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Determining the wettability of carbon fiber tows from single fiber contact angle data

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Physical adhesion between carbon fibers (CFs) and polymer matrices as well as the formation of voids at the interface between these two materials are mostly determined by the wetting properties of the fibers. Due to the hierarchical structure of CF reinforcements, it is essential to study their wetting behavior at different scales: from the single fiber (microscale) to the fabric (macroscale) via the tow scale (mesoscale). Whereas a direct measurement of the contact angle of single CFs by tensiometric means is well established, a direct measurement of the wettability of CF tows is hampered by their porous structure due to densification and liquid take up phenomena. An accurate characterization of their wettability is therefore still highly challenging.

In this Application Report we present a method for determining the wettability of CF tows based on contact angles measured on single fibers. For this we performed a combined and synchronized analysis of tensiometrically and optically determined contact angles. The wettability of single CFs and CF tows composed of unsized and sized CFs were measured using a Force Tensiometer – K100SF and optical means. The contact angles of CFs at meso- and microscales have then been successfully linked using a modified Cassie-Baxter model.

Experimental section

Materials

The CF tows considered here are composed of two types of untwisted polyacrylonitrile-based CF tows. These two materials are laboratory made unsized and untreated CF tows provided by Deakin University and commercially available sized CF tows (see Figure 1) named FT300- 3000-40A (T300) purchased from Toray CFs Europe S.A, respectively. Distilled water was used as the test liquid for the contact angle measurements.

Method

The method proposed by Qiu et al. [1] for measuring static advancing contact angles was used to characterize the wettability of sized and unsized single CFs according to the Wilhelmy method. Each fiber was repeatedly dipped in and withdrawn from the liquid vessel three times at a velocity of 3.6 mm/min to measure a series of dynamic advancing and receding contact angles.

To measure static advancing contact angles at the CF tow scale, the samples were slowly soaked in the liquid over a length of 1 mm and stopped at that position for 500 s to make sure the external meniscus around the tow reached a static configuration. The vessel was then moved down until complete withdrawal from the liquid bath. The forces exerted on the tows were detected continuously every 200 ms by the Force Tensiometer — K100SF during the whole procedure (including approaching, wetting and withdrawing from the liquid bath). High-resolution pictures were simul-taneously taken to monitor the densification of the tow due to elasto-capillary forces. A sketch of the apparatus is shown in Figure 2.

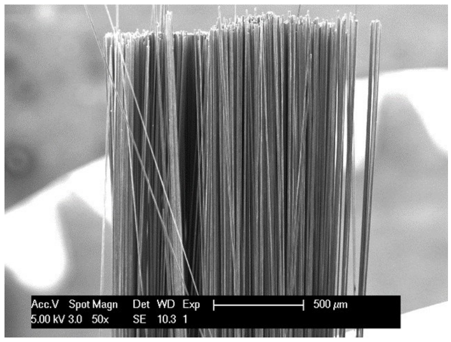

Fig. 1: SEM image of a T300 CF tow (containing 3000 CF filaments)

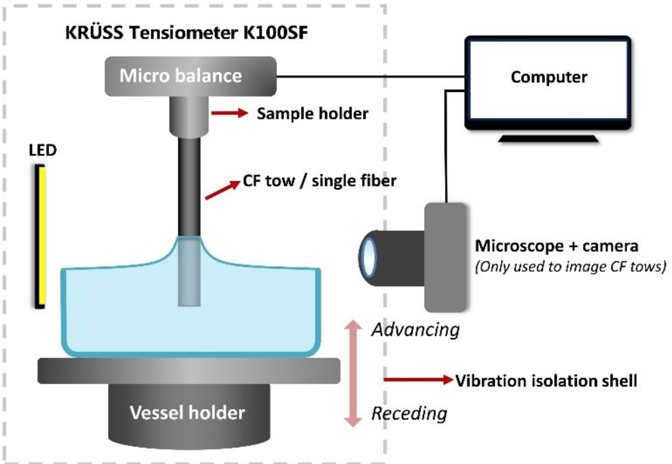

Fig. 2: Schematic representation of the experimental setup showing the combination of tensiometry and optical methodologies to characterize the wettability of CF tows. Figure as adapted from [2].

