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Interface characterization in fiber-reinforced polymer-matrix composites

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Abstract

A novel methodology is presented and applied to measure the shear interface strength of fiber-reinforced polymers. The strategy is based in fiber push-in tests carried out on the central fiber of highly-packed fiber clusters with hexagonal symmetry, and it is supported by a detailed finite element analysis of the push-in test to account for the influence of hygrothermal residual stresses, fiber constrain and fiber anisotropy on the interface strength. Examples of application are presented to determine the shear interface strength in carbon and glass fiber composites reinforced with either thermoset or thermoplastic matrices. In addition, the influence of the environment (either dry or wet conditions) on the interface strength in C/epoxy composites is demonstrated.

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1. Introduction

The application of fiber-reinforced polymers (FRP) as structural materials has rapidly increased during the last 50 years owing to the unique combination of low density, high stiffness and strength as well as toughness. This set of properties is determined by the reinforcement architecture at the ply and laminate level as well as by the constituent (fiber. matrix and interface) properties [1-2]. While a lot research has been carried out to improve the mechanical properties of carbon and glass fibers and of thermoset and thermoplastic matrices, optimization of interface properties was more elusive because of the difficulties associated with the mechanical characterization of the fiber/matrix interface. Nevertheless, the ratio between the interface surface and the material volume in a typical composite reinforced with 60 vol. % of fibers with 6 μm in diameter is $\approx 2.0\ 10^5\ m^2/m^3$ and damage often begins by interface fracture (Fig. 1). Thus, the development of sound experimental methods to measure the fiber-matrix interface properties is an important task to design composites with improved performance.

Different testing techniques have been used to determine the mechanical properties of fiber-matrix interfaces. They include the pull-out test [4], the microbond test [5-7], the fragmentation test [8-10] as well as the push-out [11-16] and push-in tests [16-20]. Nevertheless, the interface strengths obtained from different tests often differ and even the results from one type of test are subjected to a large scatter [21], very likely because the analysis of the experimental data to obtain the interface properties is based on simplified shear lag analytical models. Moreover, it is not clear that tests based on coupons containing one fiber embedded in the matrix (pull-out, fragmentation, microbond) are representative of the actual mechanical and physico-chemical conditions in real composites

Due to the recent developments in nanoindentation, push-out and push-in tests of single fibers within a composite are very easy to perform and provide information about the interface properties within the actual composite. However, push-out tests require a thin membrane from the composite specimen, whose preparation is time-consuming, wheras push-in tests can be readily carried out on polished surfaces of the composite. Thus, a methodology based on the push-in test was recently developed to obtain the interface shear strength in FRP. The new approach takes accurately into account the effect of the local fiber environment, the thermal residual stresses and the fiber elastic anisotropy [22]. The application of this methodology to carbon and glass fiber composites reinforced with either thermoset or thermoplastic matrices

is presented in this paper. As another example of application, the influence of the environment (either dry or wet conditions) on the interface strength is measured.

2. Interface characterization by means of the push-in test

The mechanical characterization of the interface in the push-in test is carried out by loading a single fiber in compression by means of a flat punch connected to a nanoindentor (Fig. 2a). The schematic of the corresponding load (P) – displacement (h) curve is plotted in Fig. 2b. After an initial region (in which there is no full contact between the indenter and the fiber), the load-displacement curve presents a linear region, characterized by the stiffness S_0, which is controlled by the elastic deformation of the fiber and of the matrix. This is followed by a non-linear zone due to the progressive propagation of a crack along the fiber-matrix interface from the surface. The interface shear strength, τ_c^{SL}, is obtained from the critical load, P_c, which marks the onset of crack propagation, according to the shear lag model [4, 23-24]

$$\tau_c^{SL} = \frac{nP_c}{2\pi r^2} \tag{1}$$

where r stands for the fiber radius and n is a parameter which depends on the fiber and matrix elastic properties and on the local fiber volume fraction, i.e. the constraint imposed by the neighbor fibers. Following the shear-lag model, n can be determined from the stiffness of the elastic region in the P–h curve, S_0, according [4, 24]

$$n = \frac{S_0}{\pi r E_1^f} \tag{2}$$

where E_1^f stands for the longitudinal fiber elastic modulus. Nevertheless, it was shown [24] that the interface strength obtained with this methodology underestimated the actual properties, particularly in the case of composites with a large fiber volume fraction (> 50%) because it did not take into account accurately the constrain of the surrounding fibers. Moreover, the influence of other factors (elastic anisotropy of carbon fibers, thermal residual stresses or interface friction) was not taken into account in this analysis.

