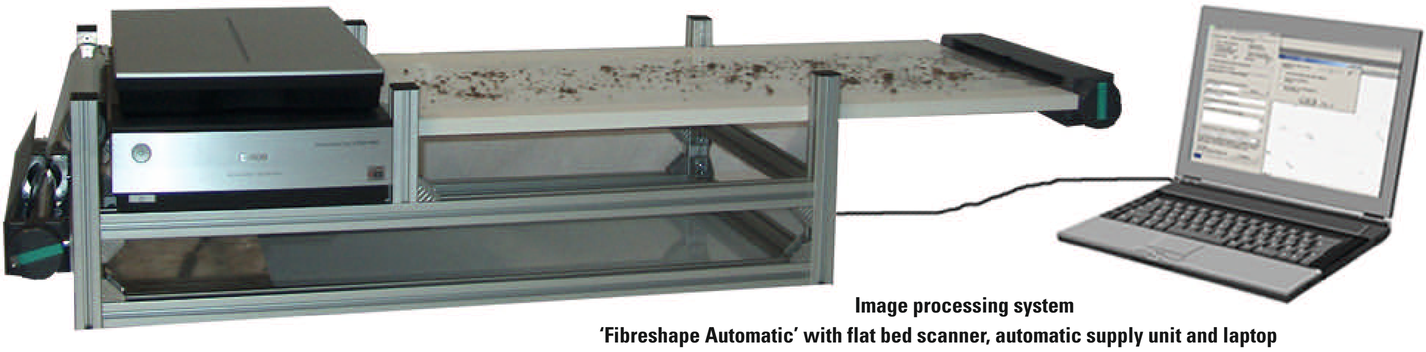

Image processing system 'Fibreshape Automatic' with flat bed scanner, automatic supply unit and laptop for control and data processing (figures: Faserinstitut Bremen)

Quality Control for Recycled Carbon Fibers

Testing. The market for carbon-fiber reinforced plastics has been growing for many years. Consequently the recycling of production wastes and end-of-life parts has been gaining more and more importance. The aim is to re-use the fibers, which are potentially high-value materials. Based on optical and mechanical tests, this article explains the quality of the materials offered and which methods of analysis are suitable.

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The use of carbon fiber reinforced plastics (CFRP) in high-end sports equipment like e.g. bicycle frames or surfboards etc. [1] is well-known, but the main market shares are in aircraft and wind energy industries [2]. Furthermore there is an increasing use in the automotive industry. A huge future potential is seen especially in the sector of electric mobility. Even now, from these industries there are emerging quantities of offcuts from textile semi-finished products and end-of-life wastes (i.e. returned CFRP parts) [2].

State of the art is the so-called 'thermal utilization' of these wastes (i.e. incineration), but this is not a reasonable pathway having in mind the energy-intensive production of carbon fibers. The better alternatives are recycled materials like those which are already offered commercially. These are mainly roving snippets in defined length or milled material, i.e. short carbon fibers for injection molding. The material is either directly gained from offcuts or rest bobbins, or from composite parts by pyrolysis [3]. According to the material's origin, the mechanical quality of these recyclates corresponds either with new carbon fibers, or they can be strongly degraded by the process of pyrolysis. The material's morphology can vary from even to strongly inhomogeneous.

The use of recycled carbon fibers in lightweight construction or similar high-performance applications contributes to saving global resources. In addition, the price advantage of the recycled fibers enables their use in new applications, which were not relevant yet due to economical reasons. The possible technologies for re-utilization of recycled carbon fibers have been examined within the framework of a research project. Main topics were the production of textile fleeces from recycled carbon fibers with new characteristics, the processing of recycled long carbon fibers to organic sheets, and the development of processes and technologies to produce CFRP from the new semi-finished materials. An essential prerequisite was the development of suitable methods for examining the recyclate materials to enable the analysis of quality and homogeneity of the materials. Based on the developed methods, it was also possible to get an overview about the real qualities available on the market.

