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Interface toughness in fibre composites by the fragmentation test

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In the present work it is suggested that the degree of fibre–matrix bonding can be quantified by means of an energy for the nucleation of an interfacial debond, rather than by using a stress-based interfacial parameter. The energy necessary to initiate/nucleate an interfacial crack from its associated transverse fibre break during a single-fibre fragmentation test is calculated. The interface energy for the initiation of debonding is shown to be a function of the fibre and matrix geometrical and material characteristics, and of the initial debonding length. The novelty of the present work lies in the in-depth examination of the relative roles of the physical parameters that appear in the proposed theoretical model.

(Keywords: fragmentation test; interfacial debonding; energy balance model)

INTRODUCTION

The adhesion between a rigid fibre and a more ductile polymer matrix is usually measured by micromechanical testing using single-fibre microcomposite specimens. The single-fibre composite (or fragmentation) test is one of the most studied[1–5]. It involves a rather complex interfacial stress state, but only simplistic force equilibrium, and shear-lag or elastic–plastic analyses are used to analyse the test results in terms of the stress transfer ability, a mechanical parameter usually loosely termed the interfacial shear strength (τ). Despite the popularity of this experimental approach and the large amount of experimental results obtained over the years, some investigators[6–9] have suggested that τ may not be the critical factor governing fibre–matrix debonding, and that in the search for fracture criteria an energy-based theory is usually a better approach than a theory based on the identification of a stress at a point. We reached the same conclusion following an experimental study of the effects of water and temperature on glass/epoxy composites[9]. In that study, it was found to be impossible to interpret the results of fragmentation tests using the classical stress-based Kelly–Tyson approach, mostly because of the presence of extensive interfacial debonding (for example, following 2 h immersion at 95°C) and of the lack of a saturation state (usually after long immersion times, or under high temperatures). A theoretical model and first direct experimental results were recently presented by Wagner *et al.*[10]. The concept proposed is an energy balance scheme for the analysis of the initial interfacial debonding that arises as fibres progressively begin to break during the fragmentation test. This approach makes use of measurements performed at low strain levels, enabling the determination of the interfacial fracture energy associated with the initial debond, even in the case of composites that would never reach saturation during a fragmentation test. Moreover, such approach has the advantage of incorporating the length of the nucleating debond zone as a parameter, in contrast with the classical force balance interpretation of the fragmentation test for which the presence of such interfacial debonding zone is a complicating factor. However, as the energy balance presented here compares energies before and after fracture of the fibre and the interface, rather than computing energy changes as the interfacial debond propagates, it is not a strict fracture mechanics analysis.

In the present article, we present a novel in-depth study of the relative roles of the physical parameters that

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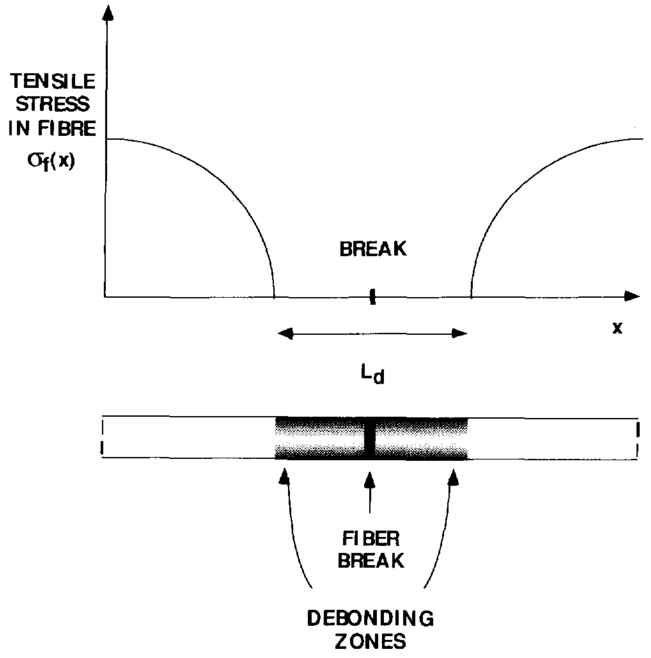

Figure 1 Isolated fibre break and associated initial interface debonding in a fibre fragmentation experiment. The applied stress is parallel to the fibre direction and the resulting tensile stress profile in the fibre, in close proximity of the fracture region, is schematically depicted

appear in the proposed theoretical model, and discuss the experimental implications.

