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Accurate, repeatable, and efficient fiber-length measurements

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Conventional fiber-length measurement techniques for discontinuous fiber-reinforced composites are evaluated and a novel characterization methodology is proposed.

Discontinuous fiber-reinforced polymers have become important materials in the transportation industry because of their favorable material properties, low manufacturing costs, and lightweight characteristics.[1, 2] The configuration of the reinforcing fibers, however, changes significantly over the course of the entire polymer production process and these changes are reflected in numerous ways (e.g., favored fiber orientation, fiber jamming, and separation of fibers from the matrix).[3–6] Indeed, one of the major challenges in processing discontinuous fiber-reinforced thermoplastics is the process-induced reduction of fiber length, often referred to as fiber attrition or fiber breakage. For example, during injection molding, the embedded fibers are subjected to extensive stresses from the plasticating phase and injection stages, which inevitably reduces their length.[7, 8] It is therefore important to quantify the residual fiber-length distribution so that the fiber breakage phenomenon can be studied experimentally. The characterization of fiber-length distribution is a cumbersome task, however, because even small samples contain millions of fibers. Moreover, no standardized measurement protocol has yet been established.[9]

The majority of the currently applied measurement procedures (see Table 1) for assessing the fiber-length distribution of fiber-reinforced thermoplastics have four main steps in common. In the first step—matrix removal—the matrix is removed via pyrolysis (after the sample has been extracted from the molded part). Next—down-sampling and fiber dispersion—a subset of the fibers is selected and prepared for scanning. In the third step—digital imaging—either a microscope or an optical document scanner is used to create a digital image. Finally—during fiber detection—image processing software is used to analyze the digital image and determine the length of the individual fibers. During this step, the fiber detection is achieved either manually (by clicking on the endpoints of the fibers) or with the support of (semi-) automated image-processing algorithms. Despite the similarities between the many measurement techniques, there are also large variations in important aspects of the procedures (see Table 1).

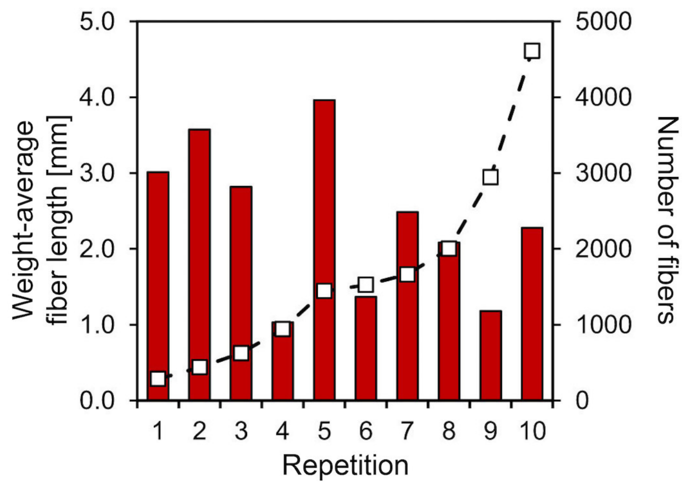

Figure 1. *Results from manual fiber-length measurements of 10 identical 40wt% glass, injection-molded long-glass fiber-reinforced polypropylene (PPGF40) samples. The weight-average fiber length (red bars) and number of measured fibers (white squares) are given for each measurement.*

To test the repeatability of conventional manual fiber-length measurement procedures, we analyzed[9] 10 identical injection-molded samples of long-glass fiber-reinforced polypropylene, with a glass concentration of 30 or 40wt% (PPGF30 and PPGF40, respectively). Following the published measurement protocols, we extracted samples from the molded part, removed the matrix via pyrolysis, and collected a random set of fibers for the fiber-length analyses. The results from a conventional measurement protocol are illustrated in Figure 1. These results show that there is substantial variation in the measured fiber-length distributions, i.e., varying from 1.03 to 3.96mm (0.95mm standard

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Plastics Research Online
10.2417/spepro.006973 Page 2/4

Table 1. Overview of fiber-length measurement techniques used in recent studies. PP: Polypropylene. GF: Glass fiber. PA: Polyamide. SFT: Short-fiber-reinforced thermoplastic. NF: Natural fibers. CF: Carbon fibers.