Analysis of the Recyclate Materials

The characterization of the different classes of recyclate materials is exemplarily shown based on selected samples. This comprises roving snippets in different lengths, medium-long filaments for fu-

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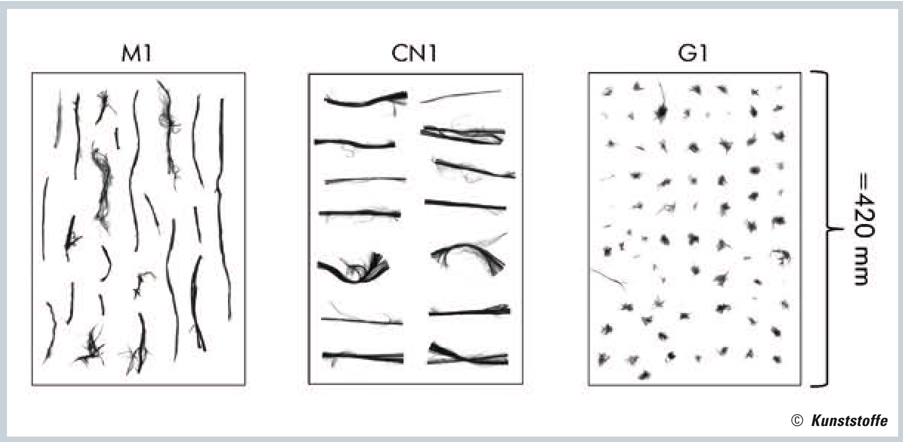

Fig. 1. Prepared samples for image processing of M1, CN1, and G1

ture use in long-fiber applications and finally short filaments for injection molding. New rovings cut in defined lengths were obtained from commercial suppliers as reference material. The samples are listed in **Table 1** combined with their specifications.

All listed materials were analyzed for their length distribution. Filaments (also from roving samples) with sufficient length were analyzed for their tensile properties, too. Recyclate samples were additionally analyzed by scanning electron microscopy (SEM) in order to identify the presence of deposits on the surface and surface damages.

The geometrical properties of the samples were analyzed by Fibreshape [4] **(Title figure)**. It was necessary to discriminate different types of samples and to analyze them with adapted parameter sets: (1) rovings with length up to 300 mm and different widths as well as medium short filaments (approx. 5–40 mm), and (2) short filaments <5 mm for injection molding. To prevent the laboratory equipment from conductive carbon fiber dust, the samples of class 1 were laminated into DIN A3 foils prior to the measurement. They were analyzed at 100 dpi in the flat bed scanner, using the actual version Fibreshape V5. Samples of class 2 were prepared in glass slide-frames and analyzed in a slide scanner at 4,800 dpi, using Fibreshape V4.2. Characteristic examples for different sample types are shown in **Figure 1**: roving snippets (M1 and CN1) and filaments (G1).

Designation	Type	Length [mm]
CN1	roving[1]	100
G1	filament	6–60
M1	roving	n/a
S	roving[2]	50
P	roving[2]	100
C1	filament	0.15
C2	filament	0.5
C3	filament	6

Table 1. Selected recyclate and reference materials and nominal length ([1] without fiber finish, [2] virgin material (reference)

The mechanical characterization was conducted on single filaments based on DIN EN ISO 5079 (1996), using a Dia-Stron System with clamping length 3.2 mm. Preceding the tensile tests the cross-section of each single specimen was measured via laser beam. At least 45 specimens were measured to ensure statistically firm results [5].

Quality and Homogeneity

The analyzed area-based lengths of the roving snippets (median) are slightly above the nominal values **(Table 1)**. Reasons are, that either the rovings are partly deformed parallelogram-like, or that single rovings protrude the cut edge. For all samples a very small minimum length is detected, caused by short filament fragments. The area share of these short fragments is small enough to have only minimal influence on the median length.

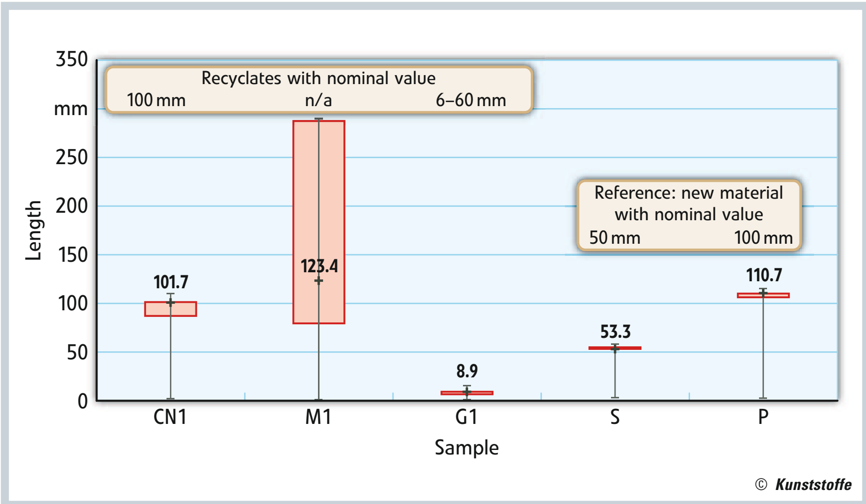

Fig. 2. Length distribution of the roving snippets with median values (n/a: not available)

