Quantification of micro-scale variability in fibre bundles

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A. Carbon fibres
B. Microstructures
C. Statistical properties/methods
D. Optical microscopy

Local variations in the random filament arrangement in carbon fibre bundles were determined by optical microscopy and automated image analysis. Successive steps of abrading, polishing and acquiring micrographs of the sample surface made it feasible to analyse the micro-structure over a series of cross-sections along the fibre bundle path. Random and systematic changes in local filament arrangements were determined. Systematic changes were related to the interaction of a fibre bundle with an intersecting binder thread leading to a local increase of the fibre volume fraction at the interface. Random clustering of filaments in areas of high or low fibre volume fractions within the fibre bundles were found to be unaffected by the relative position of the bundle.

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1. Introduction

Textile reinforcements utilised in the manufacture of composite components are based on filaments which are usually bundled in yarns and then processed into a fabric for easier handling. Due to this structure, fibre reinforced components are usually analysed on different levels, each with its own variability. Composite parts are made from layers of textile reinforcement (macro-scale) which consist of individual fibre bundles (meso-scale). The bundles can consist of several thousand filaments (micro-scale). It was shown that the variability at each scale affects the properties of a fibre reinforced composite component [1,2]. Depending on the scale of analysis, properties at the smaller scales of the material are usually homogenised, and any variability at these scales is ignored [3]. Attempts have been made to incorporate statistical variability at the smaller scales in multi-scale modelling approaches [4]. The variabilities are, however, usually obtained from randomly generated distributions only and are uncorrelated to the actual variability in the material.

For numerical analysis, fibre reinforcements are often represented by an idealised geometry of the smallest repeat in form of a unit cell [5]. To add variability of fibre bundle paths at the meso-scale, representative volume elements can be utilised [6]. In this case, the bundle paths exhibit a defined degree of variability; however, the geometry at the boundaries is usually periodic. As composite components are made from several layers of fibre reinforcement, this can introduce additional sources of variability. The stacking process introduces variability in between layers such as nesting or fibre bundle shape changes. Attempts were made to incorporate these bundle deformations into a geometrical model [7]. For the analysis of properties at the meso-scale, the filament arrangements at the micro-scale within the fibre bundles are usually treated as uniform [8]. The properties at the micro-scale are then easily estimated from bulk parameters such as the overall fibre volume fraction, Vf [9] in the yarn. Treating the fibre bundles as homogenous material ignores, however, any information about the local arrangement [10] or waviness [11,12] of the filaments. It was demonstrated numerically that the macro- or meso-scale failure of a part may be initiated by local stress concentrations at the micro-scale [13]. The local arrangements of filaments in fibre bundles should therefore not be ignored, but a systematic description is currently not available.

This paper analyses low crimp carbon fibre bundles and local effects of the reinforcement geometry on the micro-scale filament arrangements. Non-destructive examination methods such as micro-computed tomography are only feasible on very small samples and have a limited resolution [14,15]. Conventional optical microscopy is therefore used as an inexpensive alternative technique which also allows achieving a higher resolution. Automatic image analysis of optical micrographs of cross-sections of the material allows a detailed analysis in a time efficient manner [16]. The obtained data can be employed in multi-scale modelling approaches, for example, to improve the accuracy of local properties assigned to voxels in a finite element analysis [17].
2. Materials and methods

2.1. Fibre reinforcement and processing

Composite panels with dimensions 125 mm × 60 mm were made from a single layer of low-crimp plain weave reinforcement and a low viscosity epoxy resin system. The fabric consisted of aligned carbon fibre bundles with a filament count of 12 K (warp direction), stabilized by thin glass fibre weft yarns coated with a thermoplastic polymer (Fig. 1). The dry fabric was placed in a stiff metallic tool where the global fibre volume fraction, \( V_f \), is determined by the cavity height and was estimated to be 0.45. The fabric was saturated with a liquid resin matrix, MVR 444 supplied by the Advanced Composites Group, by circumferential injection at 2 bar pressure. The resin was preheated to 70°C before the injection pressure was applied which reduced the initial viscosity to approximately 0.025 Pa s.