SUMMARY OF MODEL

The full version of the new one-dimensional model is presented elsewhere[10] and only a summary is presented here. The model is based on the following line of thought. Consider a brittle, elastic fibre embedded in a more ductile, elastic, polymeric matrix that can be considered as infinite in size (*Figure 1*). For simplicity, all pre-existing fibre stresses (such as residual thermal stresses) are neglected. A tensile stress parallel to the fibre is applied to the composite, until the fibre breaks sequentially, from the weakest fibre defect to progressively less critical defects. Only the portion of such a fibre fragmentation process that takes place in the linear elastic region of the composite test is considered. A fibre break is accompanied by the simultaneous formation of an interface debonding zone, symmetrically on both sides of the fibre break site, as is often experimentally observed. This zone is small when a good interfacial bond exists, but can be very large for weakly bonded fibre–matrix systems. The length of the debond is L_d (counting on both sides), and the fibre stress, $\sigma_f(x)$, is zero at the fibre break site and considered to be negligible in the debonded region. Outside that region, the fibre stress builds up progressively towards a plateau value equal to the far-field average fibre stress. Debonding experiments are done on the first few fibre breaks. Thus the fibre breaks remain well separated and the stress build-up region can be analysed by considering stress build up from an isolated fibre break.

As a result of the fibre fracture, a portion of the strain energies in the fibre and matrix prior to the break are transformed into an energy contribution necessary for the formation of the fibre break surfaces, and into a contribution necessary for the formation of interfacial debonding. The energy available for the creation of such fracture surfaces is equal to the difference between the strain energy in the fibre and the matrix before the break (U^{prior}) and following the break (U^{post}), as follows:

$$\text{energy available} = [U_f^{\text{prior}} + U_m^{\text{prior}}] - [U_f^{\text{post}} + U_m^{\text{post}}] \tag{1}$$

where the subscripts f and m refer to fibre and matrix, respectively. Prior to fibre fracture, the fibre has uniform tensile stress and zero shear stress. Integrating over the anticipated debonding zone and stress build-up zone

$$U_f^{\text{prior}} = \tfrac{1}{2}E_f\epsilon^2\pi r_f^2\left(L_d + 2\int_0^\infty \mathrm{d}x\right) \tag{2}$$

where E_f is the axial modulus of the fibre, ϵ is the applied axial strain at the moment of break, r_f is the fibre radius, and x is the distance from the edge of the anticipated stress build-up zone. The matrix also has uniform axial stress and zero shear stress, giving

$$U_m^{\text{prior}} + \tfrac{1}{2}E_m\epsilon^2\pi(R^2 - r_f^2)\left(L_d + 2\int_0^\infty \mathrm{d}x\right) \tag{3}$$

where E_m is the modulus of the matrix and R is the radius of the matrix over which the stress is affected by the break event. After the fibre break, the fibre stress in the debond zone is zero (no interface friction), but both the fibre axial stress and shear stress in the stress build-up zone will be non-zero. Thus

$$U_f^{\text{post}} = 2\int_0^\infty\int_0^{r_f}\left(\frac{\sigma_f^2(x)}{2E_f} + \frac{\tau_f^2(x,r)}{2G_f}\right)2\pi r\,\mathrm{d}r\,\mathrm{d}x \tag{4}$$