This methodology was modified by Rodríguez *et al.* [22] to overcome these limitations and obtain accurate values of the interface strength in composites containing large volume fraction

of either isotropic or anisotropic fibers. The methodology proposed assumes that the fiber/matrix interface behavior is independent of the fiber distribution and, thus, interface parameters can be obtained experimentally by selecting the most appropriate homogeneous fiber distribution. Of course, the specific load-displacement curve will depend on the exact fiber distribution that control the residual stress state and the fiber environmental constrain, but the exact numerical analysis and the diverse casuistry of fiber locations make this problem difficult to address and simplifications should be carry out. The novel methodology takes advantage of the fact that fibers surrounded by six fibers in a hexagonal packing pattern are very often found in composites containing a large fiber volume fraction (Fig. 3). The push-in test can be carried out in the central fiber, which is always subjected to the same constraint from the nearest-neighbor fibers. Moreover, the distribution of the nearest-neighbor fibers is always very regular in these regions and the effect of constrain can be accurately determined by means of a finite element analysis of the push-in test, that can also account for the effects of fiber anisotropy, hygrothermal residual stresses and interface friction. It should also be noticed that the creation of a new free surface in the composite (to carry out the push-in test) leads to a redistribution of the residual stresses generated in the bulk upon cooling from the curing temperature. The clamping residual stresses on the fibers are released at the free surface and fibers tend to protrude from the new free surface of the specimen. They are restrained by the development of shear stresses at the fiber/matrix interface that will be of opposite sign to the shear stresses induced by the indenter during the push-in test. These shear residual stress are accounted for naturally in the finite element simulations of the push-in test.

The geometry of the 3D finite element model of the push-in test is depicted in Figure 4. It includes the central fiber of radius r surrounded by six nearest neighbor fibers in a hexagonal pattern. The seven fibers are embedded in the matrix material and this central region is surrounded by a ring of a homogeneous medium, which stands for the composite. In case of nanoindentation with a flat punch, the analysis is carried in a wedge of 30º. The radius of the wedge is $32r$ while the model length parallel to the fibers is $150r$. It was checked that these dimensions were large enough so that the load–displacement curve during fiber push-in was not influenced by them [22]. The geometrical model was discretized with solid elements and cohesive surfaces were inserted between the central fiber and the matrix to account for interface decohesion and friction during the push-in test. It should be noted that the central fiber was not in contact with the nearest neighbors. The minimum thickness of the matrix layer in between was $0.02r$, and it was selected following the detailed observation of

hexagonal fiber clusters in the scanning electron microscope and also attending to convergence problems during the simulations.

The fibers were assumed to behave as thermo-elastic, transversally isotropic solids while the polymeric matrix was modeled as an isotropic, elastic solid. Plastic deformation of the matrix was not taken into account because it did not influence the critical stress at the onset of fiber–matrix debonding for this particular hexagonal fiber arrangement. It should be noted, according to [22] that matrix plasticity should be included to compute the interface shear strength in the case of isolated fibers or when the matrix shear flow stress is below 50% of the interface shear strength. Finally, the interface behavior was characterized by a cohesive crack model and interface decohesion and growth was controlled during the push-in test by the shear interface strength, t_c, and the interface fracture toughness, G [25-26]. This cohesive behavior is coupled with a Coulomb dry friction model to guarantee that once the interface is fully debonded the tangential sliding between matrix and fiber is opposed by Coulomb friction, controlled by the friction coefficient μ.

The finite element analyses of the push-in test included two steps. The thermal residual stresses were introduced in the model in the first one by means of an initial elastic thermal step, in which the temperature was homogeneously reduced from the curing temperature up to the test temperature without external constraints. Afterwards, the push-in test was simulated in the second step by means of the vertical displacement of a rigid, cylindrical flat punch (of the same diameter as the one used in the experiments) on the central fiber. More details can be found in [22].

