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Accuracy of fiber length measurements using X-ray computed tomography for the analysis of composite materials

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Abstract

X-ray computed tomography is capable of measuring non-destructively the fiber geometrical characteristics that deeply affect both physical and mechanical properties of fiber-reinforced polymeric products. In particular, X-ray computed tomography can overcome the limitations of conventional methods used for fiber orientation analysis and fiber length measurement, which are mainly based on time-consuming and destructive optical analyses. This work especially addresses the case of fiber length measurement, which is particularly complex because – differently from the case of fiber orientation and concentration analyses – it needs the individual fibers to be identified and segmented. Moreover, the accuracy of such measurements has not been thoroughly investigated so far. In this work, a new method for accuracy evaluation is proposed. The method is based on the use of a newly developed task-specific reference object, which has been designed, manufactured and calibrated with respect to the length of a selection of fibers with different lengths and different configurations.

X-ray computed tomography, metrology, measurement accuracy, fiber length measurement, fiber reinforced polymers

1. Introduction

Fiber-reinforced polymers (FRP) are composite materials that are extensively used in industry to produce (e.g. by injection molding) components with enhanced mechanical properties and lightweight [1]. However, physical and mechanical properties are significantly influenced by the residual fiber geometrical characteristics, including fiber orientation, length and concentration. Such characteristics are typically evaluated by optical methods performed after sectioning the part or after pyrolysis of the polymer matrix, hence being particularly time consuming [2,3].

X-ray computed tomography (CT) is an advanced and accurate non-destructive measuring technique [4] that allows overcoming the limitations of conventional methods. In particular, CT is the only available technique capable of evaluating in a non-destructive way and in a relative short time the fiber geometrical characteristics mentioned above in FRP components. On one hand, CT data are already successfully used for conducting accurate fiber orientation and fiber concentration analyses of FRP components, which are respectively based on the evaluation of density gradients and on a global segmentation of the fiber with respect to the matrix [5,2]. On the other hand, for fiber length measurements each individual fiber has to be identified and segmented, which is a complex operation due to the typical formation of large fiber agglomerations with various intricate configurations after the injection molding process. Moreover, there is a lack of experimental methodologies for evaluating the accuracy of CT fiber length measurements [6].

This work aims at developing a new experimental methodology to be used for accuracy evaluation of fiber length measurements performed by means of X-ray computed tomography.

2. Material and methods

The accuracy of CT fiber length measurements is assessed in this work by using the practical approach defined in the guideline VDI/VDE 2630-2.1 [7], that is based on the comparison of CT measurements with reference measurements. For this purpose, a task-specific reference object including calibrated fibers has been developed as described in Section 2.1. CT measurements of such object are described in Section 2.2, while Section 2.3 is focused on the uncertainty determination.

2.1. Reference object

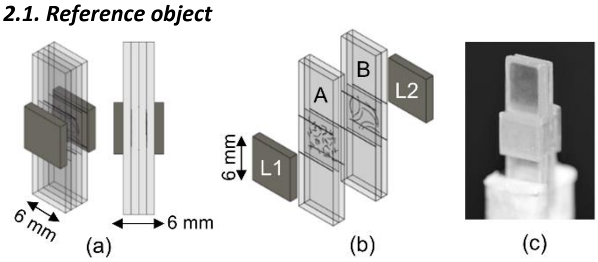
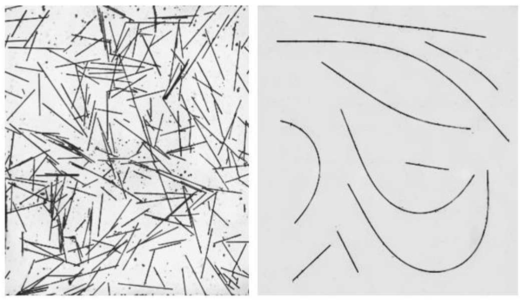


Figure 1. Dismountable reference object: (a) Schematic representation of the object in the assembly state; (b) exploded view showing the two fiber-reinforced polymeric layers (L1 and L2) and the sub-samples A and B containing the calibrated fibers; (c) picture of the reference object.

