Application Report

Single Fiber Wettability

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Method:

Single Fiber Tensiometer – K100SF

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Wettability of carbon fibers using single-fiber contact angle measurements – a feasibility study

Abstract

During the production of carbon-fiber-reinforced plastics the fibers are embedded in a plastic matrix. With respect to the necessary fiber wettability, the quality assurance of this process is not only a classic task for single-fiber contact angle measurement, but also a challenge; in comparison to hairs or textile fibers the considerably smaller fiber diameter places high demands on the precision of the instrument.

In this application note we present the development of a feasibility study on single-fiber contact angle measurement. The reproducibility and significance of the results were checked on three different pretreated fibers using a Single-Fiber Tensiometer K100SF from KRÜSS.

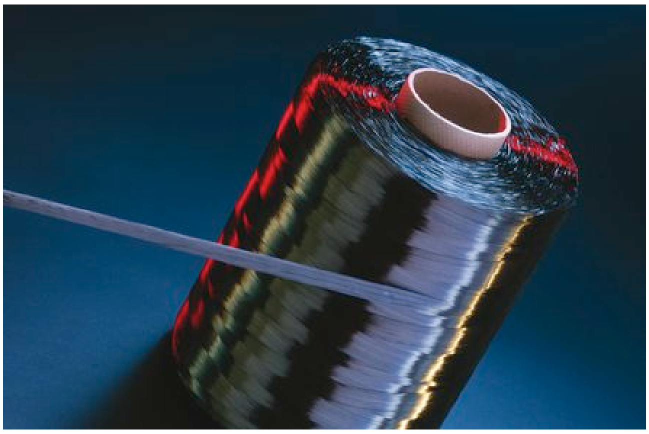

Fig. 1: Bundle of 50000 carbon fibers (Image: SGL Group)

Background

Carbon fibers are high-strength, light and conductive materials and are therefore used in composites for matching the highest demands, e.g. in aircraft construction or for sports apparatus. The embedding of the fibers in the plastic matrix – epoxy resins are frequently used – requires appropriate pretreatment. In the manufacturing process the fiber passes through a sizing bath, where it is given a thin plastic coating. Normally the sizing used is of the same class of substances as the plastic matrix – epoxide, for example, if this forms the intended matrix.

If the sizing coating is incomplete, or non-compatible with the plastic matrix, then wetting problems could occur during embedding in the matrix. Possible consequences could be loss of quality resulting from air bubbles or inadequate adhesion, for example.

The measure for the wettability of a solid with a liquid is the contact angle between the two phases: the larger the contact angle, the smaller the wetting.

With the aid of the single-fiber method it is possible to make contact angle measurements on single fibers just as they leave the manufacturing process – i.e. before they are further processed to form fiber bundles or braids.

The aim of this project was to check the feasibility of making wetting measurements with water on single carbon fibers. With regard to the detection limits of a single-fiber tensiometer, such measurements are a challenge:

1. The diameter of a carbon fiber is only about one tenth of that of a human hair. This means that the wetting forces to be measured are considerably smaller.

2. The wetting force becomes smaller when the contact angle approaches a value of 90°. Because of the hydrophobic coating of the carbon fibers, such large water contact angles and correspondingly small wetting forces are to be expected.

Against the background of these extreme conditions the reproducibility and significance of carbon fiber measurements were checked with a Single Fiber Tensiometer K100SF.

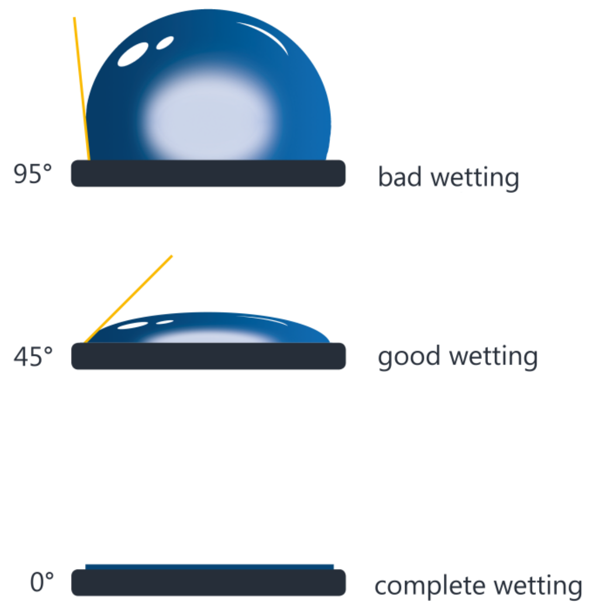

Fig. 2: Relationship between contact angle and wetting

Experimental part

Samples

Samples of three differently coated carbon fibers were studied. The given fiber diameter was 7 µm, the wetted length relevant for the measurement (circumference of the circular fiber profile) was 22.0 µm.

The exact constitution of the coating substances was not known. For this reason the verification of the hydrophobic character of the samples, which was to be expected from the coatings, was in the foreground. It was also to be shown whether any significant differences between the samples could be detected.

Test liquid

Distilled water was used as the test liquid for the wetting measurements. By always using the same test liquid it is possible to compare the wettability under uniform conditions. Although water is not the medium intended for further fiber treatment, conclusions about the wettability by hydrophobic media can be drawn from wetting with water: a poor water wettability argues for good wettability by a hydrophobic target matrix.

The measuring temperature was 22°C.

Instrument and method

The tensiometer used (Type K100SF) has a force sensor resolution of 1 µg for measuring the very small wetting forces involved.