2.2. Sample preparation and image processing

Micrographic analysis of cross-sections of these specimens, cut perpendicular to the nominal fibre bundle path, allowed to identify the filament distribution within the carbon fibre bundles (Fig. 2). By successively removing thin layers of material at the surface of these specimens and acquiring new images, the filament arrangement can be determined at different positions along the fibre bundle path [18].

Stitching of overlapping images (Fig. 2C) allows large area images of fibre bundle cross-sections to be studied at high resolution [19,20]. A stitching code was implemented based on fast-Fourier correlations programmed in Matlab [21]. Micrographs were acquired at a high magnification which resulted in a pixel size of 0.09 μm (Fig. 2B). All individual filament cross-sections were determined by means of edge detection based on colour gradients in the images and fitting ellipses to the identified boundaries [16]. It was estimated that the fitted ellipse overestimates a filament cross-sectional area by about 3% on average.

Due to differences in material hardness between fibres and polymer matrix, the mechanical polishing process during sample preparation can cause filaments, located at the edge of the fibre bundle, to be abraded more than filaments in the bulk of the material. These filaments are therefore not within the limited depth of field of a conventional optical microscope imaging the bulk of the material. This results in blurred images of these filaments located at the bundle edge which can therefore not be detected with the employed image analysis method (Fig. 3). An additional false detection rate during image processing, e.g. over-/underestimation or non-detection of filament cross-sections within the bulk of the material, of approximately 2% was estimated. For these reasons, approximately only 11,500 filaments were detected during the image processing of a 12 K fibre bundle. For further interpretation of the results, the acquired micrographs and measured data of the detected filaments have been made publicly available [22].

3. Definition of fibre bundle descriptors

3.1. Fibre bundle boundary

The fibre volume fraction in a fibre bundle is defined by the combined cross-sectional area of all filaments divided by the entire area of the bundle. The cross-sectional areas of all filaments within a fibre bundle are determined during image analysis. The total bundle area is defined by a virtual boundary enclosing all filaments in the fibre bundle cross-section (Fig. 2C). To determine the envelop enclosing all filament cross-sections, the location of the top and bottom filament edge is determined over the width of the bundle. To avoid sudden changes in the estimated boundary, the filament locations are determined for a discrete step size. The
width of this step size was selected to be approximately equivalent to the average diameter of the filaments. This process allows a more precise estimation of the actual fibre bundle area compared to, for example, fitting an ellipse enclosing all filaments [23].

The identified filament positions can be used to estimate the bundle width and thickness. The bundle dimensions are defined as the maximum distance along a reference direction between two filament centres on opposite sides of the fibre bundle plus one nominal filament diameter. Unlike the height, the exact determination of the bundle width usually depends strongly on the accurate detection of filaments at the narrow tips of the fibre bundles (Fig. 3). However, this accuracy may be limited due to the aforementioned problems related to the mechanical polishing process.

3.2. Fibre bundle volume fraction

The filament arrangement within fibre bundles is not uniform. A qualitative measure of the variations in local filament densities can be achieved by measuring greyscale levels in vertical and horizontal slices [24]. However, for exact quantification of the local \( V_f \), the exact description of all filament positions is necessary [16].