where $\sigma_f(x)$ and $\tau_f(x,r)$ are the axial and shear stress in the fibre at a distance x from the fibre break and at radial position r, and G_f is the axial shear modulus of the fibre. Because the matrix is assumed to be infinite, the fibre break causes no change in the applied axial strain. Thus, in this one-dimensional analysis, the tensile stress in the matrix can only remain uniform and equal to $\sigma_m = E_m\epsilon$. The matrix shear stress, however, need not remain zero: it will be zero in the debond zone, but non-zero in the stress build-up zone. Finally

$$U_m^{\text{post}} = \tfrac{1}{2}E_m\epsilon^2\pi(R^2 - r_f^2)\left(L_d + 2\int_0^\infty \mathrm{d}x\right) + 2\int_0^\infty\int_{r_f}^{R}\frac{\tau_m^2(x,r)}{2G_m}2\pi r\,\mathrm{d}r\,\mathrm{d}x \tag{5}$$

where $\tau_m(x,r)$ is the shear stress in the matrix at a distance x from the fibre break at radial position r, and G_m is the shear modulus of the matrix.

To evaluate the integrals in equations (4) and (5) we need expressions for $\tau_f(x,r)$, and $\tau_m(x,r)$. These are calculated elsewhere[10]. Regarding $\sigma_f(x)$, we follow the shear-lag analysis of Cox[11] as summarized by Kelly and Macmillan[6], in which the axial stress in a fibre of finite length L is

$$\sigma_f(x) = E_f\epsilon\left[1 - \frac{\cosh\beta[(L/2) - x]}{\cosh(\beta L/2)}\right] \tag{6}$$

where x is the distance from one of the fragment ends, and

$$\beta = \frac{1}{r_f}\sqrt{\frac{2G_m}{E_f\ln(R/r_f)}} \tag{7}$$

For an isolated fibre break, as $L \to \infty$, this becomes $\sigma_f(x) = E_f\epsilon(1 - e^{-\beta x})$. The energy that must be expended to form the fibre and interface fracture surfaces is

$$U_s = 2\pi r_f L_d \Gamma_i + \pi r_f^2 \Gamma_f \tag{8}$$

where Γ_i and Γ_f are the energies associated with the formation of an initial interfacial debond and a fibre fracture, respectively, referring to the energy required to create two units of surface area in each case. By equating equations (1) and (8), the following energy balance equation is obtained:

$$E_f\epsilon^2\pi r_f^2\left(\frac{L_d}{2} + \frac{1}{\beta} - \frac{\beta E_f r_f^2}{16G_f}\right) = 2\pi r_f L_d \Gamma_i + \pi r_f^2 \Gamma_f \tag{9}$$

Solving for L_d gives:

$$L_d = \frac{2\sigma_f^2 r_f\left[\frac{1}{\beta} - \frac{\beta E_f r_f^2}{16G_f}\right] - 2E_f r_f \Gamma_f}{4E_f\Gamma_i - r_f\sigma_f^2} \tag{10}$$

where $\sigma_f = E_f\epsilon$ is the stress in the fibre at the moment of the fibre break.

Experimentally, a continuously monitored single-fibre fragmentation test[5] must be performed and, starting from the first fibre breaks, each fibre–matrix initial debonding length (L_d) (thus, the length at the nucleation stage of debonding) and its associated instantaneous fibre stress ($\sigma_f = \sigma_m E_f/E_m$, where σ_m is the matrix—or composite—applied stress) should be recorded within the linear region of the stress–strain trace. Subsequently, either: (1) a best-fit procedure to equation (10) may be done, if there are enough experimental points, with Γ_i left as a free and adjustable parameter; or (2) Γ_i can be calculated individually for each point by inverting equation (10), which is the preferred approach if an insufficient number of data is available.

