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Optimization of fiber measurements performed by X-ray computed tomography to predict the mechanical properties of fiber-reinforced polymeric components

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Abstract
The mechanical properties of fiber-reinforced polymers (FRP) depend significantly on the geometrical characteristics of fibers, including orientation, length and volume fraction. The conventional methods used to analyze such characteristics often involve the use of destructive and time-consuming techniques. In this context, X-ray computed tomography (CT) offers the advantage of performing a complete and non-destructive analysis of fibers. This work focuses on the optimization of CT fiber measurements to predict the mechanical properties of FRP components accurately. In particular, the proposed optimization procedure includes the use of reference samples similar to actual industrial FRP components to assess and correct systematic errors and determine the uncertainty of CT fiber measurements. The ultimate tensile strength (UTS) of injection-molded glass-fiber-reinforced specimens was successfully predicted from the improved CT characterization with a deviation below 7% of the experimental UTS.

X-ray computed tomography, fiber-reinforced polymers, injection molding, fiber measurement, mechanical properties

1. Introduction

Fiber-reinforced polymers (FRP) are composite materials that have been increasingly used for structural parts thanks to the attractive cost/performance ratio, low density, and ease of fabrication of complex shapes by using conventional molding processes, such as injection molding (IM) [1]. The combined effect of orientation, length and volume fraction of the residual fiber determines the final tensile properties of the IM FRP components [2]. Such fiber characteristics are typically evaluated by optical methods performed after sectioning the part or after pyrolysis of the polymer matrix, hence being destructive and particularly time-consuming [3,4]. In addition, tensile properties are usually assessed from load/displacement curves obtained by destructive tensile testing [5]. Micro X-ray computed tomography (CT) is an advanced measuring technique that can potentially overcome the limitations of conventional methods, enabling a complete and non-destructive characterization of FRP materials in a relatively short time [6].

This work focuses on improving the accuracy of CT fiber measurements, with the final aim to accurately predict the tensile properties of FRP components. In particular, an optimization procedure – based on the use of a reference sample similar to actual industrial FRP components – is proposed to assess and correct systematic errors and to determine the uncertainty of CT fiber measurements. An industrial case of IM glass-fiber-reinforced components was addressed to demonstrate that the CT fiber characterization, improved with the proposed procedure, can successfully predict the ultimate tensile strength (UTS) of the investigated components.

2. FRP specimens and CT measurements

The FRP samples investigated in this work are injection molded tensile specimens with 4 mm thickness, 10 mm gage section, and a total height of 154 mm. The used material is Celanese Celstran® PP-GF30-0403 P10-FS with 30% of fiber weight fraction and fiber diameter equal to 17 µm. The IM process was conducted using an 1100 kN hydraulic injection molding machine (HM110, Wittmann-Battenfeld, Austria) with a 35 mm diameter screw.

A metrological micro-CT system (MCT225, Nikon Metrology, UK) was used to scan the specimens' central region. The obtained CT reconstructions were then used to analyze the fiber geometrical characteristics, including fiber orientation, fiber length, and fiber volume. Three different voxel sizes were tested: 4, 6, and 9 µm.

3. CT fiber geometrical characterization

The optimization of fiber geometrical characterization was addressed using the approach defined in [7], which is based on the comparison of CT measurements with reference measurements [8]. For this purpose, task-specific reference FRP objects including glass fibers with calibrated length, volume, and orientation have been developed as proposed in [7]. In order to verify and correct the systematic measurement errors, such objects were scanned within the same scanning batch of actual FRP specimens and using the same scanning parameters. Particular attention was given to the comparison of results obtained with different voxel sizes. In principle, the voxel size should be minimized to improve the CT scanning resolution. However, larger voxel sizes enable the possibility of scanning larger objects or gathering data on larger regions of the scanned objects, leading to more representative characterizations.

3.1. Fiber orientation

Fiber orientation analyses conducted with the software VGStudio MAX 3.2 (Volume Graphics GmbH, Germany) showed that, independently from the voxel size, the CT measured angles are very close to the reference angles of the calibrated objects. Figure 1a illustrates the comparison on 5 fibers taken as examples, with angles from 18 to 85 degrees. Maximum percentage deviations were determined to be equal to 3.6% for a voxel size of 4 µm, 4.9% for a voxel size of 6 µm and 5.6% for a voxel size of 9 µm. Besides, Figure 1b shows that the diagrams of the orientation tensor computed with the three different voxel sizes of 4, 6, and 9 µm are almost superimposed.

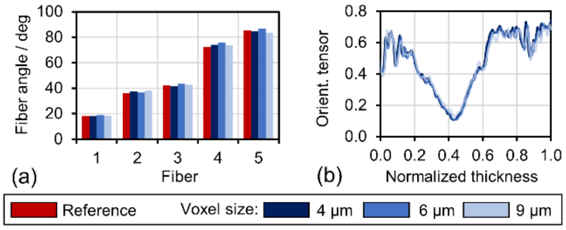

Figure 1. Fiber orientation angles measured by CT and compared with reference values (a); orientation tensor obtained with three different voxel sizes (b).