After determining all filament cross-sectional areas and the bundle envelope (Fig. 4A), the fibre bundle is segmented into a finite number of vertical slices. The width of these segments was selected to be equal to 1 pixel. The absolute number of filaments detected in each slice decreases at the tips of the fibre bundle as expected (Fig. 4B). If, however, the relative amount of filaments within the fibre bundle envelop is calculated, the \( V_f \) is more uniform (Fig. 4C). A decrease in \( V_f \) towards the tips of the fibre bundle [25] could not be found. The peaks in \( V_f \) at the bundle tips occur since only a single or a very small amount of vertically arranged filaments are present, and the step size to determine the bundle boundary (Section 3.1) is smaller than the respective filament diameters. Hence, a relatively large ratio of filaments can be present in an analysed segment locally. Other artefacts, such as separations within the fibre bundle which may be caused by handling of the dry textile (Fig. 4A), decrease the absolute number of filaments locally and therefore also reduce the internal \( V_f \) in this area. Even though the \( V_f \) is relatively constant, gradual changes over the width of a fibre bundle may be present.

3.3. Local fibre volume fractions

The cross-sectional area of a fibre bundle can be divided into an array of polygons by use of Voronoi cells [26]. Each point within the fibre bundle cross-section is associated to the centre point of a filament closer than any other filament centre. This results in a tessellation of the entire domain into multiple polygons (Fig. 5). The ratio of the determined filament cross-sectional area to the area of the enclosing polygon reflects the local \( V_f \). Alternatively, the bundle could be divided into a regular grid and the \( V_f \) within each cell be calculated [27]. Unlike the Voronoi cell analysis, the \( V_f \) results would, however, depend on the selected grid size [28].

A limitation to the concept of Voronoi cell tessellation of a fibre bundle is that the analysis is based only on filament centre positions. Varying fibre diameters or apparent ellipticity of the cross-sectional areas, e.g. as a result of filament waviness inside the fibre bundle which causes filaments not to be sectioned perpendicular to their path, are ignored. The determination of the local \( V_f \) could be refined if the actual cross-sectional area of the filaments are considered [29], i.e. the Voronoi cell in this case is not dependent on filament cross-sectional centres but based on their elliptical perimeters. In this case, the Voronoi cell boundary is not any longer located equidistant between two filament centres but located half way between the closest points of two filament boundaries. In addition, the number of strongly misaligned filaments which will result in more pronounced ellipticity of individual filament cross-sections will be limited for the analysed uni-directional fibre bundles [11,12]. For analysis of the local \( V_f \) in this work, the error of ignoring the filament cross-sectional shapes for the Voronoi cell tessellation of a fibre bundle is therefore considered small as the
average misalignment of filaments is small. Hence, the variation in ellipticity of the filament cross-sections is limited and the simpler Voronoi cell tessellation based on filament centres can be used.

Attempts were made to describe the packing configuration of filaments by the Voronoi cell tessellation [30]. Voronoi polygons sharing a side with each other represent filaments which are nearest neighbours. The average number of polygon edges is therefore equivalent to the average nearest neighbour count. This method ignores, however, varying distances between filaments. The average nearest neighbour count for any fibre bundle was estimated to be 5.7. This suggests that the filaments have a tendency to be arranged in a hexagonal array (number of edges = 6) which corresponds to values reported in the literature [31,32]. However, the filaments are not necessarily arranged hexagonally (Fig. 5). Analysing the angular distribution of filament centre points between nearest neighbours, it was demonstrated that the filaments at this low Vf have the tendency to be arranged randomly [16].

4. Changes in fibre bundle composition

4.1. Fibre volume fraction variability

For two fibre bundles, a series of 12 slices (four of which are shown in Fig. 6) was imaged at successive positions over a length of about 5.3 mm. This corresponds approximately to the length of one unit cell (Fig. 1). During each re-polishing step, between 0.3 mm and 0.6 mm of material was removed. This step size is too large to reconstruct individual filament paths [11], however, it has been demonstrated that the majority of filaments in unidirectional fibre bundles only change positions within a limited cross-sectional area [12]. Hence, the effects on Vf distributions due to filament path variability between different bundle cross-sections will be limited as well.

