FIMATEST – A New Testing System to Determine the Fibre-Matrix Adhesion Strength by Means of Pull-Out Tests

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ABSTRACT

Textechno, Germany, the well-known producer of testing instruments for man-made fibers and yarns, has a focus on the development of instruments for the characterization of the physical properties of reinforcement fibers, rovings and the fiber-matrix adhesion. In this article we discuss in detail the measurement of the fiber-matrix adhesion strength by means of a new testing system which has been developed together two research institutes, the Leibniz-Institut für Polymerforschung (IPF) in Dresden and the Faserinstitut Bremen (FIBRE). The system determines the adhesion between fiber and matrix in terms of the local interfacial shear strength, the interfacial toughness and further parameters through a reproducible pull-out test. It is suited for all kinds of fibers as well as all kind of thermoset and thermoplastic matrices with curing and melting temperatures up to 400°C.

KEYWORDS: Fibre-matrix adhesion, interfacial shear strength, testing

1 INTRODUCTION

The mechanical properties of a fibre reinforced material, e.g. toughness and strength, are significantly determined by the interfacial properties between reinforcement fibers and matrix material. This is because the bonding between the two components is usually the first one to fail in a stress induced breakage of a composite part. Optimization of the fiber-matrix adhesion strength hence plays a central role in the development of new fibers, their sizings as well as matrix materials where it must be well adjusted to lead to an enhanced composite performance (Ehrenstein, 2006). Beyond R&D purposes, the interfacial bonding is also a key quality parameter that needs to be monitored as production control.

2 MACROMECHANICAL VS. MICROMECHANICAL CHARACTERIZATION OF THE FIBER-MATRIX ADHESION

Macro- and micromechanical test methods have been employed to characterize the fiber-matrix adhesion. For macromechanical testing, most commonly composites with unidirectional aligned fibers are manufactured to perform either a tensile test perpendicular to the fiber orientation (transverse tensile test) or to measure the interlaminary shear strength (ILSS test). However, the maximum stress found in macroscopic tests does not only depend on the fiber-matrix adhesion alone, but is also altered by

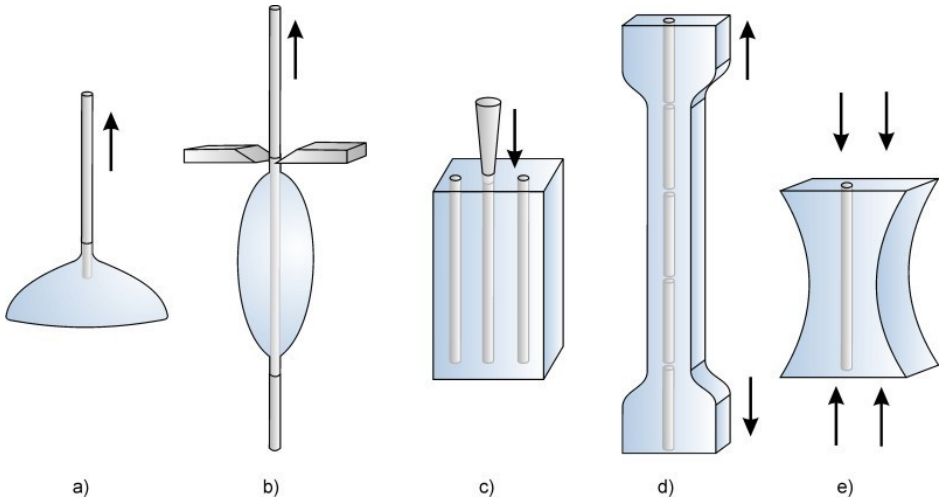

Figure 1: Overview of micromechanical tests methods for measuring the fiber-matrix adhesion: pull-out (a), microbond (b), push-out (c), fragmentation (d) and Broutman (e) (Mäder et al., 2016).

additional factors: the fiber content, orientation, length, diameter and fiber distribution homogeneity, the pore void of the test specimens, and the mechanical properties of the fiber and the matrix.