As a result of an extensive finite element simulation campaign of the push-in test, it was shown that the actual interface strength, t_c, was given by

$$t_c = A t_c^{SL} - B\Delta T \qquad (3)$$

where t_c^{SL} was the interface strength given by the shear lag model (equation 1) from the experimental values of P_c and S_0, ΔT stood for the difference between the curing and the test temperature and A and B were two constants provided by the finite element simulations. It was found that A and B only depended on the thermo-elastic constants of the matrix and of the fibers and were independent of the interface fracture energy G and of the friction

coefficient μ, while the influence of the fiber elastic modulus in the longitudinal direction was included in t_c^{SL}.

The initial non-linear region observed in the small load regime of the *P-h* curve was attributed to the lack of parallelism between the indentor and the fiber surface. Once this region is exceed, the *P-h* curve becomes essentially linear until the debonding load P_c with an initial stiffness S_0 that is obtained by the least square fitting in this region, as depicted in Figure 2b. The critical load to compute the interface strength in equation (3), P_c, was determined from the intersection of the experimental *P–h* curve with a straight line that passes from two points of the *P–h* curve determined from two parallel lines to the initial stiffness S_0 drawn with offset displacements of 2% and 10% (Fig. 5). This definition of the P_c (somewhat arbitrary) provided very good estimations for the critical load at the onset of debonding in all the numerical simulations and can be readily applied to any experimental *P–h* curve.

3. Shear interface strength of fiber-reinforced polymers

The methodology presented above was applied to measure the shear interface strength at ambient temperature in three unidirectional FRPs, which include several composites frequently used for primary and secondary structures in aerospace and other engineering applications. Two of them were reinforced with AS4 carbon fiber embedded in either a 8552 thermoset (Hexcel) or a PEEK (polyether-ether-ketone) thermoplastic matrix, while the third one was made of a MTM44-1 epoxy matrix (Cytec) reinforced with S2 glass fibers. In addition, the effect of water absorption on the strength of fibre/matrix interface was assessed in the AS4/8552 composite.

3.1 Materials and experimental techniques

All the materials were manufactured in autoclave following the standard procedures recommended for composites with aerospace quality. Laminates were prepared by manually stacking prepreg sheets with different sequences ($[0]_{12}$, $[45/90/-45/0]_s$, and $[0]_8$, for the AS4/8552, AS4/PEEK and S2/MTM44-1 composites, respectively). The thermoset materials were cured following the cure cycle specified by the prepreg supplier: 2 hours at 130ºC followed by 2 hours at 180ºC with 6.2 bars of pressure for the AS4/8552 composite and 1 hour at 110ºC followed by 2 hours at 180ºC with 7 bars of pressure for the S2/MTM44-1

material. The AS4/PEEK material was also consolidated in autoclave during 20 minutes at 390 ºC with 7 bars of pressure. The fiber volume fraction was ≈58% in the AS4/8552 and AS4/PEEK laminates and the thickness of plies after curing/consolidation was ≈0.178 mm. The fiber volume fraction and the cured ply thickness was slightly higher (≈66% and 0.256 mm) in the S2/MTM44-1 composite. An optical micrograph of the cross-section of the AS4/8552 composite is depicted in Figure 6a, showing a homogeneous fiber distribution without either pores or large matrix rich regions.

The same procedure was used to prepare the composites for the interface strength measurements. Firstly, small rectangular pieces (10 x 3 x 2 mm^3) were extracted from the laminate plates and cut perpendicular to the fibers with a diamond wire. The cross section was successively polished using diamond slurries of 9, 3 and 1 μm in grain size to achieve a surface with good quality. The polished samples were dried in a stove at 60 ºC during 5 days prior to testing to remove the water absorbed during polishing. In the case of the AS4/8552 composites, a second set of polished samples was aged at 70ºC and 85% of relative humidity in an environmental chamber. The samples were smaller than the ones recommended by the DIN EN2823 standard [27] in order to accelerate the water uptake. The specimen weight was periodically measured and the aging was finished when the specimens were fully saturated (typically after 12 days of exposure to the hot/wet environment). It was assumed that all the water was absorbed by the epoxy resin.

