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Letter to the Editor

Estimation of the local interfacial strength parameters of carbon nanotube fibers in an epoxy matrix from a microbond test data

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ABSTRACT

Local interfacial strength parameters (local interfacial shear strength (IFSS), critical energy release rate) and interfacial frictional stress between continuous carbon nanotube fibers and epoxy matrix have been estimated using published experimental data from a microbond test. The 'indirect' method (from the maximum recorded force as a function of the embedded length) and the calculation from an individual force–displacement curve yielded very similar results. The estimated local IFSS value (about 50 MPa) is much greater than the effective IFSS reported for this fiber–matrix pair (14.4 MPa).

Zu et al. [1] determined the effective interfacial shear strength of continuous carbon nanotube (CNT) fibers in DER 353 epoxy resin by means of a microdroplet (microbond) test. They recorded 10 force–displacement curves for specimens in which interfacial debonding occurred and plotted the maximum fiber axial force, F_{max}, recorded during the test, as a function of the embedment area. (In Fig. 1, we replotted their data as F_{max} *versus* the embedded length, l_e). Then these data were fitted to the linear function passing through the origin (curve 1), and the average effective interfacial shear strength (IFSS) was obtained from the slope of the fitting line. The authors found the effective (apparent) IFSS value (τ_{app} = 14.4 MPa) to be comparable to those of glass fiber/epoxy and carbon fiber/epoxy composites.

However, as is known, interfacial failure in a microbond specimen occurs not simultaneously over the whole embedded length, but gradually, through interfacial crack propagation, which is governed by a *local* interfacial strength parameter. There are two groups of models which describe the laws of crack initiation and propagation. In the stress-based approach, it is assumed that interfacial debonding starts when the shear stress at some point at the interface reaches its critical value, τ_d, which is called *the local interfacial shear strength*, and during the crack propagation, the shear stress near the crack tip is close to τ_d. The energy-based approach based on fracture mechanics considers the *critical energy release rate*, or *interfacial toughness*, G_{ic}, as a failure criterion which must be nearly constant during crack initiation and propagation. In contrast to the *apparent* IFSS, which depends on the specimen geometry and, first of all, on the embedded length, the τ_d and G_{ic} parameters can be considered as the composite properties constant for a given fiber–matrix pair. Besides, interfacial friction plays

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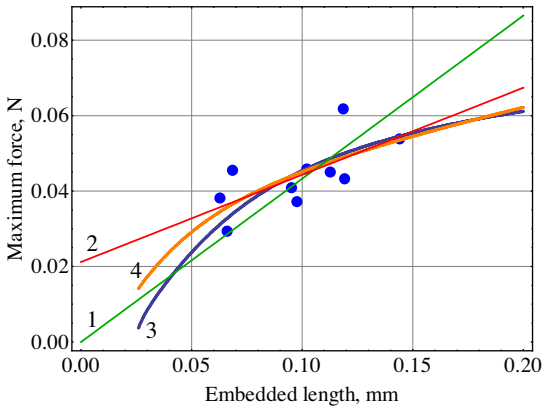

Fig. 1 – Maximum force *versus* embedded length in the microbond test on CNT fibers in DER 353 epoxy matrix. Filled circles denote experimental points from [1]. Curve 1 is the best "proportional" fit [1] (τ_{app} = 14.4 MPa); curve 2 is the best "non-proportional" linear fit estimating interfacial frictional stress; curve 3 is the best-fit stress-based theoretical curve (τ_d = 54.8 MPa, τ_f = 0); and curve 4 is the best-fit energy-based theoretical curve (G_{ic} = 9.10 J/m^2, τ_f = 4.39 MPa). (A colour version of this figure can be viewed online.)