The samples exhibit significant differences **(Fig. 2)**: S and P as reference samples are very homogeneous in their length distribution. The very small boxes comprise 50 % of the measured data. The distribution width of CN1 is comparable. The outlier is M1 with its very inhomogeneous length distribution: roving snippets ranging from 70–300 mm will be a problem for subsequent processing. On the lower end of the spectrum is the filament sample G1 with short length <10 mm, which is too short for long fiber processing and too long for injection molding. For this sample type flocks of filaments were prepared on the laminating foil, and the maximum diameter was analyzed as length. This implies that the measured median of 8.9 mm is still too high. To get an exact length distribution for this sample type, it would be necessary to manually separate the single filaments for the measurement. However, →

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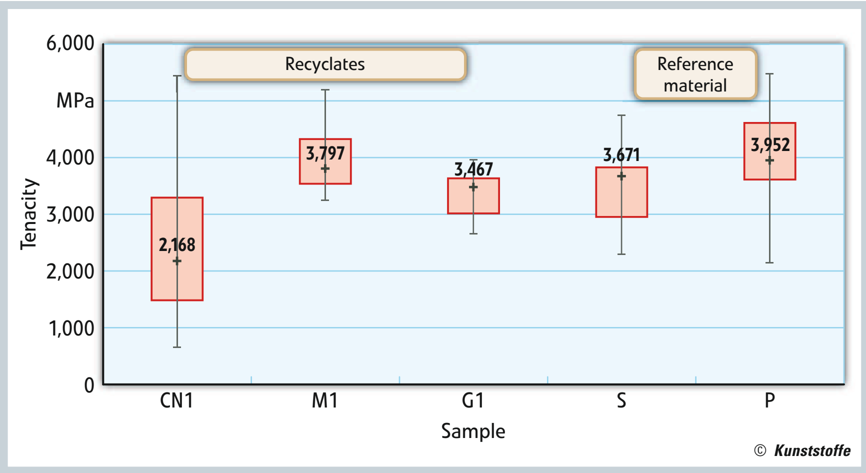

Fig. 3. Tenacity distribution of the roving snippets with median values

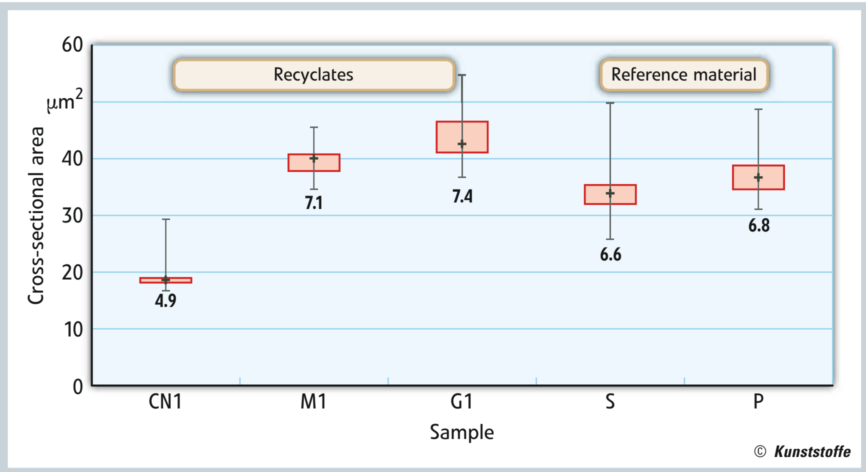

Fig. 4. Cross-sectional area distribution indicating the mean diameter (mean diameter in μm calculated from the median cross-section area via the circle formula)

this work was not performed, since this batch is too short for long-fiber processing and still too long for injection molding.

The results of the tensile tests on single elements are displayed in **Figure 3.** Except CN1 all samples are in the range of 3,500–4,000 MPa, but with different deviations. Only CN1 is significantly lower: 2,168 MPa. The difference is that this sample exhibits significantly finer filaments **(Fig. 4)**: the median of its cross-sectional area corresponds to 4.9 μm diameter. M1 and G1 are slightly above 7 μm, whereas S and P are slightly below 7 μm.

The results of SEM analysis confirm the findings discussed above. On most of the pyrolyzed samples surface deposits (pyrolysis residues) were detected. Two typical findings are displayed in **Figure 5:** (a) larger matrix residues in sample M1 and (b) surface damages (crater formation) in sample CN1. This elucidates the low tenacity and strong inhomogeneity of CN1.

