Composites: Part A 71 (2015) 157–167

Contents lists available at ScienceDirect

Composites: Part A

journal homepage: www.elsevier.com/locate/compositesa

Influence of plastic deformation on single-fiber push-out tests of carbon fiber reinforced epoxy resin

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ARTICLE INFO

Article history:
Received 7 October 2014
Received in revised form 7 January 2015
Accepted 17 January 2015
Available online 31 January 2015

Keywords:
A. Polymer-matrix composites (PMCs)
B. Plastic deformation
C. Micro-mechanics

ABSTRACT

In our study we present a procedure to measure and analyze single-fiber push-out force–displacement curves on carbon fiber reinforced polymers using a cyclic loading–unloading scheme. The measured cyclic force–displacement curves allow an energy-based evaluation of the interfacial failure, taking into account elastic, plastic and other dissipative energy contributions. Experimental and modeling results demonstrate that a deviation of the push-out curve from linear behavior does not correspond to crack opening but to a plastic deformation of the matrix. Evaluating the plastic energy yields a linear increase of the total plastic energy after a certain indenter displacement. This linear increase is attributed to stable crack propagation. Back-extrapolation of the linear part to zero total plastic energy using a linear regression yields the initiation of crack growth. It is concluded that for ductile matrix materials like polymers, a reliable interpretation of push-out data has to take into account plastic material deformation.

1. Introduction

Fiber–matrix adhesion is one of the key parameters for the mechanical performance of carbon fiber reinforced polymers (CFRP). Before a specific improvement of the fiber–matrix adhesion can be addressed, a characterization and quantification of the mechanical properties of the interface is required. So far mostly macromechanical tests with a shear loading of the fiber matrix interface are performed for this purpose, e.g., tests of the interlaminar shear strength (ILS). Although such tests deliver characteristic values for macroscopic samples, they average over different modes of failure and the inhomogeneity of the sample and, therefore, do not characterize the pure fiber–matrix adhesion.

To overcome this problem micromechanical tests, like the single-fiber pull-out test [1,2], the microbond test [3,4] or the fragmentation test [5,6] have been established. The specific values obtained from these tests characterize the fiber–matrix adhesion directly, but the tests have to be performed on model composites. A direct transfer of the measured quantities to realistic, macroscopically manufactured composites is questionable. An alternative is the single-fiber push-out test, which allows a micromechanical characterization of the fiber–matrix adhesion on industrially manufactured composites. During a single-fiber push-out experiment an individual fiber within a thin, plane-parallel composite sample (thickness below 100 μm), is loaded by a diamond indenter tip. With increasing load an evolution from crack-initiation, fiber–matrix debonding to fiber push-out occurs. During the experiment, the force–displacement curve is recorded, which allows to extract values to quantify the fiber–matrix adhesion.

Fig. 1 shows schematic push-out force–displacement curves of a ceramic matrix composite (a), of a polymer matrix composite (b), and the effect of the indenter hitting the matrix surrounding the fiber (c).

The force–displacement curve measured on ceramic matrix composites initially shows a nonlinear increase of force, which results from the formation of a stable contact area between indenter and fiber (cf. Fig. 1a). After this initial stage, a linear force–displacement relation is observed. This is generally interpreted as elastic deformation of the fiber–matrix system. A further increase of the indenter displacement results in a sharp change of slope, indicating crack initiation, followed again by a linear increase of force interpreted as stable crack growth [14,15] until maximum load is reached.

For fiber reinforced polymers an initial non-linear force increase is followed by a linear force–displacement relation (cf. Fig. 1b). In contrast to ceramic matrix composites, a further increase of the indenter displacement results in a gradual change to non-linear behavior in the majority of cases. The onset of this non-linearity is often interpreted as crack initiation followed by crack growth [7–11]. Various models were proposed to deduce the interfacial shear strength based on this non-linear behavior [9–12]. Most of

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http://dx.doi.org/10.1016/j.compositesa.2015.01.011
1359-835X/

the analytical models rely on assumptions concerning the stress distribution along the interface and, therefore, are restricted to special push-out geometries and types of loading. Such use of stress based values to characterize the fiber–matrix adhesion only allows a comparison of push-out tests using a similar experimental setup (e.g., same indenter geometries). This limitation can be overcome following the approach of e.g., Ref. [13] or Ref. [7] which is based on fracture mechanics to calculate a fiber–matrix adhesive energy. All these models assume that crack initiation occurs when a deviation of the force displacement curve from linear behavior is observed. The potential contributions of other dissipative mechanisms, e.g., plastic deformation of fiber or matrix, are not taken into account. This can be a valid approach to describe brittle systems like ceramic matrix composites. For carbon fiber reinforced polymers, however, the appearance of a plastic energy contribution is likely, since polymers typically show significant plastic deformation before failure. After the onset of non-linear behavior in CFRP, an increase of force up to peak load is observed [8,10,13,16].

