NUMERICAL AND EXPERIMENTAL STUDY OF SINGLE FIBER PUSH-OUT TEST: INFLUENCE OF FIBER/MATRIX INTERFACE MECHANICAL PROPERTIES

D.I. Batsouli[1], D.A. Dragatogiannis[1], S. Corujeira Gallo[2], Z. Zhang[2], H. Dong[2], G. Kotsikos[3] and C.A. Charitidis[1]

[1]Research Unit of Advanced, Composite, Nano Materials & Nanotechnology, School of Chemical Engineering, National Technical University of Athens, 9 Heroon Polytechniou st., Zographos, Athens, 15780, Greece
Email: dbatsouli@chemeng.ntua.gr, ddragato@chemeng.ntua.gr, charitidis@chemeng.ntua.gr,
Web Page: http://nanolab.chemeng.ntua.gr/
[2]School of Metallurgy and Materials, University of Birmingham, Edgbaston, Birmingham, B15 2TT, UK
[3] Policy Officer in Materials Nanotechnologies Biomaterials & Processes, European Commission, Rue de la Loi / Wetstraat 170, B-1049 Brussels, Belgium

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Abstract

Single fiber push-out experiments are used to characterize the mechanical behavior of fiber/matrix interface and have attracted significant attention due to their versatility. The effect of the interface mechanical properties on the corresponding composite mechanical behavior is complex and it clearly involves both experimental and theoretical challenges. With this regard, push-out tests were conducted on thin sections of a commercial composite at room temperature by applying various loading protocols to assess the micromechanical interaction between the carbon fibers and the polymeric matrix. The microscopic observations confirmed the push-out of individual fibers up to a distance in the order of 1.2 μm. To elucidate the deformation mechanism of the fiber/matrix interface during push-out test, a 2-D numerical model was developed utilizing a cohesive zone model (CZM) based on the commercial finite element (FE) software ANSYS 16.0.

1. Introduction

Carbon fiber-reinforced polymer composites (CFRPs) include a large carbon fiber volume fraction, with the result that the interface is the key region defining the set of composite materials properties. The nature of the interface between carbon fibers and polymeric matrices has important consequences for the mechanical properties of composite materials [1]. Therefore, the quantification of the interfacial shear strength (IFSS) and its link to the macroscopic properties is of technological importance, and several testing methods have been developed for this purpose. The testing techniques include: micro-bond, pull-out, push-in / push-out, fragmentation tests and Raman spectroscopy measurements [2-4].

The push-out and the push-in tests are attracting increasing attention because they can be conducted on small samples extracted from real composite materials [5]. It is nowadays accepted that the values of the IFSS obtained with these tests are good indicators of the interface strength from a comparative viewpoint but it is also recognized that the local environment in the single fiber composites is very different from the actual environment within the composite. Moreover, it has been shown that the local fiber volume fraction, the thermal residual stresses, and the polymer crosslink density (which are different in single fiber composites) can lead to significant changes in the properties of the interface. Therefore, the push-in and push-out tests, which are performed directly on composite samples, stand as the best options to obtain quantitative values of the IFSS [6].

In the case of the push-out test, the load is applied on a single fiber using a punch or indenter and a counter plate is used to support a thin sample. A complete debonding of the fiber is only possible for very thin specimens, with a thickness typically below 100 μm [7]. As the load increases, cracks initiate at the top of the specimen and they propagate along the interface. The interfacial shear strength at the fiber/matrix interface was approximated by the average shear strength, τ_s given by the following equation:

$$\tau_S = \frac{P}{2\pi r L} \tag{1}$$

where P is the load applied to a fiber when the interfacial de-bonding or sliding occured (the peak load), r is the fiber radius, and L the specimen thickness [8].

In addition, the assumption of uniform stress distribution may not be reasonable for the push-out tests, because of the very thin specimens required to produce complete debonding. With this regard, a computational model based on finite element (FE) method [9] is applied to provide new insights into the deformation mechanisms during push-out test of single fiber embedded within the polymer matrix and the subsequent determination of mechanical properties. The applied model is compared with results extracted by fiber push-out tests.

2. Materials and experimental techniques

A rod of Toray Rebar S12 composite, consisting of T700 carbon fibers in an epoxy matrix was used for the push-out tests. Initially, 700 μm thick discs were extracted from the rod and material was removed from each side of the discs by wet grinding with #1200 SiC abrasive paper. Finally, both sides of the discs were ground with #2500 and #4000 SiC abrasive paper and polished for 10 minutes with an OP-S suspension (oxide polishing suspension). In their final condition, the discs had a thickness between 25 μm and 70 μm.