The filament cross-sections and fibre bundle boundaries were determined for each section, and the resulting Vf (Section 3.2) are shown in Fig. 7A. It can be shown that the average Vf is not constant and varies significantly over the analysed bundle segments. In areas where a transverse binder yarn is present, Vf increases. As single layers of material were analysed at a constant mould cavity height, the compaction force acting on the fibre bundles in this area increases. This results in a compaction of the filaments locally, and hence in a local increase in Vf. The value of Vf and the fibre bundle height have therefore opposite trends (Fig. 7A and B). In areas where the fibre bundles are only compacted by the mould tool, the Vf is lower and the bundle thickness higher. The maximum peak height of Vf depends also on the bundle width (Fig. 7C). At the same height, a more narrow fibre bundle will have a higher Vf. The changes in fibre bundle width are, however, gradual and hardly affected by local bundle compaction. This suggests that changes in width of a fibre bundle are more affected by the initial state during processing. A similar effect was found when analysing the aspect ratios of bundle dimensions in a twill weave composite, which suggest the bundle width to be fairly constant over the length of two unit cells [33]. It should mentioned that the
The estimated standard deviation in $V_f$ for the samples analysed in this work seems to be independent of the slice position and ranges between 0.06 and 0.09.

The variation in fibre bundle dimension over approximately one unit cell length in this work was determined to be about 3.2% for the thickness and 1.3% for the bundle width. These variations are lower than presented for the randomly selected bundles found in twill weaves (14% and 6% for bundle thickness and width) [23] or non-crimp fabrics [34]. However, these changes in bundle dimensions will be affected by the fabric geometry and the amount of compaction [16]. It should be kept in mind that, depending on the bundle shape (e.g. lenticular), the bundle tips may not involve many filaments [35] and the width may therefore not significantly affect the average yarn $V_f$. For other properties such as permeability, the bundle tips may however significantly affect the resin flow.

4.2. Change in local fibre arrangements

The surface of the determined fibre bundle envelope is irregular in areas of lower local $V_f$, e.g. where no transverse binder thread is present (Fig. 6). It does, however, gradually change and becomes smooth in areas where the transverse thread is crossing. The interface between the fibre bundles and the thin glass threads is subjected to a higher normal force during mould closure which results in a local compaction of filaments. The filaments at the perimeter of the fibre bundle are able to move and rearrange along the path of the continuous glass thread. As the glass threads are coated with a thermoplastic material and have a low fibre count, movement of the filaments within these binder yarns are thought to be insignificant.

Analysing changes in local $V_f$ on bundle cross-sections by means of Voronoi cell analysis, a higher $V_f$ can be observed at the boundary where the transverse thread is present (Figs. 6D and 8D). This suggests that the filaments rearrange locally in response to the compaction force. It can also be seen, that this higher $V_f$ is confined to a small area only and does not affect the filament arrangement in the entire fibre bundle. The affected zone has been estimated to have a width between 5 and 13 filament diameters for the analysed samples. This zone may be larger if samples with a higher global $V_f$, i.e. larger amount of compaction, are analysed.

The filament arrangements in the centre of the fibre bundle seem not to be affected by the local increase of $V_f$ at the bundle perimeter. Areas of low or high $V_f$ in the centre of the fibre bundle change gradually independent of the relative position of the weft thread. Within the fibre bundle, filament clusters of high $V_f$ are present and separated by areas of low $V_f$. For this case, the filament arrangements were quantified in terms of distance and angle distributions between nearest neighbours and have been presented in an earlier study [16]. In the case of liquid resin flow during composite processing, it is likely that these areas of lower local $V_f$ will act as main flow channels [36].

4.3. Effects of voids

In several bundle sections, voids were detected after abrading and re-polishing the sample surfaces (Fig. 6D). These voids are a result of air entrapment in the liquid resin matrix during the impregnation process. In maps of local $V_f$ these voids appear as areas of reduced $V_f$ within the fibre bundle (Fig. 8D). As the voided area does not contain a matrix to fixate the filaments locally, filaments in this area may fracture during the polishing process. The voids therefore appear to contain no filaments when using optical microscopy, which may not be the case.