Immediately after the peak load is reached some fiber reinforced systems show an abrupt decrease of load [7,17]. For other systems a gradual decrease of load is reported [14,18,19]. In both cases the load maximum is associated with a complete debonding of fiber and matrix followed by a push-out of the fiber. Load and indenter displacement at fiber push-out are important input parameters for several evaluation methods presented in literature. In some approaches the interfacial shear strength is evaluated based on a simple balance of force at the moment of push-out [8,15,17,20]. The values of indenter displacement and force at push-out are also required for energy based approaches, e.g., to determine the fracture toughness [14].

To ensure a reliable interpretation of a single-fiber push-out experiment contact between indenter and the matrix surrounding the fiber has to be avoided, since such contact to the matrix causes a superposition of different loading effects [8,14,15,20]. In the force–displacement curves a strong increase of load at indenter-matrix contact is observed (cf. Fig. 1c).

In the presented study we describe an experimental procedure to measure and analyze single-fiber push-out force–displacement curves using a cyclic loading–unloading scheme following the approach introduced by Refs. [14,25,27]. The resulting cyclic force–displacement curves allow an energy-based evaluation of the push-out test, taking into account elastic, plastic and further dissipative energy contributions during interfacial failure. Finite element simulations and microscopy measurements are used to back up the interpretation of the push-out experiment. The method of evaluation presented is independent of the specific shape of the force–displacement curve and is applicable to all fiber–matrix systems. Based on the experimental data obtained we are able to determine the force at crack initiation and the fracture energy dissipated during a push-out experiment. The experimental and simulation results emphasize the significance of plastic deformations of the polymeric matrix during push-out tests on CFRP and the necessity to take these effects into account to model failure behavior in polymeric composite materials.

2. Samples

The sample investigated is a carbon fiber reinforced polymer laminate containing HTA fibers (company: TohoTenax) and a RTM6 epoxy resin matrix (company: Hexcel). The resin is completely cured using a standard curing cycle (0.5 h at 120 °C, 2.5 h at 180 °C). Using a precision low speed diamond saw the resulting CFRP sample has been cut into thin slices (dimension 1 cm × 0.5 mm × 0.5 mm) with fibers aligned perpendicular to the cut face. These slices have then been lapped from both sides using a silicon carbide suspension (grain size: 3 μm), to achieve a plane parallel sample of a final thickness of 45 μm. In removing the outer 220–230 μm of sample material on each side it can be ensured that there is no residual damage introduced by the previous cutting process. The front and back surface of the 45 μm slice were finally polished with a SiO_2-suspension (grain size: 15 nm). The roughness R_a of the polished sample was quantified by atomic force microscopy (AFM) to be less than 100 nm. For the push-out experiments, the outer part of the sample is mounted with hot wax on a cleaned glass substrate. During cooling the sample is pressed onto the glass substrate to achieve full contact. The glass substrate itself contains a 50 μm wide groove positioned underneath the fibers investigated.

3. Experimental

3.1. Push-out measurements

The push-out experiments have been performed utilizing an *Universal Nanomechanical Tester* (UNAT) from ASMEC in combination with a diamond flat cone indenter (diameter at the top ~4.8 μm, Fig. 2). Compared to a standard Berkovich indenter, the flat end cone indenter has a much steeper opening angle. As was shown in a previous work, this effectively avoids hitting the matrix surrounding the fiber investigated during a push-out experiment [14]. The lateral positioning accuracy of the indenter is ±1 μm. For microscopic analysis of the push-out progress, standard push-out tests, i.e., tests using a displacement controlled loading rate of 40 nm/s up to push-out, have been stopped and unloaded at different stages of the push-out test, namely indenter displacements of

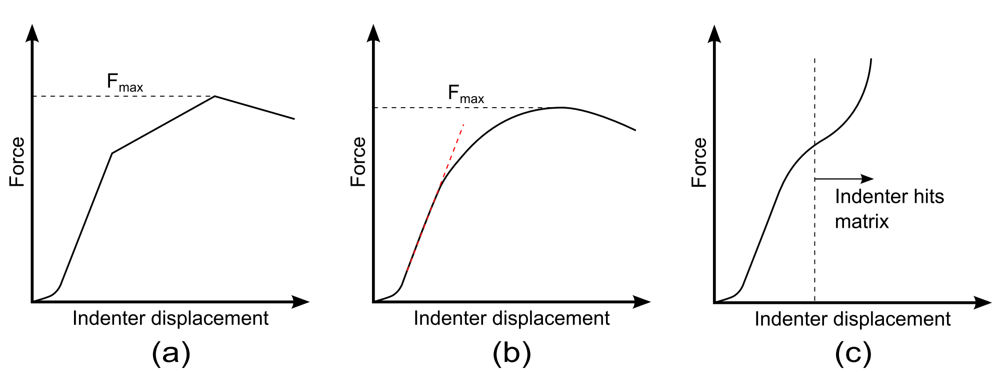

Fig. 1. Schematic force–displacement curves of: (a) push-out test of a ceramic matrix composite, (b) push-out test of a polymer-matrix composite, and (c) push-out test of a polymer matrix composite with the indenter hitting the matrix. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)