Note that all parameters in equation (10) are relatively easy to measure experimentally, with the exception of R (which is included in β), the radius of the matrix cylinder. Recent micro-Raman spectroscopic measurements[3] as well as theoretical estimations suggest a value of R in the range $2r_f$ to $10r_f$, depending on the material properties of the composite system. The parameter R plays the role of a 'tuning' coefficient for the stress distribution, as it depends on the materials and geometrical properties of the composite components.

The above model offers a method for possible reinterpretation of the fragmentation test in terms of energies rather than of stress. It is, however, only an approximate model:

- a classical fracture mechanics analysis would involve the incremental, self-similar growth of a debond crack along the interface. The energy balance presented here compares energies before and after fracture of the fibre and the interface, and is not a fracture mechanics analysis;
- pre-existing stresses (such as residual thermal stresses due to sample preparation) are neglected;
- the debond region is assumed to be frictionless, although in most composites stable debonding most probably occurs with increasing load precisely because of the presence of such frictional forces; and
- dynamic effects related to fast fibre fracture, such as the energy dissipated as shock or acoustic waves, are ignored but may become important when thicker fibres (such as boron) are used in small dogbone-shaped specimens.

The relative importance of various physical parameters in the model is now examined.

DISCUSSION

Figure 2 reports the length of the initial debond around a fibre break in a fragmentation test, in terms of the stress in the matrix at the instant of break. The physical data used to produce the plot correspond to E-glass fibres in a typical glassy polymer such as epoxy. Not recalled in the plots are the values of Poisson's ratios of the fibre and the matrix, $\nu_f = 0.2$ and $\nu_m = 0.35$ respectively, and the fibre surface energy $\gamma_f = 5\,\mathrm{J\,m^{-2}}$. The basic notion arising from *Figure 2* is that if the fibre break occurs at relatively low stress, the initial debond will be shorter, and the

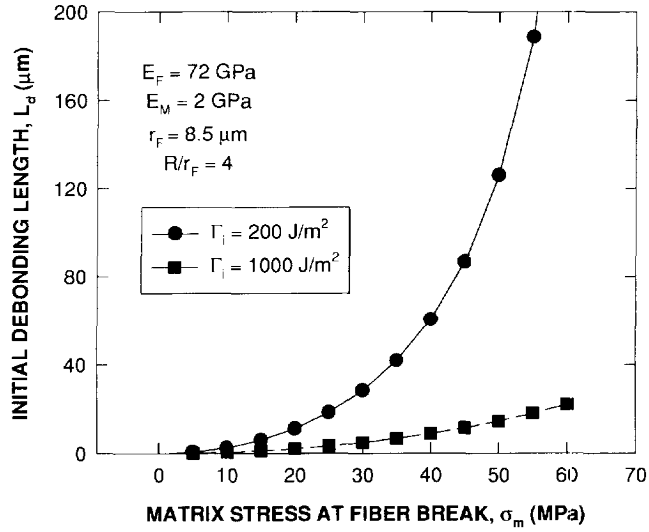

Figure 2 Effect of the level of matrix stress at the moment of fibre break upon the length of initial interface debond, for tough and weak interfaces, according to equation (10)

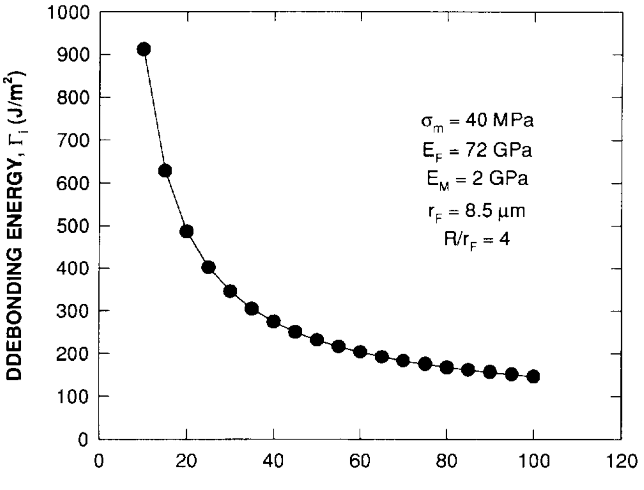

Figure 3 Dependence of the interfacial debonding energy on the initial debonding length, according to equation (10)