In the case of micromechanical testing techniques, a fiber-matrix compound is created using just a single reinforcement fiber. Compared to the macromechanical testing, the additional factors listed above that result from the composite manufacturing are excluded. Suitable analysis techniques allow to receive the adhesion strength alone as for instance demonstrated by Zhandarov et al. (2003) and Zhandarov and Mäder (2003) for the pull-out test.

3 THE MICROMECHANICAL SINGLE FIBER PULL-OUT TEST

The single fiber pull-out test is distinguished from other micromechanical tests in the advantageous fact that it is applicable to both soft and stiff fibers in combination with both ductile and brittle matrices regardless of if they are thermoplastic or thermoset (Zhandarov and Mäder, 2005). An overview over various micromechanical test methods is given in Fig. 1; a sketch of the single-fiber pull out test is seen in Fig. 1 (a).

Many test procedures to prepare the samples and perform the pull-out test have been developed by research institutes. However, since those are not standardized, the measured values are hard to compare. For the following discussion, we employ the FIMATEST system (Web-1) developed by Textechno together with the Leibniz-Institut für Polymerforschung (IPF) in Dresden and the Faserinstitut Bremen (FIBRE). FIMATEST is the first commercially available system to characterize the fiber-matrix adhesion strength by means of a pull-out test, the system is displayed in Fig. 2.

3.1 Sample Preparation for the Pull-Out Test

The embedding of the fiber into the matrix is the most critical point to gain reproducible results through the pull-out test. The fiber must be embedded in the center of the matrix droplet perpendicular to the surface of the matrix to avoid additional undesired shear forces during the measurement. Fig. 3 demonstrates an embedding process as performed with the FIMABOND device for a glass fiber embedded in a PA 6 matrix. To process thermoplastics, the sample chamber of the FIMABOND is flushed with inert gas, e.g. nitrogen. Typically, a pallet of the thermoplastic matrix is laid upon a crucible which can be heated up to 400°C with a contact heater from the back (Fig. 3, left). Once the matrix is

Figure 2: The FIMATEST System, consisting of the FIMBOND for sample preparation and FAVIMAT+ for performing the pull-out test

molten and a symmetric droplet of approx. 2.5 mm diameter has formed, the fiber is approached to the top of the matrix (Fig.3, mid) until contact is made. Then the fiber is embedded to predefined depth into the matrix where typical embedding depths range up to a few hundred microns. The embedding length for the evaluation of the results is determined during the actual pull-out by the force that is necessary to fully debond the fiber from the matrix. Finally, the matrix is cooled to room temperature (Fig.4, right). In the case of thermoset matrices, the embedding procedure is similar with temperature cycles that contain appropriate degassing and curing periods.

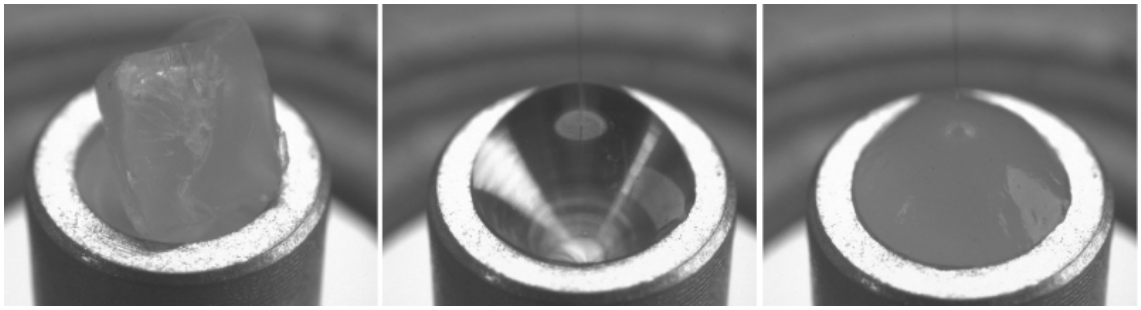

Figure 3: Process of embedding a glass fiber into PA 6 with the FIMABOND, left: flushing sample with inert gas, center: melting of the matrix material and contact between the fiber and the matrix surface and embedding, right: cooling and consolidation of the sample

