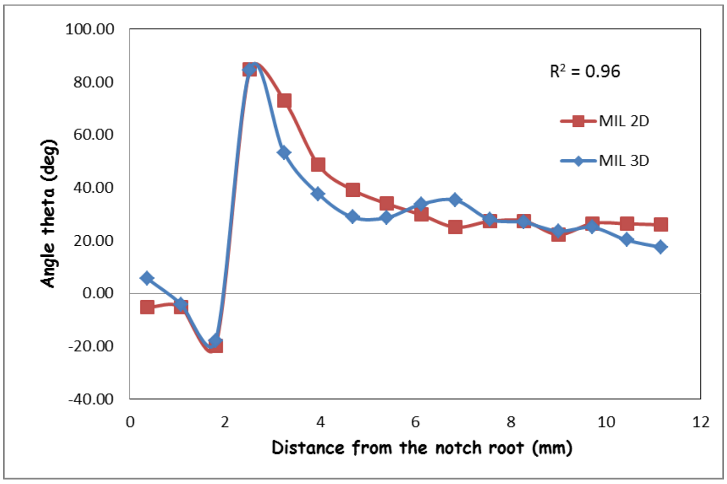


Figure 7 – values of the angle θ_x obtained by 2D MIL analysis of Sample B and the corresponding values evaluated by 3D MIL analysis of sample A

The definition of the angle φ evaluated on the XZ plane by the optical method coincides with that of angle θ_x used in both the 2D and the 3D MIL method. Therefore it is interesting to compare the values of φ_{max} obtained by the analysis of sample B by the optical method and those obtained by applying the 2D MIL method on the same image of Sample B used for the optical analysis. This comparison is reported in the graph of Figure 8 where values of φ_{max} and θ_x are plotted as a function of the distance Z form the notch root. On the basis of the results of the comparison between MIL 2D and MIL3D reported in the preceding section it is possible to affirm also that a very good agreement exists between θ_x evaluated by MIL 3D and φ_{max} evaluated by the optical method.

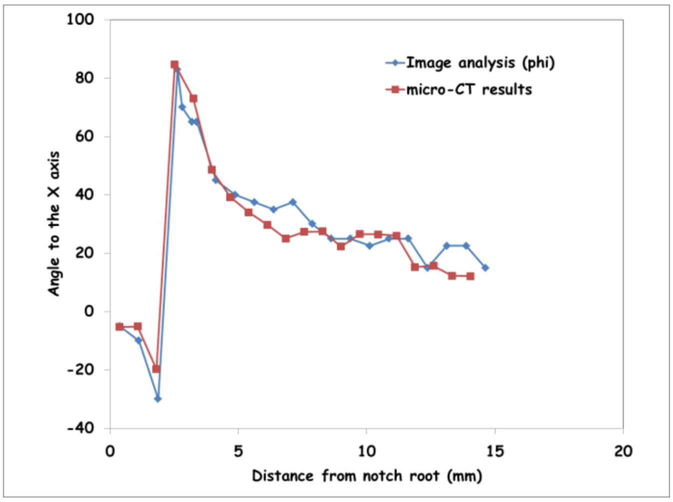


Figure 8 – Comparison between the values of angle φ_{max} evaluated by the optical method and of angle θ_x evaluated by MIL 2D, as a function of the distance from the notch root in sample B

It can therefore be concluded that the poorer agreement seen in Figure 5, between the angle θ from the MIL method and the angle θ from the optical method (YZ plane), is due to the errors in calculating θ when the fibres meet the sectioned plane at a small angle. When the most appropriate sectioned plane is used for the optical method (the XZ plane with the fibres mainly in the sectioned plane) then the agreement between the two methods is excellent.

5 Conclusions

The fibre orientation distribution in a material sample of short fibre reinforced polyamide extracted from an injection moulded notched plate was analysed using two different methods, one based on micro-CT and the MIL concept and another based on the classical optical section method.

The comparison was carried out with reference to the preferred fibre orientation, i.e. the fibre orientation angle occurring with the maximum frequency within the observed volume/area (the method based on micro-CT analyse volumes, whereas the optical method analyses surfaces). The analysis was limited to the core layer in proximity of one of the notches, where large variations in fibre orientation were expected.