Push-in tests were carried out in a Hysitron TI 950 TriboIndenter with a flat punch of 5 μm in diameter, which was smaller than the diameter of the AS4 carbon fibers (7 μm) and of the S2 glass fibers (9 μm). The diameter of the flat punch is large enough to guarantee no plastic residual imprint is produced in the tested fiber and the non-linearity of the load-displacement curve can be attributed to the fiber/matrix interface debonding. Fiber push-in tests were performed on the central fiber of highly-packed fiber clusters with hexagonal symmetry, an arrangement easily found in the three materials. All tests were performed under displacement control at 40 nm/s, which is slow enough to neglect any strain rate effects on the polymeric matrix but fast enough to avoid thermal drift effects during the test. Around 15 push-in tests were carried out at ambient temperature in each material and condition (dry and wet in the case of the AS4/8552 composite).

3.2 Results

The force-displacement curves corresponding to the push in tests in the AS4/8552, AS4/PEEK and the S2/MTM44-1 composites are plotted in Figures 7, 8 and 9, respectively. All curves showed a similar shape, which was depicted in Figure 2b. The initial non-linear region (due to the accommodation of the indenter tip to the fiber surface) was followed by a linear region controlled by the elastic deformation of the fiber under the indenter. The slope of this linear region, S_0, was determined by the least squares fitting of the force-displacement curve in a load range that was typically between 20 and 30 mN. The behavior becomes nonlinear at the onset of debonding that takes place at the critical load P_c . This load was determined for each curve following the procedure indicated in Fig. 5 and is marked with a solid square in each curve. Crack propagation along the fiber-matrix interface was mainly responsible for the non-linearity observed afterwards. It is worth noting that the experimental scatter was small in all cases, and this is indicative of the good quality of the materials (which show consistent properties) as well as of the robustness of the experimental procedure to determine the interface strength.

The values of P_c for the three materials are presented in Table 2. In the case of the AS4/8552, the results include the values obtained under dry and wet conditions. The interface strength, as predicted by the shear lag model, t_c^{SL}, can be obtained from P_c using equations (1) and (2), together with the experimental stiffness S_0 of the load-displacement curves (Table 2) and the elastic modulus of the fiber in the longitudinal direction, E_1^f (Table 1). The shear interface strengths provided by the shear lag model, t_c^{SL}, provide an adequate ranking of interface strengths among the different composites but the quantitative values do not take into account the influence of the constrain induced by the surrounding fibers as well as of the thermal residual stresses induced upon cooling from the curing/consolidation temperature as well as the residual stress redistribution induced by the presence of a free surface. Moreover, matrix swelling due to water uptake in the case of the wet composites (which also influences the residual stresses) is neither accounted for.

The influence of these factors is included in the parameters A and B of equation (3), shown in Table 3, which were obtained from the finite element simulation of the push-in test (Figure 4). The thermo-elastic properties of fibers and matrices used in the simulations can be found in Table 1. The thermo-elastic properties of the homogenized composite surrounding the central

fibers and the six nearest neighbors in the finite element model (Figure 4) were calculated from these using the Chamis equations and the corresponding fiber volume fraction for the carbon-fiber composites [30], and are given in Table 2. In the case of the S2/MTM44-1 composite, they were obtained from the finite element simulation of a representative volume element of the microstructure of the unidirectional ply, following [31]. Moreover, matrix swelling due to the water uptake has to be taken into account in the simulations of the AS4/8552 composite tested under wet conditions. This was carried out by modifying the matrix coefficient of thermal expansion, α^m, to include matrix swelling according to

$$\alpha^{m*} = \alpha^m - \frac{\beta \Delta M}{\Delta T} \tag{4}$$

where β is the coefficient of moisture expansion ($2.68\ 10^{-5}$) for the 8552 epoxy resin [32]) and ΔM is the increment in water content in the composite (divided by the weight of the dry resin), which was equal to ≈3%.

