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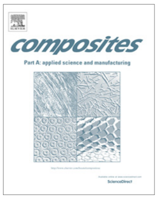

Non-destructive automatic determination of aspect ratio and cross-sectional properties of fibres

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ABSTRACT

A novel method for computerised estimation of the aspect ratio distribution and various cross-sectional geometrical properties of fibres in short-fibre reinforced composites is proposed. The method, based on X-ray micro-computed tomography, is non-destructive and does not require user intervention. Based on results on specially fabricated model material, the accuracy and precision of the method seems adequate. The method is applied in analysing a manufacturing process of wood fibre reinforced thermoplastic composite. The results indicate a significant decrease of the aspect ratio of fibres during the processing steps. Finally, the feasibility of the method is assessed by estimating parameters of a micromechanical model for flax fibre composites and comparing the results with those from tensile tests.

1. Introduction

Within micromechanical approach to estimating composite material performance, quantities describing the microscopic or mesoscopic structure of a composite are combined with mechanical properties of the constituents in order to estimate the macroscopic properties of the material [1–4]. For short-fibre reinforced composite materials the relevant statistical quantities describing the microstructure include, *e.g.*, diameter, length and orientation distributions of fibres, and volume fractions of the constituents. The microstructure of such materials is formed during the manufacturing processes that typically include, *e.g.*, pelletization, compounding, extrusion and moulding, each of which affect the geometry and distribution of the precursors [5]. Information on the microstructure of the material [6,7] evolving through the different processing stages may be used to optimise the processing chain and the properties of the final product through selection of proper processing methods [8,9] and their parameters [10].

Traditionally, composite material microstructure is studied by dissolving the matrix and studying the residual fibres by optical- or electron microscopy. Alternatively, polished cross-sections of the composite may be used. The methods are accurate, but time-consuming and destructive. Additionally, the applied chemical or mechanical treatments involved in the analysis process may change the structure of the fibres and, furthermore, in the dissolution process information about orientation and dispersion of the fibres is lost.

X-ray microtomography (X-μCT) is a non-destructive method to characterise the structure of heterogeneous materials. The method is based on computationally reconstructing the three-dimensional structure of the sample from a number of two-dimensional X-ray attenuation images taken from different directions [11]. Contrast in such a tomographic image typically originates from local X-ray attenuation coefficient that correlates with local density of the material sample. The spatial resolution available (~1 μm) is adequate for distinguishing the different phases, *e.g.*, fibres, matrix and void in many composites (see Fig. 1b). Traditional image processing algorithms [12] may be used to segment the different phases and thereby to produce, for further processing, a three-dimensional binarized image showing, *e.g.*, the fibre phase only.

Several methods for estimating fibre properties from a binary image have been recently proposed. Majority of the methods are

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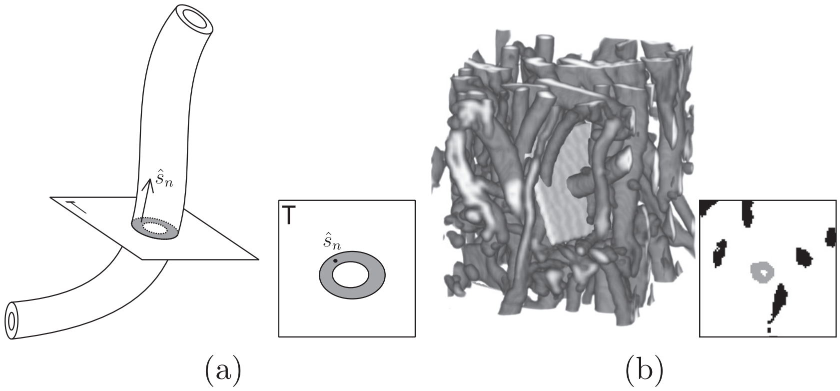

Fig. 1. (a) Slicing a single fibre. Slice T is taken such that its normal $\hat{s}_n$ is parallel with the local fibre orientation vector. (b) Small part of X-μCT image of a composite material (see Section 5). A slice taken from the shaded region is shown in the inset.

based on separating individual fibres from each other. Methods based on analysing the medial axis of fibres [13–17] are often applicable to solid fibres, whereas methods based on recognition of lumen [18–21] apply to hollow fibres. In the case of natural fibres, processing may break and crush the fibres such that their shape becomes highly irregular (see Fig. 1b). The fibres may thus be partially hollow and partially solid, hindering the use of above methods. Algorithms introduced in [22–24] are based on analysis of the local orientation of fibres in order to separate them from each other, and thus seem to be better suited for fibres of irregular shape.