A first comparison between micro-CT and the optical method applied to a transverse section (i.e. the specimen’s gauge section) showed non negligible differences which were attributed to the low accuracy of the optical method in measuring the orientation angles of fibres almost perpendicular to the observed surface and to the ambiguity of the sign of the angle.

To confirm this, the optical method was applied at the same location, but on a surface parallel to the core layer (a longitudinal section corresponding to the midplane of the specimen), the agreement between measurements based on MIL and those obtained by the optical method was very good. In facts, the analysis of the fibre orientation in the longitudinal midplane is free from both the ambiguity of the sign of the measured angle and the low accuracy affecting the measurements in the transverse section.

The results confirm the assumptions made to explain the differences observed in the first analysis and allow for concluding that, provided the right section plane is chosen for the optical method, the two methods give very similar results in terms of preferred fibre orientation. The choice of the method has to take into account that the optical method requires a simpler experimental setup but is fully destructive, whereas micro-CT results into non-destructive method (at least partially), but requires more expensive experimental facilities.

References

[1] Guild FJ, Summerscales J. Microstructural image analysis applied to fibre composite materials: a review. Composites 24 (1993) 383-393
[2] Fakirov S, Fakirova C. Direct determination of the orientation of short glass fibers in an injection-molded poly(ethyleneterephthalate) system. Polymer Composites 6 (1985) 41-46
[3] Fisher G, Eyerer P. Measuring spatial orientation of short fiber reinforced thermoplastics by image analysis. Polymer Composites 9 (1988) 297-304
[4] Moeginger B, Eyerer P. Determination of the weighting function g(βi,r,vf) for fibre orientation analysis of short fibre-reinforced composites. Composites 22 (1991) 394-399
[5] Bay RS, Tucker III CL. Stereological measurement and error estimates for three-dimensional fiber orientation. Polymer Engineering and Science 32 (1992) 240-253
[6] Davidson NC, Clarke AR, Archenhold G. Large-area, high-resolution image analysis of composite materials. Journal of Microscopy 185 (1997) 233-242
[7] Zak G, Park CB, Benhabib B. Estimation of three-dimensional fibre-orientation distribution in short-fibre composites by a two section method. Journal of Composite Materials 35 (2001) 316-339
[8] Clarke A, Davidson N, Archenhold G. Measurements of fibre direction in reinforced polymer composites. Jornal of Microscopy 171 (1993) 69-79
[9] Clarke AR, Archenhold G, Davidson NC. A novel technique for determining the 3D spatial distribution of glass fibres in polymer composites. Composites Science and Technology 55 (1995) 75-91
[10] Eberhardt C, Clarke A. Fibre-orientation measurements in short-glass-fibre composites. Part I: automated, high-angular-resolution measurement by confocal microscopy. Composite Science and Technology 61 (2001) 1389-1400
[11] Hine PJ, Davidson N, Duckett RA, Ward IM. Measuring the fibre orientation and modelling the elastic properties of injection moulded long glass fibre reinforced nylon. Composites Science and Technology 53 (1995) 125-131
[12] Kim EG, Park JK, Jo SH. A study on fiber orientation during the injection molding of fiber-reinforced polymeric composites (Comparison between image processing results and numerical simulation). Journal of Materials Processing Technology 111 (2001) 225-232
[13] Bernasconi A, Cosmi F, Dreossi D. Local anisotropy analysis of injection moulded fibre reinforced polymer composites, Composites Science and Technology, Vol. 68 (12), pp 2574-2581, 2008
[14] Cosmi F, Bernasconi A, Sodini N. Phase contrast micro-tomography and morphological analysis of a short carbon fibre reinforced polyamide. Composites Science and Technology 71 (2011) 23-30
[15] Bernasconi A, Cosmi F, Zappa E. Combined effect of notches and fibre orientation on fatigue behaviour of short fibre reinforced polyamide. Strain 46 (2010) 435-445
[16] Bernasconi A, Cosmi F. Fibre orientation at notches and fatigue behaviour of short fibre reinforced polyamide. ICCM 17 Edinburgh 2009
[17] Ketcham RA, Ryan T. Quantification and visualization of anisotropy in trabecular bone. Journal of Microscopy, 213 (2004) 158-171.