The push-out tests were conducted in a Micro Materials Vantage nanoindentation instrument, fitted with a Berkovich tip. A custom-made holder having 60 μm wide slots, where the fibers could be pushed out was used. The samples were fixed onto the holder using cyanoacrylate adhesive. Individual fibers were selected at random for the tests, using an optical microscope with a magnification of 400x. The loading conditions used for the experiments are described in Table 1.

The specimens for the push-out tests were observed in a Jeol 7000 FEG-SEM (Scanning Electron Microscopy). The observations were conducted once the tests were finished so that the damage by the vacuum environment and the localised heating under the electron beam would not affect the results of the push-out tests. Atomic Force Microscopy (AFM) observations were conducted in a Veeco MultiMode, fitted with a SiN cantilever for contact mode. The elastic constant of the selected tip was 0.12 N/m. The results were analysed using the Bruker NanoScope Analysis 1.5 software.

Table 1. Loading conditions used for the push-out tests.

Mode	Cycles	Maximum depth (μm)	Maximum load (mN)
Double-loading	2	2 x 2	200

3. Finite element analysis of push-out test

A 2-D numerical model was developed utilizing a cohesive zone model (CZM) based on the commercial FE software ANSYS 16.0 to simulate the progressive failure of the fiber-matrix interface during push-out test. The specimen was modeled by using 2-D Structural Solid elements (PLANE182), 2D 4-node cohesive elements (INTER202) which is used to setup interface between fiber and matrix sections, TARGET 169 and CONTA172 where used as contact elements for Surface-to-Surface contact between the indenter (as the more rigid material) and the surface of the specimen in the region of indentation. The geometry of the push-out model was built based on experimental setups. Fig.1 depicts the schematic representation of the axisymmetric problem of fiber-matrix push-out test. The y axis was chosen as the symmetry axis, while two types of boundary conditions (BCs) were applied. The first type of BCs corresponds to the axial symmetry and the second one concerns the bottom of the matrix, for which no movements are permitted along the x, y-axis. Emphasis was placed on the fine meshing close to contact of the fiber with the matrix, where the largest deformation is expected and the mechanical behavior of the fiber/matrix interface was studied using CZM. The material properties of the carbon fiber, the matrix, the indenter and fiber-matrix interface are presented in Table 2.

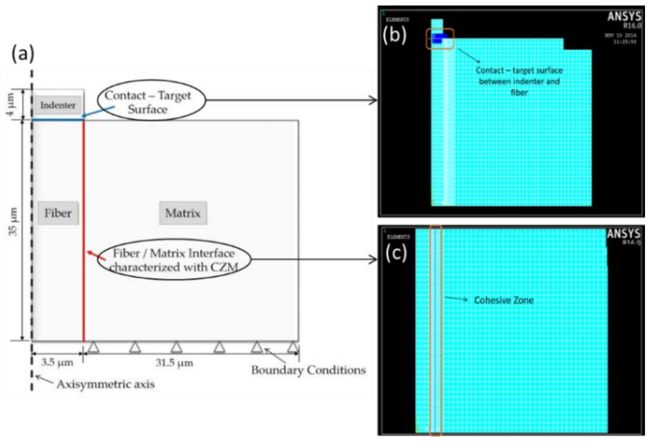


Figure 1. (a) Schematic representation of fiber/matrix push-out test with CZM, (b) Depiction of contact – target surface between indenter and fiber and (c) Depiction of CZM.

Table 2. Mechanical properties of materials used in FE simulation**.**

Materials	**Properties**		
	Young Modulus (GPa)	Poisson's ratio	Tensile strength (MPa)
Carbon Fiber (T700) *(7 μm diameter)*	230	0.3	4900
Matrix	3.8 / 10 / 100	0.35	70
Indenter	1141	0.07	-
Fiber/matrix interface	100 / 150 / 200	0.3	50 / 100 / 150

FE simulations were performed, and post-processed results were used in order to validate the model. Taken into account the parameter of thickness (push-out experiments were conducted on samples with various thicknesses); push-out simulations were performed and the load-displacement curves were extracted and correlated with experimental data.

