Application Report

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K100 & Tensíío Force Tensiometers

TO0810 sample holder

Method: 

Keywords: Carbon fiber, single fiber wetting, contact angle, Wilhelmy method

Measuring wettability of single carbon fibers using a tensiometer with a regular high-resolution force sensor

Strongly increasing the wetting force at ultrathin fibers with a multiple-fiber sample holder

In fiber composites, a lack of wettability of the fibers leads to air inclusions in the polymer matrix and consequently to a lack of strength as well as other quality losses. The contact angle and surface free energy of carbon fibers are therefore of high interest. However, analyzing a single carbon fiber with its tiny diameter of just a few microns requires an extremely sensitive instrument such as the KRÜSS K100SF, which is specialized for this purpose but requires demanding installation and ambient conditions, e.g. a vibration damping system.

To overcome this difficulty, we have developed a sample holder that allows the measurement of multiple single fibers in parallel, thus multiplying the wetting force. With this accessory, it is possible to conduct fiber measurements with a high-resolution Force Tensiometer such as Tensíío or K100 without having to resort to ultra-sensitive force sensors and the challenges associated with them. Previous comparative measurements between K100 with a five-fold fiber holder and K100SF with single fibers had proven the reliability of this new approach.

Background

Carbon fiber wettability

Carbon fibers (CFs) are widely used in industry to create fiber-reinforced composite materials with outstanding mechanical properties. To optimize wetting in the process of embedding the fibers, the surface tension (SFT) and surface free energy of the polymer matrix and of the CFs must be carefully matched. Thus, there is an interest in measuring the contact angle of differently modified CFs.

The wettability of CFs can be investigated at different scales: from macroscopic woven over nonwoven fabrics to fiber bundles down to the microscopic single fiber level. [1] The latter is often advantageous because the fibers are then examined at an early stage of the manufacturing process.

For single fiber measurements, the K100SF Force Tensiometer has been established. It comes with a highly sensitive microbalance, which is capable of precisely measuring the tiny wetting forces, typically in the range of some hundred nN, that act on individual carbon fibers. [2,3] However, measurements with this instrument require an extremely low-vibration environment, which cannot always be guaranteed. It is therefore a great relief if single fiber measurements can be examined with a conventional high-resolution force sensor.

Multi fiber sample holder

The wetting force is linked to the wetted length of the fiber, i.e. the circumference at surface level when the fiber is immersed. Using several fibers simultaneously with the sample holder TO0810 means that the wetted length and thus the wetting force is multiplied. This increased force offers the option to measure the contact angle at the single fiber stage using a regular high-resolution force sensor with Tensíío or K100.

Experimental section

The experiments shown here have been conducted in 2021 as part of a customer project using a K100 Force Tensiometer, the predecessor of our state-of-the-art Tensíío instrument. The Wilhelmy method [4] was used to measure the advancing contact angle during wetting and the receding contact angle during dewetting of the fiber sample at room temperature (23 ± 2 °C) using ADVANCE software. The carbon fibers had a diameter of 7 µm. Five single fibers were prepared and attached to the multi fiber sample holder (fig. 1).

Fig. 1: Loaded sample holder TO0810

DI water (SFT of 72.8 mN/m and a density of 1.0 g/cm^3) as well as diiodo methane (SFT of 50.8 mN/m and a density of 3.3 g/cm^3) were used as test liquids. The fibers were immersed (wetting process for advancing contact angle) to a maximum immersion depth of 4 mm and subsequently pulled out (dewetting for receding contact angle), all at a speed of 1 mm/min. Force data readings were taken with 0.01 mm steps.

Comparative measurements of the very same single fibers were taken with the K100SF as described in previous application reports. [2,3].

Results

K100 with a five-fold sample holder

The contact angle vs. immersion depth for the multi fiber sample holder is plotted in fig. 2. The resulting contact angle data are summarized in table 1.