The actual temperature drop, ΔT, in equation (3) deserves some additional considerations. In the case of thermoset matrices, ΔT was equal to the difference between curing and room temperature because the glass transition temperature of 8552 and MTM44-1 epoxy resins was very similar to the curing temperature (180 ºC). Thus, it may be assumed that the build up of residual stresses starts from the beginning of cooling process. However, the residual stress generated upon cooling from the consolidation temperature in the case of the AS4/PEEK composite would be overestimated under the same hypothesis because the glass transition temperature of PEEK (180 ºC) was much lower than the consolidation temperature (390 ºC). Dynamic mechanical analysis of PEEK [33] showed that the elastic modulus of PEEK increased rapidly below 220 ºC and, thus, ΔT = 200 ºC was chosen in this case.

The actual shear interface strength, τ_c, for each composite material, computed according to equation (3), is shown in Table 3. It is worth noting that both residual stresses and fiber constrain (which are not considered in the standard shear lag approach) increased the interface shear strength by a factor of ≈2 or ≈2.5 for carbon and glass fiber composites, respectively. Moreover, water uptake reduced the shear interface strength of the AS4/8552 composite by 19% (from 64 to 52 MPa). This effect can be endorsed to the relief of the residual stress due to the matrix swelling, which is opposed to the matrix contraction upon cooling. It should also

be noticed that water absorption in the matrix may also reduce the matrix moduli and yield strength, which can also lead to further reductions of the interface strength [22].

The experimental results in Table 3 also show that the interface strength of the AS4/PEEK composite is significantly lower than that of the AS4/8552 thermoset counterpart. In both cases, the matrix shear strength is larger than interface strength so the push-in test is controlled by the interface and matrix plasticity effects can be neglected. This result (that may be critical for interface-dominated properties, such as the interlaminar shear strength) may be due to different factors. For instance, Gao and Kim [34] showed that the cooling rate may have a strong influence on the interface strength of AS4/PEEK composites. A large increase in the interface bond strength was observed when the cooling rate was decreased, which was attributed to the higher crystallinity and the flattened lamella chains at the fiber/matrix interface in composites processed at a low cooling rate. In addition, the differences in the interface strength between AS4/8552 and AS4/PEEK composites may be attributed to the changes in the fiber sizing that modify the chemical adhesion between matrix and fibers. Finally, the strongest interface was formed between the S2 glass fibers and the MTM44-1 epoxy matrix. In general, it is well established that sizing glass fibers with silane coupling agents enhances the adhesion and the durability of the fiber/epoxy interfaces [35] and the experimental results obtained by means of the fiber push-in test are in agreement with these observations. It should be noted, however, that yielding of the epoxy matrix near the fiber/matrix interface before the onset of interface fracture may have occurred in this case due to the high shear strength of the interface and this behavior may lead to an overestimation of t_c^{SL}.

4. Conclusions

A novel methodology is presented to measure the interface shear strength of fiber-reinforced polymers. The new method is based on push-in tests carried out on the central fiber of highly-packed fiber clusters with hexagonal symmetry, a feature easily found in structural composites reinforced with high fiber volume fractions. These tests require minimum specimen preparation and can be easily carried out with modern nanoindenter platforms. The shear interface strength is obtained from the critical load for interface decohesion, P_c, and the elastic stiffness of the load-displacement curve, S_0, be means of a methodology based on the shear lag that is able to take into account the influence of the hygrothermal residual stresses, of the fiber constraint and of the anisotropic fiber properties.

The methodology was applied to determine the shear fiber/matrix interface strength in various thermoset and thermoplastic matrix composites reinforced with either carbon or glass fibers. In addition, the influence of moisture absorption on the interface strength was assessed in one material. The results were consistent with the current knowledge on the strength of interfaces in fiber-reinforced polymers and the experimental scatter was very limited in all cases, demonstrating the robustness of the technique to measure the shear interface strength.

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FIGURES

Figure 1. (a) Development of damage by interface decohesion in a glass fiber reinforced polymer. (b) Detail of the onset of interface fracture between to adjacent fibers. The composite was subjected to compression in the vertical direction. The fiber diameter was in the range 15-30 μm. ~~From~~ Adapted from [3].

Figure 2. (a) Schematic of the fiber push-in test. (b) Representative load–fiber displacement curve during the push-in test. Reprinted with permission from [22].

Figure 3. (a) Optical micrograph of a cross-section of a carbon fiber/epoxy matrix composite. The regions enclosed by circles show hexagonal packing formed by one fiber surrounded by six nearest neighbors. (b) Scanning electron micrograph detail of one hexagonal packing showing the regular distribution of the neighbors around the central
fiber. Reprinted with permission from [22].