Recently, we introduced an automated method to evaluate the fibre length distribution of short-fibre composites from a binarized three-dimensional tomographic image of material sample [25,26]. The method does not require identification of individual fibres and includes an algorithm for correcting bias caused by finite image size. In addition, transverse dimensions of fibres could be estimated based on a specific scaling assumption for fibre volume. In this work we relax the need for such an assumption and introduce an improved method to determine the statistical distribution of various geometrical properties of fibre cross-section as well as of fibre length. In particular, the present method can be used to correlate fibre length and cross-sectional properties such that, *e.g.*, the aspect ratio distribution can be obtained. The technique is validated using a model composite with known structure.

The method is then applied in assessing the effects of various manufacturing unit processes on the aspect ratio and on the cross-sectional shape of wood-fibres used as reinforcement in a biodegradable composite material. Finally, we apply the method in finding the necessary parameters for a micromechanical model for estimating Young's modulus of flax fibre composites, the results of which are compared with those from tensile tests.

2. Estimation of microstructural properties

Let us denote a three-dimensional binarized tomographic image of a short-fibre reinforced composite material sample by $I(x_1, x_2, x_3)$. The numeric value of each voxel, located at position $\vec{x} = (x_1, x_2, x_3)$, indicates whether or not that particular point is inside the fibre phase. In order to collect the necessary experimental data for estimating the statistical distribution of various structural descriptors of the fibre phase, the image I is sampled uniformly at random locations such that a statistically significant number of points $\vec{x}_n,\ n = 1, \ldots, N$ located inside the fibre phase is found. The local orientation of a fibre in the vicinity of point $\vec{x}_n$ can be found using the structure tensor defined by [27]

$$S_{ij}(\vec{x}_n) = \big(G_\sigma * (I_i I_j)\big)(\vec{x}_n), \tag{1}$$

where G_σ is a Gaussian function with zero mean and standard deviation σ in all directions, and $*$ denotes convolution. The quantities I_i are the partial derivatives of I, and can be approximated by [27]

$$I_i(\vec{x}_n) = \frac{\partial I(\vec{x}_n)}{\partial x_i} \approx \left(\frac{\partial G_\sigma}{\partial x_i} * I\right)(\vec{x}_n). \tag{2}$$

The eigenvector $\hat{s}_n$ of $S_{ij}(\vec{x}_n)$ corresponding to the smallest eigenvalue defines the local fibre orientation at $\vec{x}_n$.

Next, a slice is extracted around $\vec{x}_n$ such that its normal direction is parallel with $\hat{s}_n$ (see Fig. 1). The fibre cross-section is extracted from the slice and its area A_n is calculated. The cross-sectional dimensions of fibres with non-circular cross-section are characterised here using the lengths of the projections of the cross-section in its two principal directions [28]. For each cross-section we thereby find the principal dimensions d_{1n} and d_{2n}, with convention $d_{1n} \geq d_{2n}$. The length of the fibre containing $\vec{x}_n$ can be estimated using the constrained path transformation $I(\vec{x}) \rightarrow T(\vec{x})$, where T is an image where the value of each voxel is the length of the longest regular path passing through the corresponding voxel in I, and contained entirely inside the fibre phase. The degree of regularity of the path is controlled by a set of topological constraints specific for the algorithm [25,29]. The length L_n of the fibre at $\vec{x}_n$ is then defined as the mode of voxel values in T over the cross-section. Finally, two aspect ratios corresponding to the two principal dimensions d_{in} can be defined as $r_{in} = L_n/d_{in},\ i = 1, 2$.

Given the data set $\{\hat{s}_n, A_n, d_{1n}, d_{2n}, L_n, r_{1n}, \ldots\},\ n = 1, \ldots, N$, estimates for various multivariate distributions for the structural descriptors can be generated by a straightforward statistical binning. The probability that a randomly selected point in the sample is inside a fibre is proportional to the volume of the fibre. Thus, denoting by $\{\xi_1, \xi_2, \ldots, \xi_K\}$ any subset of the descriptors (random variables) $\{\hat{s}, A, d_1, d_2, L, r_1, \ldots\}$ for which the data is measured, the resulting normalised multivariate distributions are of a general type

$$f(\xi_1, \xi_2, \ldots, \xi_K) = \frac{1}{V_f} \frac{\partial^K V_f}{\partial \xi_1 \partial \xi_2 \ldots \partial \xi_K}, \tag{3}$$

i.e., ξ_k-distributions of fibre phase volume V_f.

3. Test case with a model composite

In order to substantiate the method, a model composite was made from hollow and solid fibres and moulding rubber. Steel capillary tube (0.5 mm outer diameter and 0.3 mm inner diameter) was used as hollow fibres, while copper wire (0.35 mm diameter) was taken as solid fibres. The tube and the wire were cut into