4. Results and discussion

4.1. Experimental results

The SEM observations conducted on the samples after push-out tests clearly revealed single fibers which were depressed on the loading side, i.e. the surface of the fibers subjected to the loading cycle was at a lower plane compared to its nearest neighbours (Fig.2a). The surface of the loaded fibers exhibited minimum damage, although the edge of the Berkovich indenter clearly made contact with the surrounding material, causing some damage on the neighbouring fibers. In addition, observations on the back side of the samples showed fibers standing proud (Fig.2b), i.e. the surface of the loaded fibers was at a higher plane relative to the surrounding material, which was an indication of the success of the push-out approach. High magnification observations of the pushed-out fibers revealed elongated filaments of polymer still attached to the carbon fiber, even after debonding (Fig.2c). These filaments would continue to resist the displacement of the fiber, even at large strains, with the viscoelastic behaviour which is characteristic of the polymeric matrix.

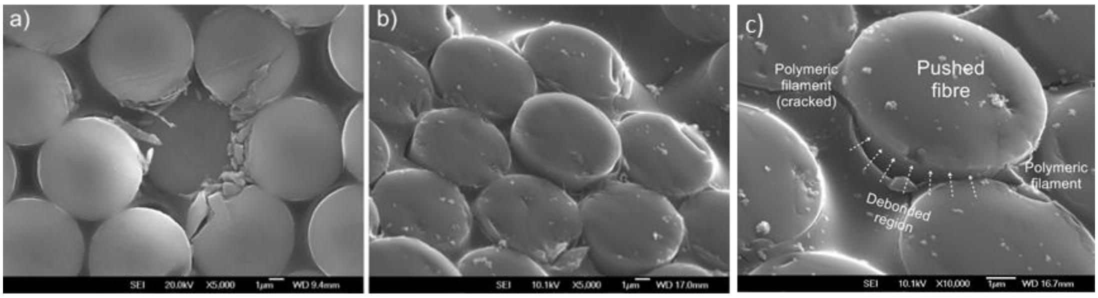


Figure 2. SEM micrographs of a specimen after the push-out tests: (a) front side (pushed-in), (b) back side (pushed-out) of the specimen and (c) high magnification SEM micrograph of a pushed-out fiber.

The AFM observations on the back side of the specimens confirmed that the fibers were pushed-out of the matrix (Fig.3). The push-out distance was between 0.8 μm and 1.2 μm in all cases, which is in agreement with the geometrical limitations of the Berkovich indenter used for the experiments. Once this displacement threshold was reached, the Berkovich indenter made contact with the surrounding material, thus pushing or damaging the surrounding fibers. A careful observation of Fig.3 shows the damage on the surrounding fibers and the displacement of the nearest neighbouring fibers, relative to the rest of the sample. Based on these observations, it was concluded that the load-displacement data obtained during the push-out tests for displacements below the 1.2 μm is representative of the micro-mechanical response of single fibers in the composite.

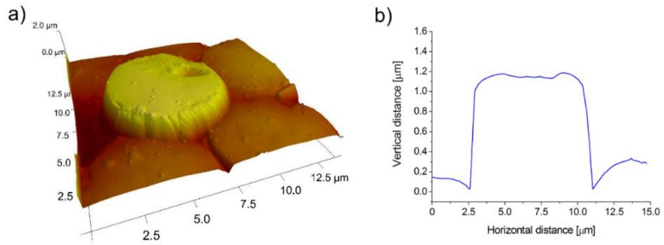


Figure 3. AFM observations of a specimen after the push-out test: (a) 3D image of the pushed-out fiber and (b) cross section profile of the same fiber.

The typical load-displacement curve of the push-out tests is illustrated in Fig.4, in which the characteristic regions are indicated. The curves typically show a non-linear region at the beginning of the loading cycle, which finishes at displacements of approximately 200 nm (point 1). This section of the curve is associated with elastic and plastic deformation of the loaded fiber, until a conformal contact with the Berkovich indenter is reached, as described in Fig.2. The curve shows a linear region (2), which is attributed to the loading response of the interface or the stable crack growth between the carbon fiber and the polymeric matrix. This region typically finishes at displacements in the order of 650 nm (point 3). At this point, the displacement increases more rapidly and a departure from the linear trend is observed, which is attributed to the debonding of the fiber from the matrix or the unstable crack growth, which often ended with a large displacement at constant load. The inset in Fig.4 shows the displacement during the dwell time at constant load corresponding to the plateau in the load-displacement curve (region 4). The increasing deformation at constant load is characteristic of a viscoelastic behaviour and some push-out events were observed in this region. The maximum displacement at the end of this region was invariably in the order 1.2 µm, in agreement with the AFM observations of the fiber displacement after the test. At this point, the load was removed to allow for an elastic recovery of the carbon fiber and a hysteresis loop formed upon reloading, which is frequently attributed to energy loses due to friction between the carbon fiber and the polymer. The displacement of the indenter beyond the 1.2 µm threshold is associated with a considerable increase in load, because of the physical contact between the Berkovich indenter and the surrounding fibers, as mentioned in Fig.2b.