Material	Correction for downsampling	Fiber dispersion	Fiber detection	Fibers per sample	Study
PP/GF	No	Manual dispersion	Manual	100	Bajracharya et al.[10]
PP&PA12/GF	No	Manual dispersion	Manual	300	Bumm et al.[11]
PP/GF	No	Manual dispersion	Manual	500	Priebe et al.[1]
PP/GF	No	Manual dispersion	Manual	500	Wang et al.[12]
PP/GF	No	Manual dispersion	Manual	500	Thomason[13]
PP/GF	No	Manual dispersion	Manual	500	Jin et al.[14]
PP/GF&CF	Yes	Manual dispersion	Manual	2,000	Nguyen et al.[15]
PP/GF&CF	Yes	Manual dispersion	Manual	2,000	Kunc et al.[16]
PA6/GF	No	Diluted suspension	Manual	400	Inceoglu et al.[17]
PA6/GF	No	Diluted suspension	Manual	400	Yilmazer et al.[18]
PA6/GF	No	Diluted suspension	Manual	1,000	Lafrance et al.[19]
PP/GF	No	Diluted suspension	Manual	1,000	Inoue et al.[20]
GF	No	Manual dispersion	Semi-automatic	1,000	Huq et al.[21]
PP/GF	No	Diluted suspension	Semi-automatic	800	Zhuang et al.[22]
PP/GF	No	Diluted suspension	Semi-automatic	2,000	Teixeria et al.[23]
PP/GF	No	Diluted suspension	Semi-automatic	2,000	Ren et al.[24]
PP/GF	Not needed	Diluted suspension	Semi-automatic	2,000	Rohde et al.[8]
PP/GF	Not needed	Diluted suspension	Semi-automatic	3,000	Hartwich et al.[25]
PP/GF	No	Manual dispersion	Automatic	1,000	Giusti et al.[26]
SFT	No	Diluted suspension	Manual	360 ± 60	ISO[27]
GF/CF/NF	Yes	Turbulent air	Automatic	>15,000	Goris et al.[9]

deviation). In addition, the number of analyzed fibers can vary by more than an order of magnitude, and it is unclear whether a set of 1000 (or fewer) fibers can be statistically representative. Our results thus emphasize the difficulty of characterizing (in terms of comparability, repeatability, and accuracy) fiber length samples, and highlight the need for a standard measurement protocol. To that end, we have developed a novel technique that can be used to provide highly reproducible measurements in a timely manner.

In the first stage of our measurement technique (illustrated in Figure 2), we conduct pyrolysis to remove the sample matrix. We then perform a representative downsampling step, adapted from previous work.[15,16] All fibers in the defined region are collected by injecting a UV-curable epoxy resin through the thickness of the fiber bed, by using a fine hypodermic needle. This downsampling step allows the collection of a controlled and representative subset of fibers that statistically resemble the fiber-length distribution of the initial sample. After the epoxy plug has cured, we use a brush to carefully remove all the surrounding fibers and we conduct a second pyrolysis step to recover all the fibers that are embedded within the epoxy. In the next step, we use bursts of slightly compressed air to disperse the fibers within an enclosed chamber system.[9] In this way, the air turbulence causes the fibers to disentangle and disperse uniformly. Finally, after we have obtained a digital image of the dispersed fibers, we use an image processing algorithm (developed in-house) to automatically detect all the fibers (including bent and intersecting fibers) in the image. To ensure statistically representative results, we analyze at least 15,000 fibers for each sample and process the data set statistically (i.e., we calculate the number-average and weight-average fiber length).

As part of our study, we also compared our novel measurement technique with a manual measurement, the commercially available (from Karg Industrietechnik, Germany) 'FASEP' measurement system,[25] and a full analysis (provided by SABIC, the Netherlands). During the full analysis, the entire fiber population in the sample was measured, i.e., there was no downsampling step and up to 750,000 fibers were assessed in a single measurement. The results of our comparison, for the PPGF40 and PPGF30 samples are summarized in Figure 3.