Table 1: Results of multiple fiber measurements with K100

	Advancing CA [°]	Receding CA [°]
Water	75.2 ± 4.2	39.2 ± 8.5
Diiodo methane	47.7 ± 6.5	3.6 ± 8.2

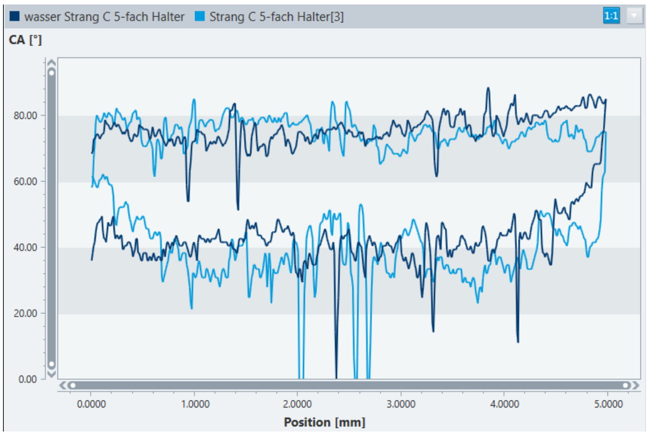

Fig. 2: Water contact angle vs. immersion depth as determined for five single fibers in parallel (double determination)

K100SF repeat measurements with single fibers

The results for the single fiber measurements performed with the K100SF are summarized in Table 2. The results are mean values and standard deviations for a total of $n = 5$ individual fibers. Exemplary graphs of contact angle vs. immersion depth are shown in fig. 3.

Table 2: Results of single fiber measurements with K100SF

	Advancing CA [°]	Receding CA [°]
Water	78.8 ± 4.6	40.4 ± 9.3
Diiodo methane	49.5 ± 4.3	11.9 ± 7.9

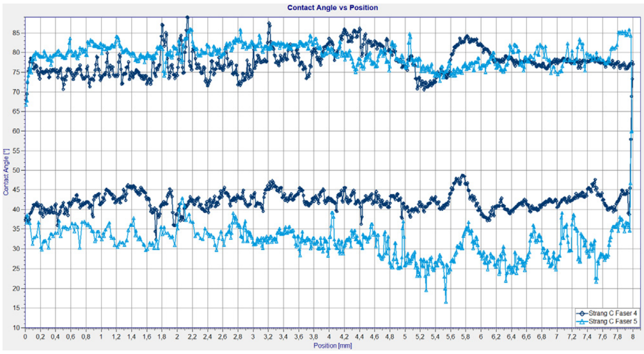

Fig. 3: Contact angle vs. immersion depth of single fibers in water measured with the K100SF. For the sake of clarity, results for only two fibers are shown.

The advancing contact angle for water and diiodo methane are in good agreement for both methods/instruments used. The standard deviation for the advancing angle is also identical. In both cases, the receding angles show significantly larger error bars, especially for diiodomethane. This is typical for ultrathin fibers due to the even lower forces acting, among other reasons.

The advancing angle, however, is far more relevant for fiber embedding since this also represents a wetting rather than a dewetting process. Here, the good agreement between the two methods/instruments indicates that the K100/Tensíio with the multi fiber sample holder is a good alternative for the K100SF approach without having to compromise on the precision of results.

Summary

We present how a multi-fiber sample holder as an accessory for a Tensíio or K100 Force Tensiometer allows to measure fibers of few µm in diameter at the individual fiber level. By measuring several fibers in parallel, the wetted length and thus wetting forces are multiplied so that the high-resolution force sensor of a Tensíio/K100 tensiometer proves sensitive enough for precise contact angle determination at a signal-to-noise ratio comparable to a K100SF. This extends the sample diameters accessible for measurement for the KRÜSS premium tensiometers and makes them even more versatile instruments for wetting analysis.

Literature

[1] KRÜSS Application Report AR228

[2] KRÜSS AR271

[3] KRÜSS AR284

[4] https://www.kruss-scientific.com/en/know-how/glossary/wilhelmy-plate-method (accessed 2025.03.04)

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