We developed a modified Cassie-Baxter model which relates the contact angle θ_s measured on the single fiber to the "average" external contact angle around a CF tow θ_{CB} by the following equation:

$$\cos\theta_{CB} = \frac{2\sin\theta_s}{\sqrt{\frac{\pi}{(1-P)\cos 30°}}}(\cos\theta_s - \cos\theta_i) + \cos\theta_i$$

θ_i can be assumed to be 0° because the tows are infiltrated by water so that the air between the CF filaments is replaced by water, leading to water/water "interfaces". P is the non-solid volume fraction of the wetted CF tows which can be calculated based on the mass, the density and the optically measured diameter of CF tow samples (which normally assume circular shape). Assuming that there are no remaining air pockets in the tow, P can also be equated to the volume fraction of fluid retention f_r. This can be calculated from the weight of the fluid taken up by capillary flow when weighing the wetted tow afterwards, using

$$f_r = \frac{V_r}{V_r + V_{fibers}} = \frac{\frac{W_r}{\rho}}{V_r + V_{fibers}}$$

where V_r is the volume of fluid in the tow, W_r the weight of the liquid retention, V_{fibers} the volume of CF fibers and ρ the density of the fluid. [2]

Results

Figure 3 shows a comparison of the advancing contact angles measured on the single fibers using the K100SF only, on the tows using the optical method and the calculated contact angle based on the measured single fiber data following the equation above for the unsized and sized CFs, respectively. As shown, the comparison between the results obtained from the measurements and theoretical predictions for both unsized and T300 CF tow shows good agreement indicating that this method successfully quantifies the effects of densification and water up-taking on the static advancing contact angles.

Conclusion

In this Application Report we describe a method to measure contact angles around CF tows. This method provided consistent results for single CFs and CF tows composed of unsized and sized CFs. This method confirms that contact angles at the meso- and microscales can be linked using the Cassie-Baxter model. With this it is now possible to deduce the contact angle of CF tows based on contact angles measured on single fibers.

A Force Tensiometer – K100SF in combination with a standard optical device was essential to correctly set up and proof our theoretical model. However, based on this study a K100SF instrument together with the simple theoretical model is now sufficient to describe the wettability of CF tows, thereby facilitating a better prediction of the adhesion between carbon fiber tow and polymer matrix.

References

[1] S. Qiu, C.A. Fuentes, D. Zhang, A.W. Van Vuure, D. Seveno, Wettability of a single carbon fiber, Langmuir. 32 (2016) 9697–9705.

[2] J. Wang, C.A. Fuentes, D. Zhang, X. Wang, A.W. Van Vuure, D. Seveno, Wettability of carbon fibres at micro- and mesoscales, Carbon N. Y. 120 (2017) 438–446.

Further information

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

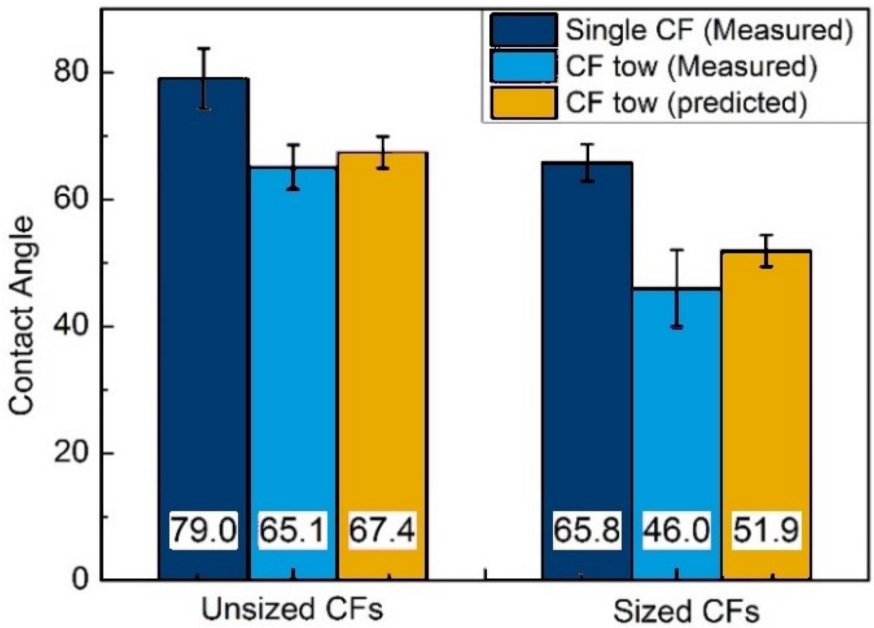

Fig. 3: Comparison of static advancing contact angles between unsized CF tows and T300 CF tows with water [1][2]. The single fibers' data were measured using the K100SF and the CF tow data were measured using the optical method. The predicted CF tow data were calculated from the single fiber K100SF results. Figure as adapted from [2].