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A non-destructive X-ray microtomography approach for measuring fibre length in short-fibre composites

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ABSTRACT

An improved method based on X-ray microtomography is developed for estimating fibre length distribution of short-fibre composite materials. In particular, a new method is proposed for correcting the biasing effects caused by the finite sample size as defined by the limited field of view of the tomographic devices. The method is first tested for computer generated fibre data and then applied in analyzing the fibre length distribution in three different types of wood fibre reinforced composite materials. The results were compared with those obtained by an independent method based on manual registration of fibres in images from a light microscope. The method can be applied in quality control and in verifying the effects of processing parameters on the fibre length and on the relevant mechanical properties of short fibre composite materials, e.g. stiffness, strength and fracture toughness.

1. Introduction

Composite materials reinforced by synthetic short fibres are widely used in many applications such as interior panels in automotive applications. Due to their recyclability and renewability, also natural fibres have gained increased interest in such applications during the last years. In order to exploit the full potential of natural fibres in composite materials, better understanding on the effect of processing parameters on fibre morphology and spatial distribution is necessary.

Fibre content and fibre length are important material properties that provide mechanical strength, stiffness and toughness. For high-volume applications, a balance between processability and mechanical performance must be found. Short fibres make processing easier but may compromise mechanical properties. Long fibres tend to make melt processing more difficult since the viscosity of the fibre–matrix blend increases dramatically with increased fibre length. The mechanical properties of composite material reinforced with long fibres are generally better, since the ineffective length in the shear-stress transfer zones at the ends of the fibres is small compared to the total length of the fibre. These shear-stress transfer zones are the ineffective lengths close to the fibre ends, where the axial stress is built up by interfacial shear as stresses are transferred from the matrix to the aligned fibre. One example is wood-fibre reinforced plastics, where wood-polymer composites (WPCs) composed of wood particles with aspect ratio around 1–2 and polypropylene matrix, are readily extruded into planks as building material. The mechanical properties of such wood-fibre reinforced plastics are, in general, worse than of composites based on wood-fibres with retained fibre length [1,2].

Natural fibres are of finite length (e.g. bounded by wood tracheid dimensions), and the length is typically degraded during processing. The average fibre length and length distribution can be used both as indicators of processing conditions during manufacturing, and of mechanical properties of the product. Especially, properties such as strength and fracture toughness are affected by the spread and distribution tails [3,4]. A balanced fibre length is therefore necessary for finding a good compromise between processing properties and mechanical properties, since shorter fibres reduce process viscosity and longer fibres provide better reinforcement. In this respect, methods capable of measuring the fibre length and its distribution in fibre-reinforced composites are most desirable. These measures can be used e.g. for quality control of a composite material, tuning of processing parameters, or comparison and selection of fibres. In addition, such information will be

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most valuable as input in micromechanical models, to link microstructural properties to macroscopic engineering properties.

Fibre length distribution can be measured e.g. by dissolving the composite sample and recording the length of fibres that remain in the solvent using microscopic techniques or a suitable fibre length analyser (see e.g. Refs. [5,6]). Being a destructive method, the particular sample used in such a measurement is not available for further analyses. Obviously, such a straightforward method can give little additional information e.g. on the original structure of the fibre network and additionally, the dissolvement process may change the length distribution. Another possibility is to do the measurement before manufacturing the composite. However, the relevance of such measurement is questionable as, before forming the final structure, the fibres will go through severe processing stages possibly affecting the length distribution. Thus, *in situ* measurement achievable e.g. by X-ray tomographic methods is preferred.

1.1. X-ray microtomography

X-ray microtomography (X-μCT) is a non-destructive method for obtaining three-dimensional structure information of heterogeneous materials [7]. The method is based on taking a large number of two-dimensional X-ray projection images of a material sample from various directions. Within the absorption mode tomography, these projections are then reconstructed computationally to obtain a three-dimensional spatial distribution of the X-ray attenuation coefficient in the sample. Visualization of the distribution by a suitable colour map, e.g. grayscale, then yields a 3D image representing the structure of the material. Especially for non-crystalline (non-diffractive) materials the image thus obtained correlates with the local density distribution of the sample, see Fig. 1.

