Quality assessment of composites

The glass and carbon fibre industry continues to grow rapidly and into many new applications. A key quality characteristic of composite materials is the bond strength between fibre and matrix. This article describes the different mechanisms, as well as future testing solutions to ensure that a constant quality can be measured and achieved.

By

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The composites market is continuously growing as these materials are used in many applications. Key sectors such as automotive, aerospace and electronics require constant quality assurance methods and reliable testing equipment. The interphase between fibre and matrix is a key parameter for the quality of a composite material, both for thermoplastics and thermoset resins.

Glass fibre is still the dominant reinforcement fibre for all composites, growing at 5% per year (reference: AMAC GmbH) with a market already exceeding 5.2 million tons annual production in 2014. The carbon fibre market is growing at an even faster rate of more than 10% per year but, with approximately 60,000 tons annual production in 2014 (AVK market study), it is still significantly smaller but with more high-end segment applications.
This article describes the importance of the adhesion strength between the reinforcement fibre and the polymer matrix, providing a theoretical background for the bond strength of these two elements of a composite system.

Advanced fibre-matrix pull-out test equipment

The interphase between fibre and matrix is the key region of a composite material as it transfers the load from the matrix to the fibres and largely determines the effect of the reinforcement [1].

A challenging research issue is to reliably determine the interfacial bond strength and to relate the interphase characteristics to the static and fatigue properties of the composite. The interphase concept is discussed in the literature at the molecular and micro levels ([2] and its references). This paper presents micromechanical techniques for the characterization of fibre/matrix interfacial properties and the prediction of real composite properties [3]. This ap-

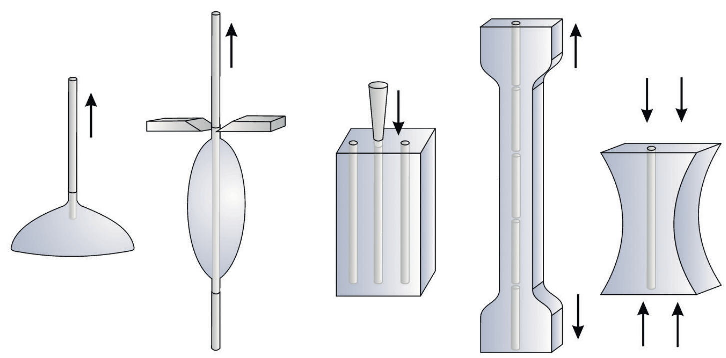

Fig. 1: Micromechanical tests can be divided into two groups: 1) tests where an external load is applied directly to the fibre: pull-out (a), microbond (b) and push-out (c); and 2) tests where the load is applied to the matrix: fragmentation test (d) and Broutman test (e)

Focus

This paper is written by Prof. Edith Mäder, a world-renowned expert in the field of interphases from the Leibniz-Institut für Polymerforschung Dresden e.V., and by Textechno, a leading designer and manufacturer of precision test equipment/systems for textile and man-made fibres. It describes how such test equipment can be designed and used for routine and ongoing quality assurance, both for glass and carbon fibre composites. Textechno's CEO Dr. Ulrich Mörschel and Dr. Michael Effing from AMAC GmbH recently announced, in a press release dated September 2015, that they are cooperating in the field of composites for business development and market introduction of Textechno's testing systems.

proach includes three closely interrelated tasks: (1) comparison of micromechanical tests for interface characterization; (2) review of existing practice, advantages of the pull-out test and adequate data treatment, and (3) development of semi-automatic equipment.

Comparative study of micromechanical tests for interface characterization

A large number of micromechanical tests have been developed to determine the parameters of interfacial interaction between fibres and matrices. These tests can be divided into the two groups [2] shown in Figure 1.

It is obvious that in an adequate test configuration, the stress distribution must be similar to that in a real composite. For composites with ductile matrices and brittle fibres (the matrix elongation-to-break is several times greater than the fibre elongation-to-break, e.g. carbon fibre-reinforced polymers), the fragmentation test is the most appropriate. On the contrary, for composites with brittle matrices, which fail through multiple transverse cracking (with reinforcing fibres bridging the cracks), the pull-out test appears to be closer to reality. However, as a tool for investigating interfacial adhesion, it can also be successfully used for matrices with large elongation-to-break, under the condition that interfacial debonding occurs at moderate relative matrix deformation near the fibre. Tests based on single fibre pull-out, which are regarded as especially interesting and important because they make it possible to relate the load transfer ability of the interface to adhesion parameters at molecular level, are considered below. The results obtained and the applicability of different models to the data will be discussed taking into account specific mechanical properties of the components.

Review of existing practice and advantages of the pull-out test

Since their invention more than 50 years ago, the pull-out and microbond tests have probably remained the most popular micromechanical techniques for determining the bond strength between fibres and matrices. This is due to their experimental simplicity, well-defined test geometry and high reproducibility of experimental results. In these tests, an adhesion contact is formed between the fibre and a matrix (Figure 1). After matrix curing/consolidation, the fibre is pulled out of the matrix. The applied force is recorded as a function of the displacement of the loaded fibre end. A typical force-displacement curve is presented in Figure 2.

An interfacial crack is initiated at some point of the interface (close to the fibre entry) when the applied force reaches a critical value ("debond" force, Fd) and then propagates along the embedded length towards the opposite fibre end. Interfacial friction plays an important role in this process. The frictional force arising in the debonded regions is added to the adhesion force contribution in still intact interfacial areas, and the force applied to the loaded fibre end continues to grow (segment AB). Only when the intact embedded fibre part becomes too short, the force begins to decrease. The maximum force, Fmax, recorded during the test can be much greater than the debond force, Fd. Then the whole embedded length fully debonds and the measured force drops from Fmax to a smaller value, Fb. From this moment and until complete pull-out, the "tail" force (segment CD) is due to friction between the fibre and matrix.