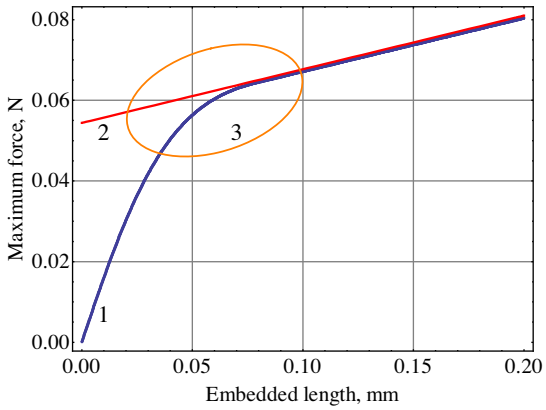

Fig. 2 – Theoretical plot of F_{max} as a function of the embedded length (1) and its asymptote as $l_e \to \infty$ (2) determined by interfacial friction. (3) denotes the region where experimental points are assumed to fall. (A colour version of this figure can be viewed online.)

an important part in fiber/matrix debonding; the recorded applied force is the sum of the adhesion force developing in the intact part of the interface and the frictional force in already debonded regions. As a result, the F_{max} and τ_{app} values are determined not by solely adhesion but by an intricate combination of adhesional and frictional contributions. It seems to be very interesting to separate these contributions and estimate both τ_d (or G_{ic}) and the frictional stress in the debonded zones. Fortunately, almost all information required for this estimation is available in [1].

In our analysis, we will use our own models described in detail elsewhere [2–4], in which the maximum force, F_{max}, for a microbond test is derived as a function of the embedded length, interfacial adhesion and friction, and some other parameters. In the stress-based approach this can be expressed as

$$F_{max} = F_{max}\,(l_e,\ \tau_d,\ \tau_f,\ \tau_T,\ \beta,\ V_f, \ldots), \qquad (1)$$

where τ_f is the frictional interfacial stress in debonded areas, which is assumed to be constant [5]; τ_T is a term having dimensions of stress, which appears due to residual thermal stresses; β is the shear-lag parameter as defined by Nayfeh [6]; V_f is the fiber volume fraction within the specimen, and the ellipsis designates mechanical properties of the fiber and the matrix (elastic moduli, Poisson's ratios, etc.) We have shown that a typical plot of F_{max} as a function of l_e has the form shown in Fig. 2. For small embedded lengths, the F_{max} value is determined predominantly by interfacial adhesion; however, for large l_e's, when the ultimate specimen failure occurs at the crack length close to the whole embedded length, the plot tends asymptotically to a straight line whose slope is proportional to τ_f. The experimental points usually fall somewhere between these extreme positions. The authors of [1] have chosen the fitting line such that $F_{max} \propto l_e$; this would be correct if the whole interface failed simultaneously (e.g., through plastic deformation or pure friction). However, if we admit that the fitting line may *not* pass through the origin, we obtain curve 2 (see Fig. 1), whose slope corresponds to the interfacial stress of 7.68 MPa. In our approach, this value should be considered as the *upper estimate* for the *interfacial frictional stress*, τ_f. Indeed, this line is parallel to the tangent drawn to the theoretical curve in the region with real experimental points, and its slope is always greater than the slope of the asymptote. And the fact that line 2 intersects the vertical axis at the point where $F_{max} > 0$ clearly indicates that interfacial interaction between the fiber and the matrix involves an essential adhesional contribution.

This contribution is determined by the local adhesion strength parameter (τ_d or G_{ic}) which can be estimated in several ways. First, we can fit experimental data by a theoretical curve (Eq. (1)) whose explicit form has been obtained by us in [4], using a non-linear least-square method with τ_d and τ_f as fitting parameters. The problem for a microbond test is that, though both τ_d and τ_f are assumed to be constant for all specimens, τ_T and β are functions of the fiber volume fraction, V_f, which, in turn, depends on the droplet shape. In order to calculate V_f, the droplet shape can be approximated by an ellipsoid. However, to do this, we must know at least the droplet diameter, D. The authors of [1] gave no information about the transverse size of the droplets; fortunately, we can calculate the diameter of the droplet on a fiber using a theory [7] which relates it to the droplet length (embedded fiber length), provided that the wetting angle at the matrix–fiber interface is known. We measured the wetting angle from the photograph in Fig. 1 in [1]; it was approximately $\vartheta \approx 51°$. Then, using this value, we calculated the diameters of all 10 droplets involved in the experiment. With good accuracy, these appeared to be linearly related to the embedded lengths: $D = 0.906\, l_e - 5.89$ (μm), similarly to the results reported by Scheer and Nairn [8] for glass/epoxy and Kevlar/epoxy systems. Having calculated the fiber volume fractions, as proposed in [2,4], and taken the mechanical properties of