3.2 Contact angle measurement during sample preparation

The images shown in Fig. 3 are recorded by the FIMABOND device under 45° with respect to the fiber axis and are used to tightly control the embedding process. An additional camera system is monitoring the contact region under 90° angle to measure the contact angle between the matrix and the fiber. In this way, wettability and impregnation are additional topics that are addressed with the FIMATEST system. Notably, FIMATEST enables the observation of contact angles under elevated temperatures and thus also on thermoplastic matrices. An exemplary meniscus recorded under 90° is shown in Fig. 4 (left) and the corresponding evaluation of the contact angles in Fig. 4 (right).

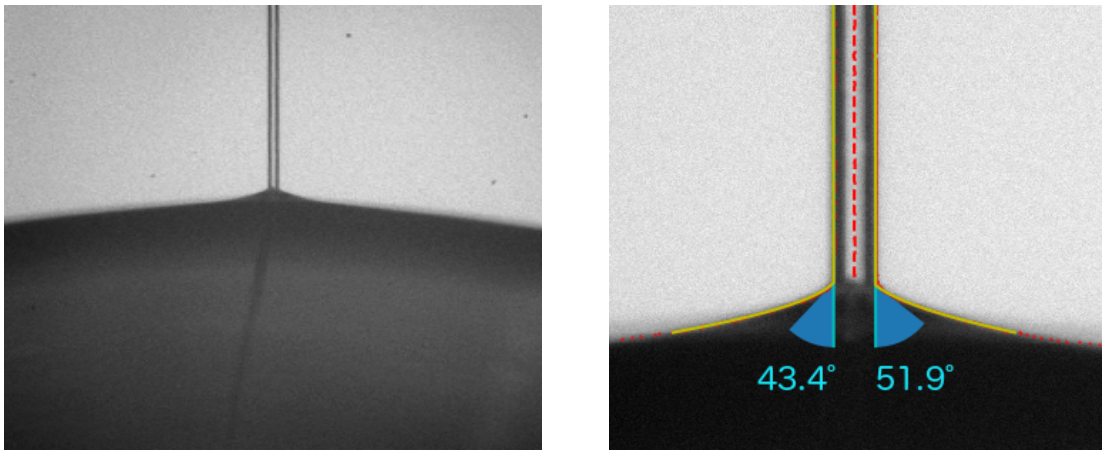

Figure 4: Observation of the meniscus forming between fibre and matrix during the embedding process as seen by the FIMABOND device – here, between a glass fibre and an epoxy resin. Left: the full image, right: the automatic evaluation of the contact angle on both sides of the fiber.

3.3 Performing the Pull-Out Test

Textechno's single fiber linear-density and tensile tester FAVIMAT+ (Web-2) is used to perform the pull-out test (Fig. 2, right). The FAVIMAT+ is equipped with a high-resolution load cell (1 µN at 200 cN full range) and precision mechanics. The circumference of the embedded fiber must be known to fully evaluate the measured data. The circumference is determined – before embedding and pull-out test – by the FAVIMAT+ as well.

For the pull-out test, the prepared single-fiber composite sample is placed in the direct clamping system of the FAVIMAT+. To ensure a precise alignment of the fiber to the jaw faces and the matrix surface, a microscopic camera is integrated in the tester. In Fig. 5 the process of clamping the fiber as seen by this camera is shown. As soon as the clamp is closed the pull-out test is started, the force-displacement curve is recorded, and evaluated automatically by the software of tester.