Figure 4. Finite element model of the push-in test.

Figure 5. Construction to determine the critical load at the onset of interface decohesion, P_c, from the experimental load-displacement curve.

Figure 6. (a) Optical microscope of a cross-section of an AS4/8552 composite ply. (b) Atomic force microscope image of a hexagonal fiber cluster after the AS4 central fiber has been pushed in with a 3μm diameter circular flat punch.

Figure 7. Load-displacement curves of push-in tests in AS4 carbon fibers on the cross section of an AS4/8552 composite at ambient temperature. (a) Dry. (b) Saturated with water. The critical loads, P_c, are marked with solid squares.

Figure 8. Load-displacement curves of push-in tests in AS4 carbon fibers on the cross section of an AS4/PEEK composite at ambient temperature. The critical loads, P_c, are marked with solid squares.

Figure 9. Load-displacement curves of push-in tests in S2 glass fibers on the cross section of an S2/MTM44-1 composite at ambient temperature. The critical loads, P_c, are marked with solid squares.

Tables

Table 1. Thermo-elastic properties of fibers and matrices. Properties for the AS4/8552 composites are found in [28], while those corresponding to the PEEK thermoplastic are taken from [29]. Properties of S2 and MTM44-1 matrix were provided by the supplier.

Material	E_1 (GPa)	E_2 (GPa)	G_{12} (GPa)	G_{23} (GPa)	ν_{12}	α_1 (K^{-1})	α_2 (K^{-1})
AS4	231	13.0	11.3	4.45	0.3	$-0.9\ 10^{-6}$	$7.2\ 10^{-6}$
S2	85	85	35.4	35.4	0.2	$16.0\ 10^{-6}$	$16.0\ 10^{-6}$
8552 (dry)	4.67	4.67	1.73	1.73	0.35	$52.0\ 10^{-6}$	$52.0\ 10^{-6}$
8552 (wet)	4.67	4.67	1.73	1.73	0.35	$1.56\ 10^{-6}$	$1.56\ 10^{-6}$
PEEK	4.1	4.1	1.51	1.51	0.356	$45.0\ 10^{-6}$	$45.0\ 10^{-6}$
MTM44-1	4.0	4.0	1.43	1.43	0.4	$40.0\ 10^{-6}$	$40.0\ 10^{-6}$

Table 2. Homogenized thermo-elastic properties of the composite materials used in the finite element simulations of the push-in test.

Material	E_1 (GPa)	E_2 (GPa)	G_{12} (GPa)	G_{23} (GPa)	ν_{12}	α_1 (K^{-1})	α_2 (K^{-1})
AS4/8552 dry	136	9.11	4.87	3.24	0.32	$21.3\ 10^{-5}$	$26\ 10^{-6}$
AS4/8552 wet	136	9.11	4.87	3.24	0.32	$0.14\ 10^{-6}$	$4.83\ 10^{-6}$
AS4/PEEK	136	8.56	4.45	3.05	0.32	$18.4\ 10^{-6}$	$23.1\ 10^{-6}$
S2/MTM44-1	60.85	19.75	6.87	7.05	0.28	$16.7\ 10^{-6}$	$25.3\ 10^{-6}$

Table 3. Results of numerical simulations and push-in experiments to determine the interface strength τ_c.

		Parameters of eq. (3)			Experimental results of the push-in tests			
Material	**Condition**	A	B (K^{-1})	ΔT	S_0 (N/nm)	P_c (mN)	τ_c^{SL} (MPa)	τ_c (MPa)
AS4/8552	dry	1.4065	0.1282	160	105 ± 2.7	62 ± 3.7	31 ± 1.9	64 ± 2.6
AS4/8552	wet	1.5458	0.0414	160	120 ± 7.9	52 ± 3.8	29 ± 1.7	52 ± 2.7
AS4/PEEK	dry	1.5533	0.0617	200	96 ± 2.8	35 ± 1.6	16 ± 0.8	37 ± 1.2
S2/MTM44-1	dry	2.5006	0.0012	160	111 ± 3.9	69 ± 7.7	44 ± 6.5	110 ± 13.4