However, the trends in determined $V_f$ in this work (Fig. 7A) seem not to be affected by the appearance of voids. It was observed that all detected voids originate in resin rich zones in between filaments (Fig. 9). The voids may change shape further along the fibre bundle path but they are surrounded by an area of increased $V_f$. These densely packed filaments are restricted in their movement [18]. This suggests that voids in the samples analysed here are formed only in areas of the fibre bundle which contain none or just a very limited amount of filaments.

The additional edge of the void may lead to poor image contrast at its circumference due to rounding effects of the filaments during polishing. The effect seems, however, to be small when analysing the changes in $V_f$ over the width of a sample in adjacent bundle sections (Section 3.2). Therefore, no adjustments have been made to the analysis of the filament arrangements and these voids are treated as resin rich zones in the fibre bundle.

4.4. $V_f$ effects on properties

The increase in $V_f$ of the low crimp bundles at the crossing points with the binder threads will result in differences in local permeabilities [27]. Hence, the permeability in these zones will decrease which will increase the probability of gas entrapment during impregnating flow in these zones. On the other hand, the influence on the elastic mechanical properties in bundle direction is expected to be small. However, the observed increase in $V_f$ at
the crossing of two fibre bundles is probably present in the case of a similar transverse weft bundle as well. Numerical simulations have shown that the stresses and strains in the bundles transverse to the loading direction are increased. Using the example of plain weave unit cells, damage initiation was always observed at the interface of transverse fibre bundles [37,38]. The increased \( V_f \) at the interface will lead to a more brittle material which may exceed the matrix strength between individual filaments locally [13]. Incorporating the observation of increased \( V_f \) at the fibre bundle interface rather than using random distributions in numerical meso-scale models [37] will probably further highlight this effect. It is therefore likely, that this causes cracks in [24,39] or around [40] the transverse fibre bundles to form during mechanical loading.

5. Concluding remarks

Changes in the random arrangement of filaments in fibre bundles were quantified along the bundle path. A local increase of the fibre volume fraction, \( V_f \), towards the interface at cross-over points with transverse threads has been observed. In addition, local clusters of high \( V_f \) within a fibre bundle have been found which seem not to be affected by adjacent or interlaced fibre bundles. These clusters have different local filament arrangements and affect the fibre bundle properties differently. For example, the permeability in these high filament density clusters is decreased and may prevent full saturation with a liquid resin matrix during impregnation locally. If an area of low filament density is located within such a cluster of increased \( V_f \), this may cause air entrapment.
in this area. In addition, larger areas of low $V_f$ may act as main fluid transport channels which dominate the fibre bundle permeability. This could lead to local race tracking inside the fibre bundle leading to gas entrapment within the adjacent clusters of higher $V_f$.

In addition, the increased $V_f$ at the interface between two intersecting fibre bundles will lead to additional stress concentrations during mechanical loading as a result of an increased difference in stiffness. When numerically analysing the properties of a fibre bundle, the determined systematic and random changes in local $V_f$ inside a fibre bundle can be taken into account for more accurate predictions.

The obtained results are specific for the analysed fabric at a single global $V_f$ and should be confirmed on other fabric styles, different $V_f$ and layered materials. It is thought, however, that the effect of local compaction of a fibre bundle at the interface between interlaced fibre bundles will also be found under different conditions. This effect is expected to be more pronounced when layers of transversely arranged bundles are compacted within a mould tool. On the other hand, the local compaction effects will be less distinct when bundles are aligned in parallel and nesting is allowed. But even in this case, at an increased amount of compaction, similar local effects at the bundle interface are probable to occur as the filaments within individual fibre bundles are limited to move individually due to long range entanglements. Unlike in the analysed case of a single bundle being compacted between two rigid plates, the compaction induced effects at the interface for layered materials will be shared between adjacent fibre bundles.

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