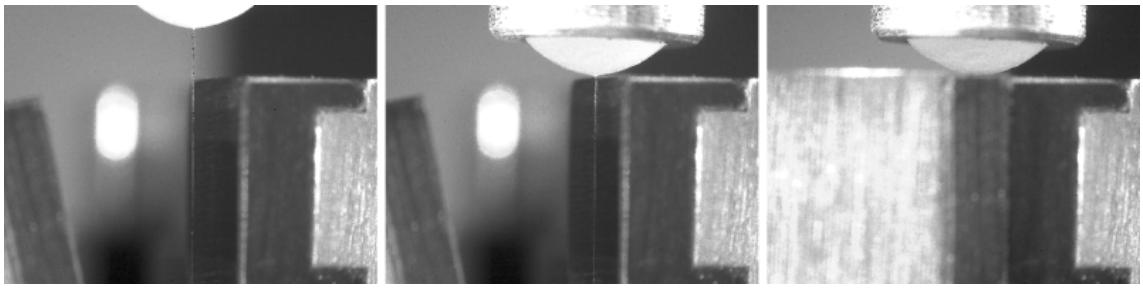

Figure 5: Process of adjusting the sample in the clamping system. The sample is placed with the fiber pointing down and held by the force transducer of the FAVIMAT+ testing machine (left). The fibre is aligned parallel to the right-hand side yaw face (mid) and clamped at minimal distance to the matrix droplet (right).

3.4 Evaluation of the Pull-Out Test Data

In Fig. 6 a typical force displacement curve recorded by the FIMATEST system is displayed. Since the force is applied to the fiber and due to the geometry of the sample, a crack is initiated where the fibre enters the matrix at a critical force value. This force is marked as F_d (debonding force) in Fig. 6 and is of

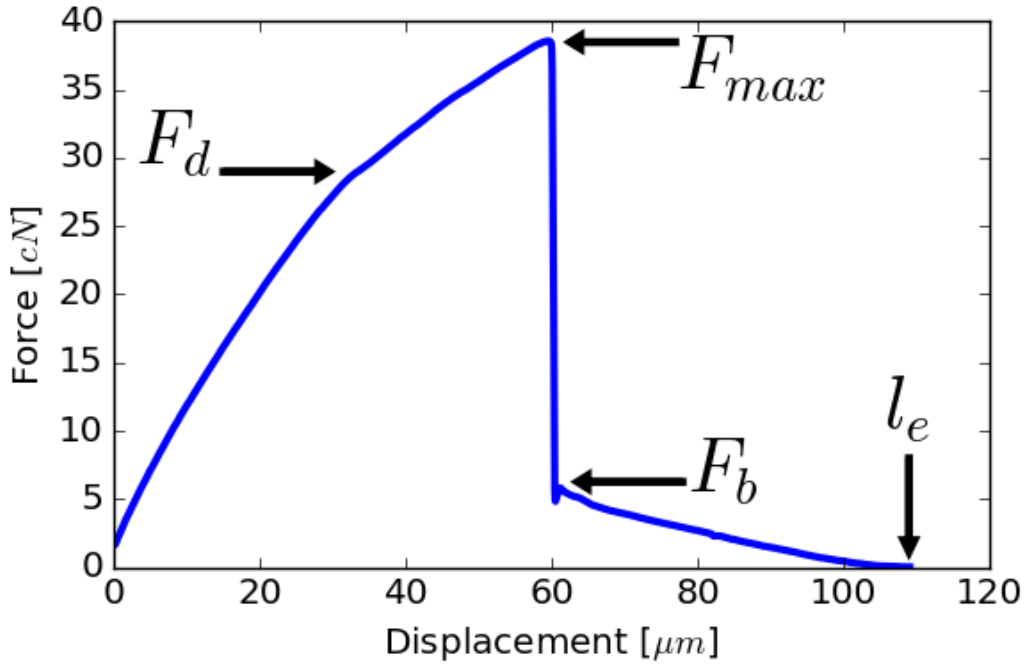

Figure 6: Force-displacement curve of glass fiber and epoxy resin.

special interest. From this point on the force still increases as one continues to pull on the fiber but with a smaller slope. Here, additional frictional forces occur between the debonded part of the fiber and the matrix as the crack is still propagated along the interface. At the maximum force F_{max} the fiber is completely debonded from the matrix and the force drops rapidly to the remaining frictional force F_b. The actual embedding depth l_e is reached, when the remaining frictional force drops to zero – the fiber has been completely pulled out of the matrix.