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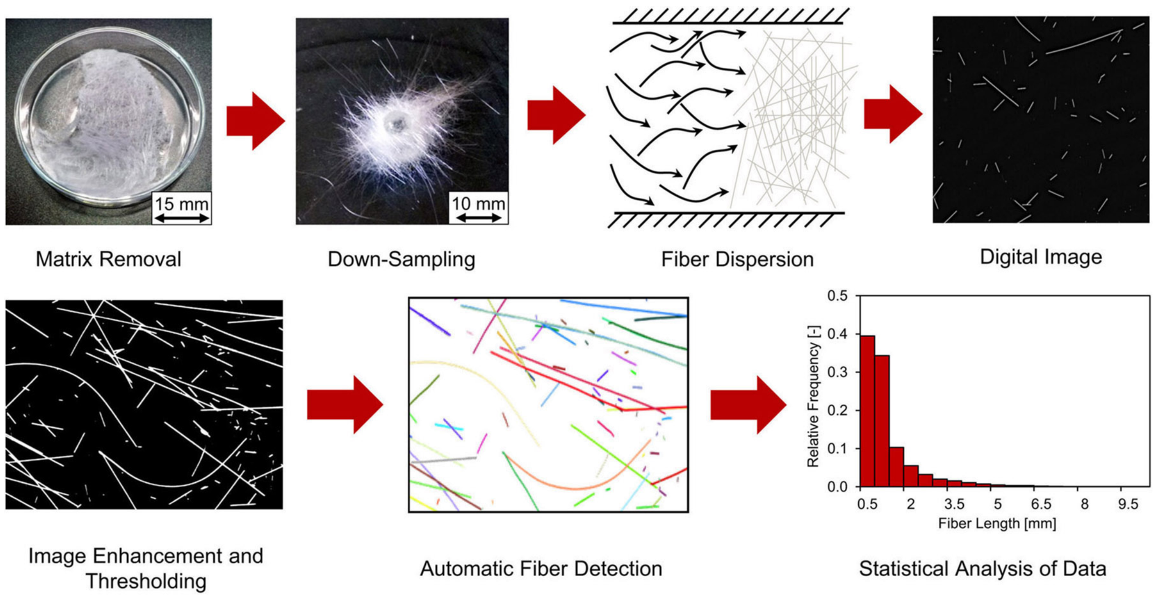

Figure 2. *Illustration of the steps in the proposed fiber-length measurement technique.*

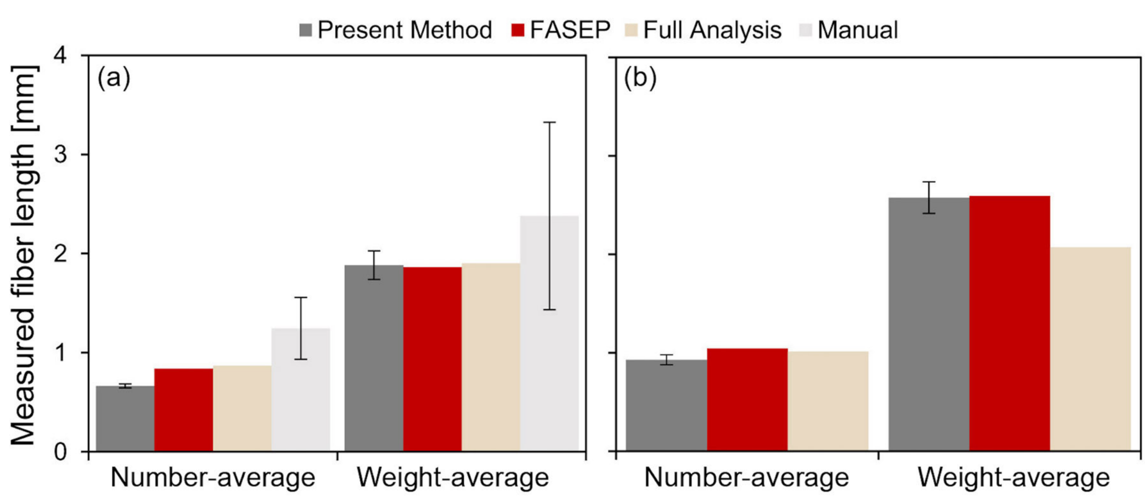

Figure 3. *Comparison of the number-average and weight-average fiber-length measurements obtained from the new method proposed in this work, the commercial 'FASEP' approach, the full analysis procedure (from SABIC), and a manual technique for the (a) PPGF40 and (b) PPGF30 (i.e., long-glass fiber-reinforced polypropylene with 30wt% glass concentration) samples. Error bars indicate standard deviations.*

In particular, from our manual measurement we obtained a value that was 27% higher than our weight-average fiber length measurement, and which had a substantial standard deviation. In general, however, we observe a strong agreement between the results of our proposed method, FASEP, and the full analysis procedures.

In summary, we have highlighted the important need for a standard fiber-length measurement technique for discontinuous fiber-reinforced composites. We have thus developed a new technique with which we can obtain accurate and highly reproducible fiber-length measurements in an efficient manner. In the next steps of our work, we will apply the developed measurement technique as part of a fundamental study on fiber attrition during long fiber thermoplastic processing. Our aim is to

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thus obtain a fundamental understanding of the underlying physics of fiber breakage.

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