The interface strength is characterized using two main approaches: fracture mechanics (energy-based approach), which considers the critical energy release rate, Gic, as a debonding criterion and the main interfacial parameter, and shear-lag analysis (stress-based approach), where interfacial debonding is governed by the local interfacial shear strength (IFSS), τd. The adequacy of Gic and τd has been

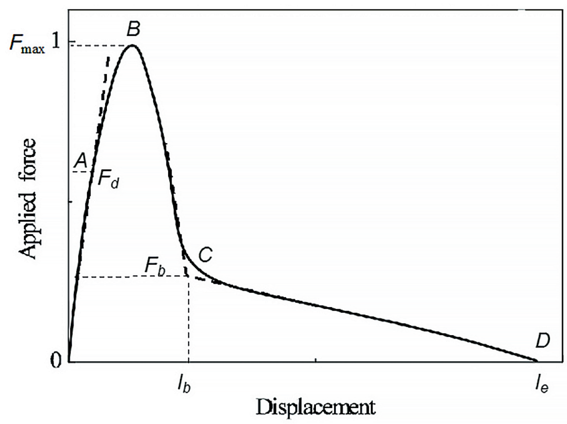

Fig. 2: Typical force-displacement curve

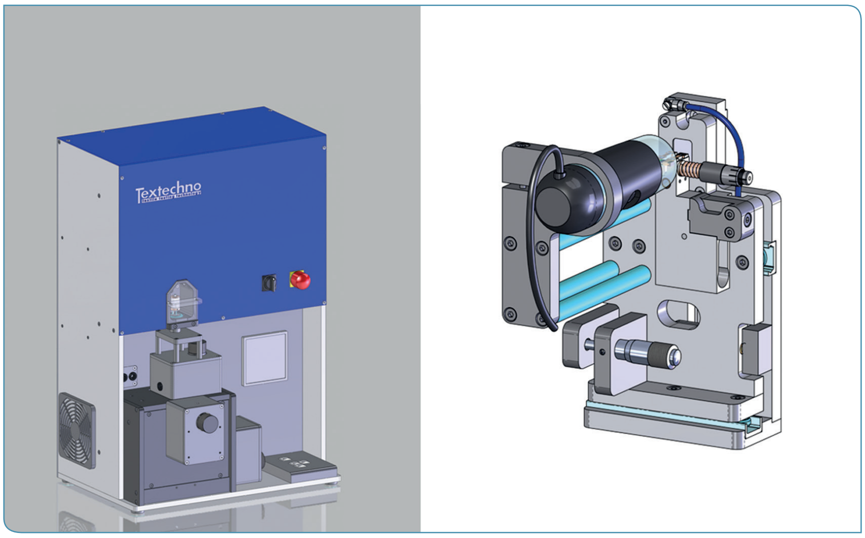

Fig. 3: (left) Textechno's new FIMABOND device for embedding the fibre in the matrix and (right) the pull-out device to be installed in Textechno's FAVIMAT+

extensively discussed in the literature, and it has been found that, for many fibre/matrix systems, interfacial failure can be satisfactorily described using either one of these criteria.

Although these tests are experimentally simple, the question of proper bond strength determination to obtain a specific numerical value remains rather ambiguous. At least eight different approaches to bond strength determination have been proposed, which can be classified according to the following alternative features:

- averaged/local (determination of the "apparent" or local interfacial shear strength (IFSS));
- stress-based/energy-based (adhesion strength parameter and failure criterion local IFSS, τd, or critical energy release rate, Gic);
- direct/indirect (adhesion strength determined from individual force-displacement curves or estimated from the data obtained for a set of specimens).

Since a theoretical comparison of different approaches is ongoing elsewhere, it seems interesting to compare these methods using reliable experimental data. A key feature is the preparation of single-fibre model composites.

New pull-out test system

The performance of composite materials strongly depends on the adhesion strength between fibre and matrix. At the microscopic level, different test procedures have been established by various research institutes. However, most results are not comparable since none of these tests is standardized or the equipment is not commercially available. In order to make a versatile and reproducible single-fibre pull-out test available to institutes and industrial customers worldwide, Textechno, leading experts in the field of fibre testing, are developing a suitable system together with the Leibniz-Institut für Polymerforschung Dresden (IPF) and the Faserinstitut Bremen (FIBRE). While the IPF has long-standing competence and experience in the field of fibre/matrix adhesion strength, FIBRE contributes by its experience in image analysis.
The system consists of two devices: the FIMABOND partially automated embedding station, which is suitable for all types of reinforcement fibres as well as for thermoset, thermoplastic or mineral matrices, and a device that performs high-precision pull-out tests as a new accessory to Textechno's single-fibre linear-density and tensile tester, FAVIMAT+.

One of the most critical points to ensure reproducible results in a micro-bond test is the precise embedding of the fibre, which is required to avoid bending forces. For this purpose, the fibre has to be embedded exactly at the centre of the matrix droplet. In the final version of the embedding station, the critical adjustment process ensuring this will be controlled by an image analysis software program developed by FIBRE.

The integration of the pull-out testing device into Textechno's FAVIMAT+ saves money for the user since this versatile single-fibre tester fulfils the requirements with its high-resolution load cell (1µN at 200 cN full range) and highly precise and sturdy mechanics. The integrated linear density test allows easy and precise determination of the linear density, cross section, modulus, breaking strength and elongation (in combination with the tensile test). A microscopic camera facilitates the adjustment of the clamps as close as possible to the matrix surface with perfect alignment. The complete set-up, called FIMATEST, consists of the FIMABOND embedding station and the FAVIMAT+ tensile and pull-out tester. The new system will be exhibited at JEC World 2016. ■

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