Traditionally, X-μCT imaging is available in many large-scale synchrotron facilities offering high-intensity, monochromatic X-ray light. Lately, however, table-top X-ray tomographic scanners have become increasingly accessible and adequate for heterogeneous materials research. Typical best resolution of the images is close to one micrometre in both types of imaging devices. Lately, devices capable of resolution below 100 nm have been introduced (see e.g. Ref. [8]). One important advantage of the method is that, despite cutting the sample to an appropriate size, little or no particular sample pre-processing is generally needed. Material samples can thus be imaged several times under various conditions, and the same physical sample can be studied with other complementary methods. In addition, various analyses of quite different nature can be made based on the same basic data, the 3D image of a material sample.

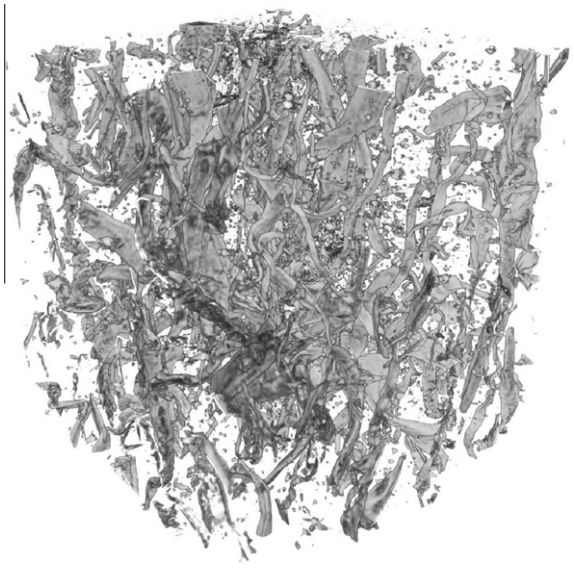

Fig. 1. 3D visualization of X-μCT image of Kraft fibre reinforced composite sample. The size of the image is $0.7 \times 0.7 \times 0.7\ mm^3$. The voxel size is 0.7 μm. The image represents a sample with 10% fibre volume fraction segmented such that the PLA matrix is not visible.

One of the limitations of the method, especially from the point of view of heterogeneous materials research, is the finite sample size [9]. The field of view of the tomographic device, and thereby the maximum physical dimension of the sample to be scanned, is proportional to the resolution used. The exact relation between resolution and field of view is given by the size of the detector CCD cell and the details of the optical set-up used. With the present devices, the maximum field of view with 1 μm resolution setting typically falls in the range 0.5–2 mm. Furthermore, the reconstructed images must usually be cropped prior to analysis to remove the boundaries of the physical sample and to obtain a final sample volume of a regular shape (see Fig. 1). The resulting size of the sample volume, i.e. the X-μCT image edge length, may thus be comparable with the length of a typical fibre in short-fibre composite material. The length distribution of fibres and fibre segments found in a sample volume is skewed towards small fibre length as compared to the actual fibre length distribution in an infinite medium. The reason for this is that the probability of a given fibre in the bulk material to be cut by the boundary of an arbitrarily placed sample volume, is proportional to the length of the fibre. Such a biasing may be significant especially in the case of small sample volumes, and cannot be fully corrected e.g. by simply discarding the fibres intersecting the boundary.

The problem of correcting for bias in a measured fibre length distribution has been discussed previously in different contexts by many researchers. For example, Fu et al. [10] studied the length distribution of composite fibres in a two-dimensional case. Mörling et al. [11] and the corresponding note [12] as well as Svensson et al. [13] studied the problem of fibre length distribution measurement in increment cores from living trees; see also references therein for examples of various correction methods. Typically, these methods are based on making specific assumptions on the form of the actual fibre length distribution, or require knowledge on which fibres intersect the boundary.

In this work a method for correcting the original fibre length distribution found in a finite sampling volume is developed. The length-dependent probability of fibres intersecting with the boundary of the sampling volume is taken into account. No *a priori* assumptions of the shape of the fibre length distribution are made. In addition, the proposed method does not require identifying the fibres that intersect the boundary of the sampling volume. Means to measure the bivariate distribution of number of fibres with respect to the longest and the shortest dimension of fibre is also discussed as a side effect of the correction method. The algorithm is first validated for simulated data and then applied to X-ray microtomographic images of wood fibre composite materials. The latter results are compared with independent experimental results obtained by extracting the fibres using a solvent and sampling individual fibre length values manually from two-dimensional images.

2. X-μCT fibre length distribution analysis method (XLDA)

Opening operation is an image processing filter that preserves structures smaller (or, equivalently, larger) than a specific size parameter r. The exact meaning of the parameter r is defined by the type of structuring element used in the opening filter. Within the granulometric approach, the size discrimination property of opening filter can be used to calculate size distribution of objects in the image. By normalizing the original image such that the