From the force-displacement curve the forces and length indicated in Fig. 6 are automatically evaluated. Based on these measured values and the individual fiber diameter the following quantities are calculated based on the extensive discussion in literature found in Zhandarov et al. (2003), Zhandarov and Mäder (2003) and Nairn (2000):

- The apparent interfacial shear strength (τ_{app}) is the maximum force normalized to the wetted area of the fiber. It is sufficient for a qualitative estimation, if different types of fiber-matrix combinations are to be compared.
- Based on the strain-based method, the local interfacial shear strength (τ_d) is calculated. Compared to the apparent interfacial shear strength, this quantity excludes the influence of the friction between the fiber and the matrix, considers the specific geometry of the sample and removes the impact of thermal stresses.
- The critical interfacial energy release rate (G_{ic})– based on an energy-based evaluation method – allows to consider the bonding on its own as well. The energy release rate is calculated as a function of the crack length and takes the deformation of fiber and matrix during the pull-out into account.

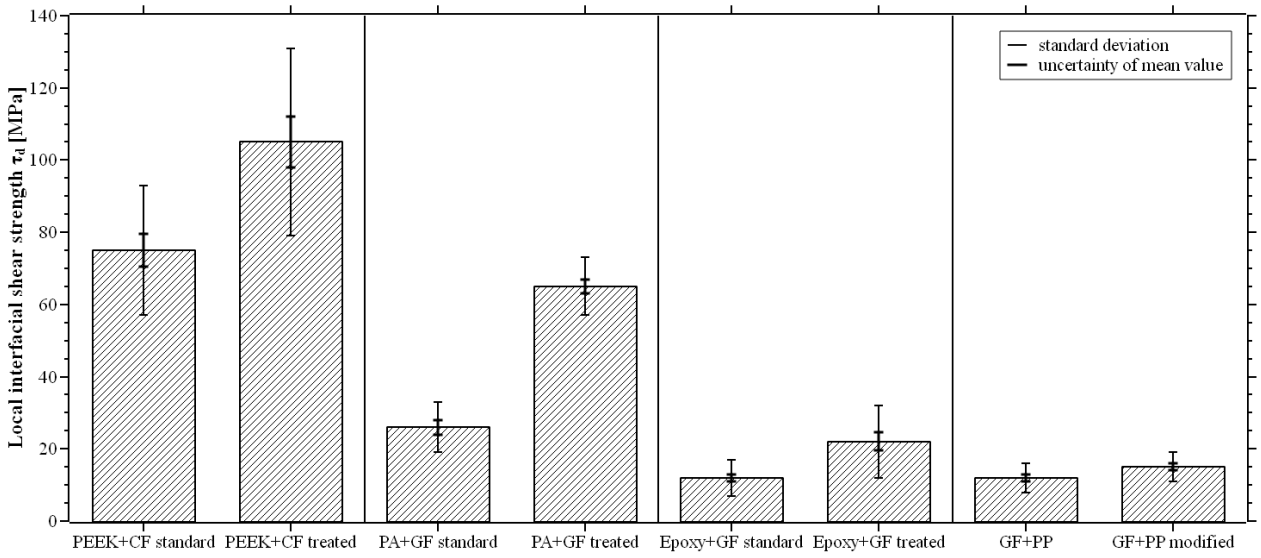

Figure 7: Local interfacial shear strength τ_d of standard and treated fibers in different matrices. The bars indicate the mean value found in 15 trials. The error bars show the standard deviation and the uncertainty of the mean value.