3.2. Fiber length

The measurement of fiber length was performed using the software open_iA (*https://github.com/3dct/open_iA*). The average registered deviations with respect to the reference lengths were equal to about 20 µm for all the investigated voxel sizes, while the maximum deviations were equal to 37 µm for voxel sizes of 4 and 6 µm, and 62 µm for a voxel size of 9 µm. The maximum percentage deviation is, in general, below 12% for all the measured fiber lengths.

3.3. Fiber volume

The effect of the voxel size on the CT fiber volume measurements was addressed by both simulations and actual experiments using the reference objects introduced in Section 3.

Simulations were done on fibers with a diameter equal to 18 µm and with different voxel sizes from 1 to 12 µm (see examples in Figure 2). The diagram in Figure 3a shows that the measurement error increases with the voxel size, following a non-linear relationship. Experimental results reported in Figure 3a show that in the case of voxel size of 4 µm and 6 µm the percentage errors correspond well with those obtained from simulations, but becomes larger for larger voxel size, probably due to the loss of contrast in actual CT reconstructions when the voxel size becomes too large with respect to the fiber diameter. The experimental percentage errors were used to correct the measurement of the fiber volume fraction (FVF), as it was done in the case reported in Figure 3b for a voxel size of 9 µm. It can be observed that the uncorrected value is significantly overestimated with respect to the nominal FVF.

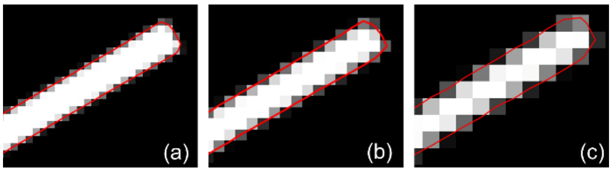

Figure 2. Zoomed view of a simulated fiber with a diameter of 18 µm and voxel size equal to 4 µm (a), 6 µm (a), and 9 µm (a). The red line represents the determined fiber surface.

4. Prediction of ultimate tensile strength

At failure, the ultimate tensile testing of FRP specimens can be predicted from the model proposed by Kelly and Tyson [9], which depends on the fiber orientation with respect to the loading direction, fiber length, and fiber volume fraction. The CT fiber characterization results – whose accuracy has been improved based on the comparison with reference fiber measurements as explained in Section 3 – were used to feed the Kelly-Tyson model [9]. The predicted UTS was then compared with the experimental UTS obtained from tensile testing. Figure 4 shows how the correction of systematic errors (bias) of CT measurements (performed with a voxel size of 9 µm) improved the UTS prediction: the deviation with respect to the experimental UTS decreased from 23 MPa to 6.6 MPa.

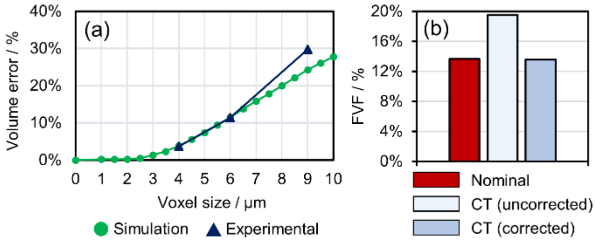

Figure 3. CT fiber volume measurement errors obtained from simulations and actual scans (a); comparison of nominal FVF with uncorrected and corrected FVF assessed with CT (voxel size = 9 µm).

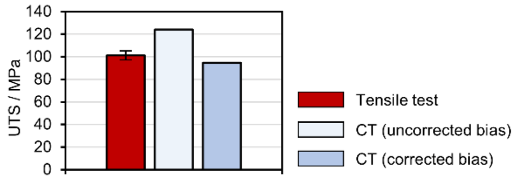

Figure 4. UTS obtained from tensile testing (the error bar represents the standard deviation of five tests) compared to the CT predicted UTS, in case of uncorrected and corrected bias.

5. Conclusions

This work was focused on optimizing CT fiber characterization to accurately predict the mechanical properties of FRP components. The proposed optimization procedure includes the use of reference objects, similar to actual industrial FRP components and used to assess and correct systematic measurement errors. An industrial case of IM glass-fiber-reinforced tensile specimens was addressed. The effect of different voxel sizes was also analyzed, showing that even with the larger voxel size of 9 µm (i.e. half the fibers diameter), the CT fiber characterization improved with the proposed procedure can be successfully used to predict the UTS of the investigated components, with achieved deviation below 7% with respect to the experimental UTS.

Future works are planned to extend the approach on other case studies and to determine the uncertainty of CT measurements as well as the uncertainty of CT predictions of mechanical properties.

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