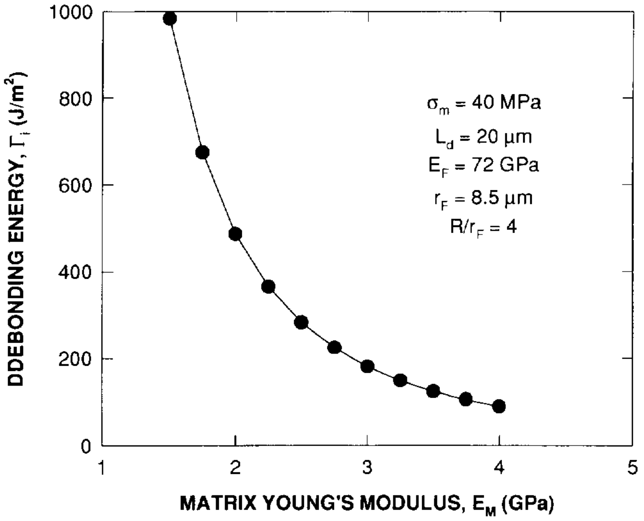

Figure 4 Dependence of the interfacial debonding energy on the matrix Young's modulus, according to equation (10)

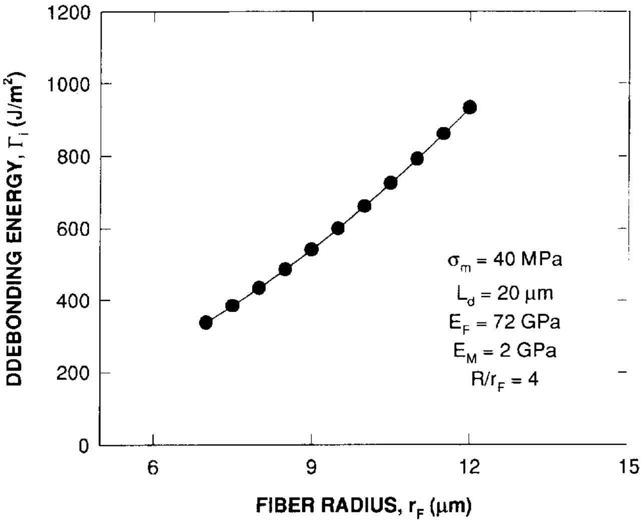

Figure 5 Dependence of the interfacial debonding energy on the fibre radius, according to equation (10)

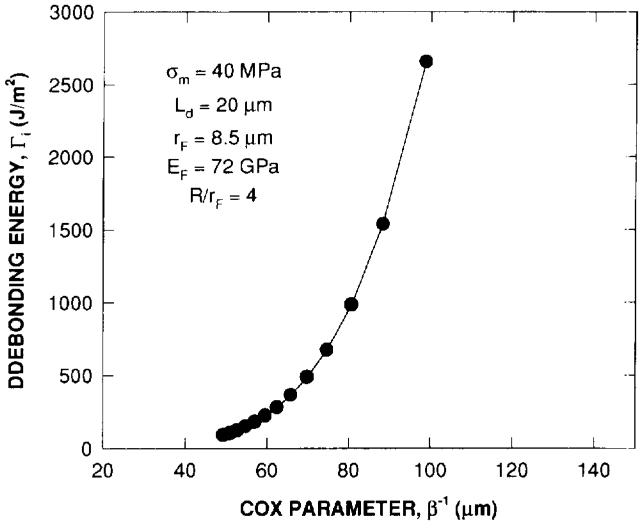

Figure 6 Dependence of the interfacial debonding energy on Cox's parameter β^{-1}, according to equation (10). The value of the Cox parameter was varied by changing the matrix Young's modulus

more so if the interface is inherently tough (for example. $\Gamma_i = 1000\,\mathrm{J\,m^{-2}}$), all other variables being constant. At higher stress, more energy is available, resulting in longer interfacial debonding, particularly if the interface is weak ($\Gamma_i = 200\,\mathrm{J\,m^{-2}}$).