During a contact angle measurement with this instrument the fiber is positioned vertically in a holder suspended from the force sensor. A vessel containing the test liquid is moved upwards towards the fiber from below. The contact between the fiber and the surface is detected automatically by a sudden jump in the measured force sensor value. During the further movement of the sample vessel the wetting force is measured as a function of the immersion depth.

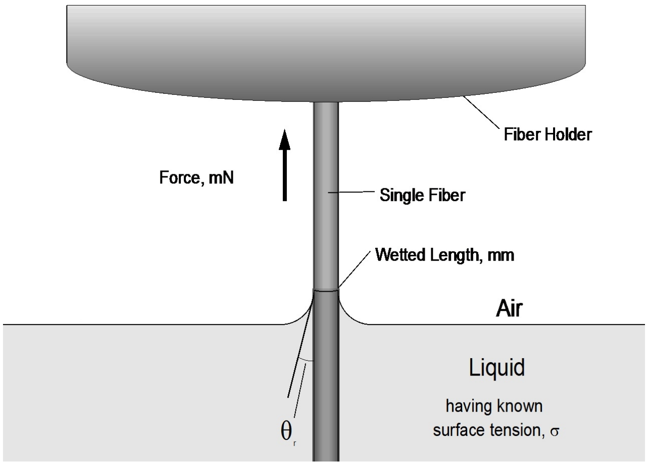

Fig 3: Schematic diagram showing a single-fiber contact angle measurement

For larger numbers of samples, sample holders containing fibers can be inserted automatically in the instrument and removed again after the measurement by using a handling system.

In order to calculate the contact angle θ this wetting force in the region of the linear dependency on the immersion depth is extrapolated for immersion depth 0.

The contact angle is measured on the upward and downward movement of the sample table, i.e. by wetting and dewetting the fiber. The measurement accordingly gives two values for the contact angle: the advancing angle from the wetting phase and the retreating angle from the dewetting phase. Because of the prewetting, the retreating angle can sometimes differ considerably from the advancing angle. As in this case the wettability of the fiber is in the foreground, only the advancing angle was interpreted.

Because the measurement was made in the lower sensitivity range of the instrument, a check was made as to how much the precision could be increased by improving the setup conditions. Setting up under a wind-shield PA0910 could considerably reduce the influence of interfering airflows, probably caused by a ventilation system. Because of the low-vibration environment, the use of the antivibration platform PA0911 did not bring any significant improvement.

Test procedure and results

During the investigations it was found that the measured contact angle depended on the speed of movement during fiber wetting. The following illustration shows a comparison between the curves at different measuring speeds, using sample C as an example.

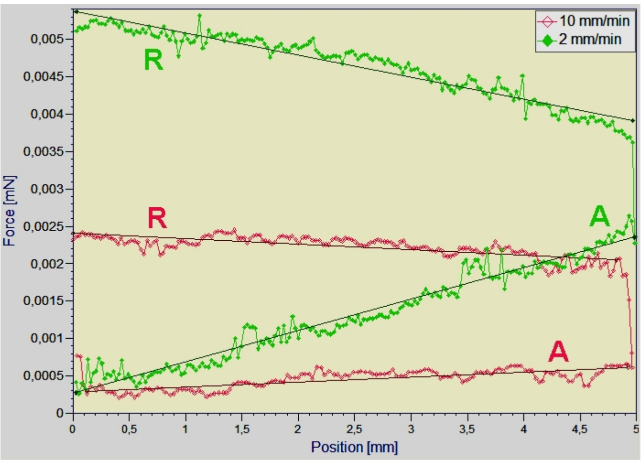

Fig. 4: Contact angle measurement at different measuring speeds (A = advancing angle, R = receding angle)

Such a finding normally indicates an alteration to the surface by contact with the liquid, e.g. by adsorption. In this case the alteration was reversible, as after a waiting period the original behavior of the fiber was again evident.

In order to measure the contact angle independently from the immersion time, the immersion speed was increased until a time-based influence could no longer be detected. The immersion speed determined in this way was 10 mm/min (compare with lower curve in Fig. 4).

The following table contains the results for samples A, B and C as mean values with a standard deviation from at least 14 measurements.

Sample	A	B	C
Contact angle	71.9±3.7°	83.1±2.7°	78.5±1.8°

Table 1: Water contact angle and standard deviations for 3 single-fiber samples

All samples lay within the range of low wettability between 70° and 90°. When taking into account that the measurements were carried out in the lower sensitivity range of the instrument, the reproducibility can be regarded as good. The extent to which the variations result from the measurement itself or reflect inhomogeneities in the fiber coating cannot be evaluated here.

As the differences between the mean values are larger than the standard deviations, the results are significant and reflect actual differences between the wettabilities of the fibers.

The exact level of the contact angle is not a quality criterion for the fiber; the similarity to the intended target matrix is much more important. Additional contact angle measurements on the particular plastic matrix can provide information about compatibility.

Summary

Because of their small diameter and the correspondingly small wetting forces, carbon fibers present a challenge for single-fiber contact angle measurement. The aim of this project was therefore to check the feasibility of single-fiber contact angle measurements between carbon fibers and water. Such measurements are desirable, because quality assurance can then be carried out before further processing to bundles or braids.

Initially the dependency of the measured value on the speed of immersion was detected; this was interpreted as being due to the reversible adsorption of water. This influence could be eliminated by increasing the speed of measurement.

The mean values from at least 14 measurements show differences in the contact angle; these exceed the range of scatter and therefore indicate differences in wettability.

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