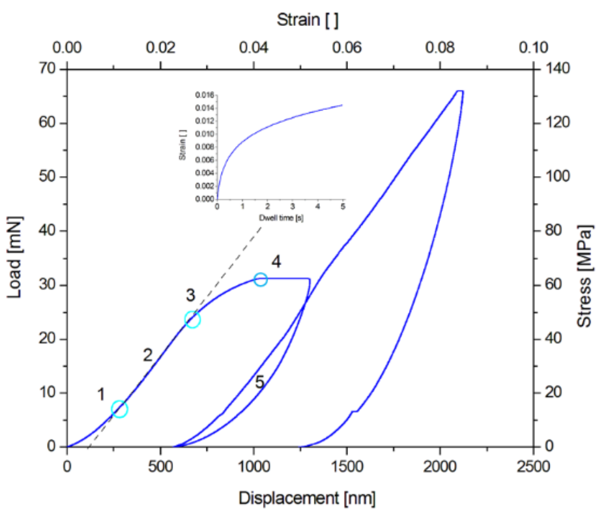
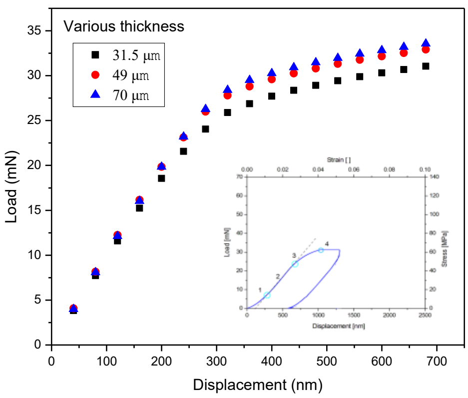


Figure 4. Typical load-displacement curve of the push-out tests.

4.2. Validation of the models

The thickness parameter was studied within simulations since push-out experiments were conducted on samples with various thicknesses. Fig.5a depicts the load-displacement curves obtained for three different thicknesses during push-out simulations. In the insert of Fig. 5a, it is shown the corresponded experimental load-displacement curve. It was observed that the maximum load of simulation results is comparable with experimental results (insert curve). In Fig.5b, the influence of matrix and fiber/matrix interface elastic properties in load-displacement curves is depicted. It is shown that the increased elastic modulus values of matrix and interface, as well as increasing stiffness, result in narrow elastic deformation region. Comparing parts with the same value of matrix and interface elastic modulus (E_M=3.8GPa / E_I=100GPa and E_M=100GPa / E_I=200GPa, respectively), one can see that an increase in tensile strength of the interface increases the elastic regime, characterized by a linear dependence between load and displacement, during the push-out of the fiber (Fig.5c). This is a worthy comparison as it indicates that by increasing the value of tensile strength, the interface becomes more rigid i.e., higher loads will be needed to reach the point at which the fiber part debonded from the matrix.

a)



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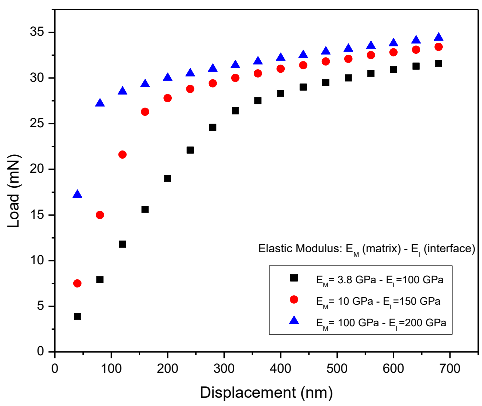

c)