In this work, the targeted actual workpieces are injection molded components characterized by a polymer matrix reinforced with glass fibers. In order to apply the VDI/VDE 2630-2.1 [7] approach, the reference object has to be similar to the actual workpieces in terms of material, size, geometry, measurement procedure and environmental conditions (e.g. temperature and humidity). As seen in Figure 1, The reference object was produced with a dismountable configuration including 4 components: (i) two sub-samples (A and B) characterized by individual fibers dispersed between two thin (1 mm) transparent polymer layers (whose material density is similar to the matrix density of actual components), enabling optical calibration of the fiber length and (ii) two fiber-reinforced polymeric layers cut out from an actual component (L1 and L2) surrounding the two sub-samples to satisfy the similarity conditions reported above. Sub-sample A contains straight fibers with different configurations (see Figure 2-a), while sub-sample B only contains isolated fibers (both straight and curved; see Figure 2-b). A total of 41 fibers ranged between 0.076 mm and 4.2 mm belonging to sub-samples A and B were calibrated by means of a CMM equipped with image probing sensor (Werth Video Check IP 400). Possible form errors and mis-orientation of fibers where included in the calibration uncertainty.



(a) (b)

Figure 2. Fibers disposition in sub-sample A (a) and sub-sample B (b).

2.2. CT measurements

A metrological micro-CT system (Nikon Metrology MCT225) was used to scan five times the reference sample described in Section 2.1. The scan resolution was maximized by keeping the focal spot size at a minimum (i.e. 3 µm) and by achieving a small voxel size of 3 µm. The analysis and visualization software VGStudio MAX 3.1 (Volume Graphics GmbH) was used for CT data elaboration. In particular, in order to accurately identify and segment the glass fibers, a local-adaptive surface determination procedure was conducted. The length of the calibrated fibers was then measured by virtual probing of fiber points operated manually by the analyst.

2.3. Determination of measurement uncertainty

The reference object described in Section 2.1 and the approach described in the guideline VDI/VDE 2630-2.1:2015 [7] were used to determine: (i) systematic errors (i.e. difference between average CT measured length and corresponding calibrated value) and (ii) uncertainty of CT length measurements that can be associated to actual FRP components similar to the reference object.

3. Results and discussion

Figure 3 shows the deviations calculated as difference between measured fiber lengths and corresponding reference values. The results are reported with the associated CT expanded measurement uncertainty (U_{CT}), determined as explained in Section 2.3. It was found to be equal to 8.7 µm. The calibration uncertainty (U_{cal}) is reported in the diagram as well (dashed red lines). The maximum registered deviation is equal to 9 µm (for two fibers), while the major part is lower than 6.6 µm. The curved fibers show deviations comparable to those obtained for the straight fibers. The maximum ratio between the uncertainty and the measured length is equal to 12 % for the shortest measured fiber (i.e. with length equal to 76 µm). The ratio between the average systematic error and the measured length is equal to 2.4 % at maximum, for the shortest measured fiber. The normalized error E_N [8] was used to evaluate the agreement between CT measured values (X_{CT}) and reference values (in this case calibrated values, X_{cal}), according to the following formula:

$$E_N = |X_{CT} - X_{cal}| / \sqrt{{U_{CT}}^2 + {U_{cal}}^2} \quad \text{(Eq. 1)}$$

When E_N is below 1 a good agreement exists, while if E_N is above 1 the results are not in good agreement. The normalized errors obtained for the CT measurement of the 41 calibrated fibers were found to be below 1 in all cases, proving the good agreement between CT measurements and reference measurements.

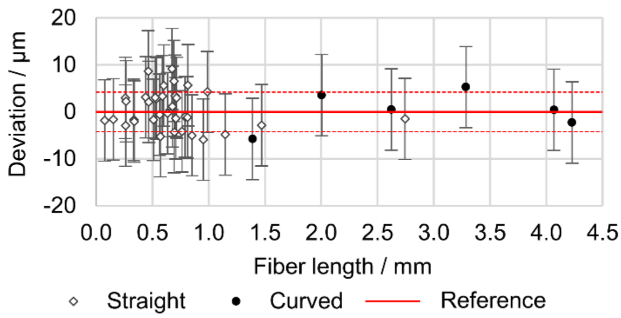


Figure 3. Deviations between measured fiber lengths and corresponding calibrated values for straight and curved fibers. The dashed red lines are positioned in correspondence of the expanded calibration uncertainty $\pm U_{cal}$

4. Conclusions

A new experimental methodology for evaluating the accuracy of CT measurement of fiber length in composite materials was presented. In particular, a reference object was designed, produced and calibrated in order to compare CT fiber measurement results with calibrated values. Outcomes obtained by processing 5 repeated CT scans showed that, with the specific scanning parameters used in this work, percentage deviations below 2.5 % can be achieved for fiber lengths ranged between 0.076 mm and 4.2 mm. The measurement uncertainty was determined to be equal to 8.7 µm, with maximum ratio between uncertainty and measured length equal to 12 % for the shortest measured fiber.

The proposed methodology will be used in future works to provide an experimental validation to the available software tools capable of measuring automatically the fiber length from CT data.

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