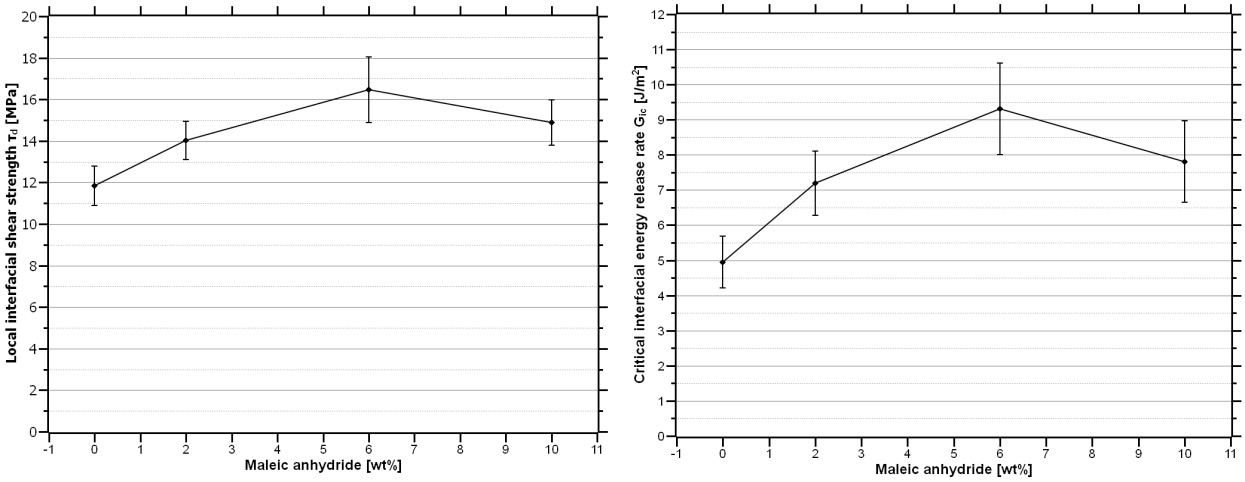

Figure 8: Fibre-matrix adhesion strength between GF and PP as a function of wt% maleic anhydride masterbatch in the PP. The error bars indicate the uncertainty of the mean value using 15 trials. Left: strain-based evaluation applied to the pull-out data, right: the energy-based evaluation is used. In both cases the maximum adhesion is found at 6 wt% masterbatch.

4 SELECTED DATA FROM DIFFERENT FIBER-MATRIX COMBINATIONS

Using the FIMATEST system described in the section above, we measure the local interfacial shear strength τ_d for several fiber-matrix combinations. Fig. 7 shows the results for standard and treated fibers of both carbon (CF) and glass (GF) embedded in different matrices – polyetheretherketone (PEEK), polyamide 6 (PA 6) and an epoxy resin (Epoxy). Moreover, a glass fiber embedded in different modified polypropylene matrices is shown. For these examples, the kind of fiber treatment or modification to the matrix or fiber is not specified in detail. However, we note that in all cases a significant improvement of the adhesion strength by treating the fibers or modifying the matrix is detected. The critical energy release

rate for the same set of fiber-matrix combinations yields the same discriminations at similar levels of significance.

Another example for a glass fiber embedded in PP is given in Fig. 8. This study has been performed together with PHP Fibers GmbH with a focus on the amount of maleic anhydride masterbatch that is added to the PP fibers in hybrid tapes to optimize the adhesion to glass fibres in the consolidated composite. Starting without any maleic anhydride, both the strain-based (Fig. 8, left) and the energy based (Fig. 8, right) evaluation of the pull-out test show that the highest fiber-matrix adhesion strength is reached at 6 wt% fraction masterbatch in the PP. The data indicates, that at 10 wt% masterbatch the optimum amount of maleic anhydride has been exceeded.

5 CONCLUSION

In Textechno's FIMATEST system the micromechanical pull-out test technique to characterize the fiber-matrix adhesion strength is combined with a highly repeatable and reliable sample preparation using the all-new FIMABOND device. In this way, it is possible to characterize the fiber-matrix adhesion strength in terms of the local and the apparent interfacial shear strength as well as the critical interfacial energy release rate (interfacial toughness). Differences in the fiber-matrix adhesion are usually due to the treatment of the fiber (e.g. sizing, plasma treatment) or the modification of the matrix material. With this advanced equipment, differences in the fiber-matrix adhesion can be traced on all kinds of fiber materials in combination with all thermoplastic, thermoset and even cement matrices. In addition, the optical measurement of the contact angle included in the FIMABOND device, opens up new possibilities to study wettability and impregnation under temperatures up to approx. 400°C and thus even on thermoplastic matrices.

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