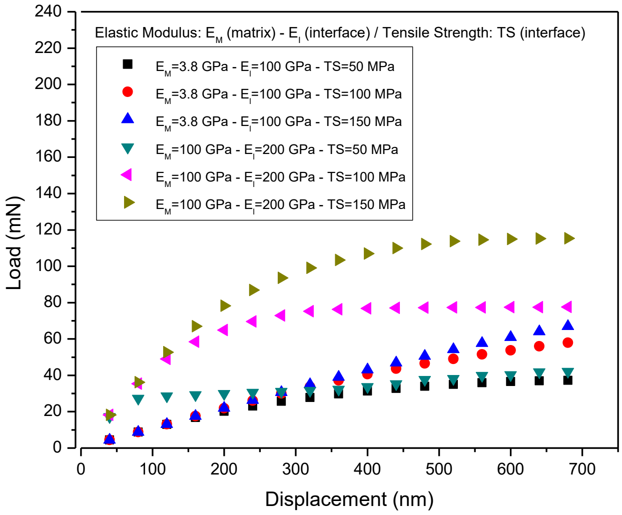

Figure 5. (a) Load – displacement curves obtained for (a) various thickness, (b) various matrix and interface Young's modulus and (c) various matrix and interface Young's modulus and interface tensile strength during FE simulations.

In Fig.6a, the distribution of shear stresses in XY plane is given for 0.68 μm displacement of the indenter into the fiber. It is observed that the maximum shear stress occurs along the fiber-matrix interface. The displacement along Y axis during the push-out test extracted from simulation, for the maximum applied load, is shown in Fig.6b. The fiber displacement was 0.68 μm, which is close to values of the push-out distance (between 0.8 μm and 1.2 μm) in all push-out experiments (Fig.6c).

Table 3 shows a summary of the measurements and calculations obtained from the push-out test curves under room conditions.

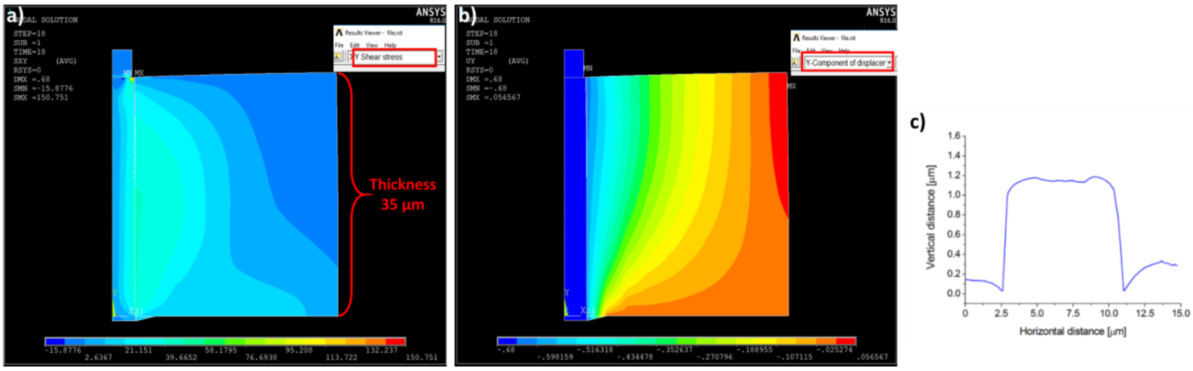

Figure 6. (a) Shear stress observation of a push-out test simulation, (b) displacement along Y axis and (c) cross section profile of the pushed-out fiber by AFM observations of a specimen after the push-out test experiment.

Table 3. Summary of results of push out tests.

Maximum load in Cycle 1 (mN) *(Experiments)*	Maximum load (mN) *(Simulations)*	Sample thickness (μm)	Average shear strength (MPa) *(Experiments)*	Average shear strength (MPa) *(Simulations)*
28.1 ± 3.88	32	35	18.2	20.5
28.1 ± 4.7	30	26	24.6	25.9
30.9 ± 1.58	31	29	24.2	23.9

3. Conclusions

A computational scheme based on Finite Element Method (FEM) has been applied to elucidate the effect of mechanical properties of interface on the deformation mechanism during push-out test. The results obtained from push-out tests under various experimental conditions (loading rates, maximum load, dwell time, thickness) provide valuable information regarding deformation mechanism and failure modes of fiber/matrix interface. The SEM and AFM observations confirmed the push-out of individual fibers up to a distance in the order of 1.2 μm. The interfacial shear strength of CFRP was successfully determined by means of the fiber push-out test when the thickness of specimen is thin enough, typically, smaller than 100 μm.

Acknowledgments

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