All short filament samples for injection molding (C1 to C3) are too short for tensile testing, but length measurement by Fibreshape is possible. The results are displayed in **Figure 6.** Obviously lots C1 and C2 are not significantly different. This is in contradiction to the nominal declaration **(Table 1)**: C2 (0.5 mm) should be approximately three times longer than C1 (0.15 mm). In reality, both of them are approx. 0.17 mm. The applied process of milling seems to be unspecific. The third sample C3 deviates 20 % from the nominal value (6 mm): the measured median is approx. 4.8 mm. Overall only one of three short filament samples complies with the supplier's declaration.

Summary

The characterization of recycled carbon fibers is possible using a new developed method of image processing. Due to the extremely different materials with lengths ranging from >100 mm (rovings) down to short filaments <0.1 mm, it is necessary to apply different resolutions and evaluation parameters. This enables the exact analysis of the different material classes. In principle, the filament fineness could also be analyzed by image analysis, if a microscope is used. This method is not validated yet.

In combination with results of single-element tensile test a comprehensive description of the recyclates is possible, which enables their use in high-value applications. For roving snippets the homogeneity of the cut length is checked by image analysis. In this way, samples with inhomogeneous length distribution like M1 are identified clearly. The quantities of offcuts or filament fragments can be detected simultaneously. If necessary, this can be used as criterion for exclusion.

For all rovings and long filaments (cut or endless) the tensile test delivers valuable information about the material's quality. Damage caused by the process of pyrolysis can be identified easily as well as erroneous declarations.

If one of the methods mentioned above delivers hints on fiber damages, a control by SEM analysis is recommended. SEM supplies information, if the process of pyrolysis is either incomplete (presence of matrix residues) or has damaged the fibers (crater formation/surface erosion). The presence or absence of fiber finish can be detected as well.

The results presented here depict clearly the necessity of a comprehensive char-

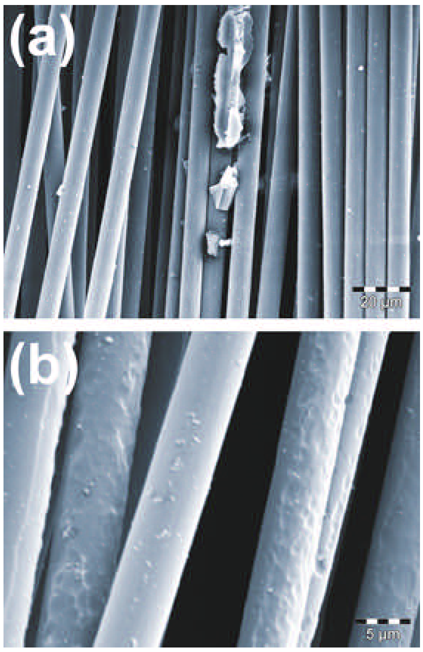

Fig. 5. (a) Matrix residues on sample M1 and (b) crater formation on sample CN1 as typical examples for irregularities

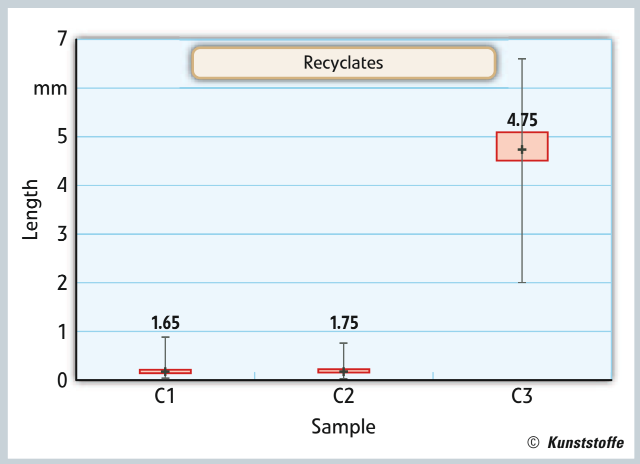

Fig. 6. Length distribution of the short filaments with median values

acterization. If roving snippets are used, strong inhomogeneity in length, or contaminations are easy to detect visually. Smaller length variances can only be detected and reproducibly quantified by image analysis. This conclusion is valid as well for the tensile test, which offers exact information about the mechanical material qualities. Furthermore, the results of short filament characterization show that a quality declaration of the supplier usually exists. However, in reality the materials can be strongly different from these nominal values. Differences up to a factor 3 were found between nominal and real lengths. ■

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