Figures 3 to *7* present the effects of various physical parameters present in the model upon the interfacial energy for the nucleation of debonding, using the inverted form $\Gamma_i = f(L_d)$ of equation (10). Again, the physical data correspond to glass/epoxy composites. *Figure 3* shows that the shorter the initial debonding length at a given applied stress, the tougher the interface. Note that for very short debonds, interface energies tend to be very high and compete with the fracture energy of the matrix, resulting in the occurrence of the often observed 'cone-like' matrix cracks. Another important parameter is the Young's modulus of the matrix, which has been observed to vary from specimen to specimen[12]. If all other parameters are kept constant (including the length of the debond), our model predicts (*Figure 4*) that the energy necessary to initiate a specified debond length decreases rather sharply as the matrix modulus increases. Preliminary tests support this[12], thus providing additional confidence in the validity of the present theoretical model. This leads to the conclusion that when producing data to assess the interface toughness, it is critical to make sure that specimens with identical physical characteristics are used. This conclusion applies as well to the radius of the fibre used in the single-fibre composite test. Indeed, *Figure 5* shows that varying the fibre radius is predicted by our model to have a strong effect on the interface energy. This again is supported by preliminary fragmentation tests using sized E-glass/epoxy samples[12], in which the glass fibre diameter varies from about 14 to 20 µm. Again, individual fibre diameters must be accurately measured if interfacial tests are to be correctly interpreted.

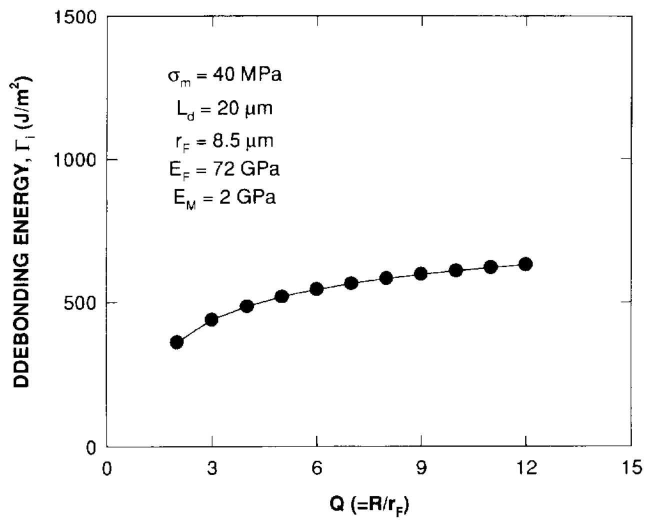

Figure 7 Dependence of the interfacial debonding energy on the ratio $Q = R/r_f$ appearing in Cox's parameter β^{-1}, according to equation (10)

Finally, it is of interest to examine the effect of the Cox parameter β on the value of the interfacial energy. In *Figure 6*, we have plotted the interfacial energy Γ_i against β^{-1}, the characteristic length of the stress profile calculated by Cox. As can be seen, small changes in the value of β^{-1} affect rather strongly the interface toughness, all other parameters being equal. There are two methods of affecting the value of β, i.e. by modifying either the matrix shear modulus G_m or the radius ratio $Q = R/r_f$. *Figure 6* was obtained by varying E_m between 1 and 4 GPa (or G_m between 0.4 and 1.5 GPa), and the effect is drastic. *Figure 7* was obtained by varying $Q = R/r_f$ between 2 and 12, and the effect is mild.

It is the belief of the present authors that the rather large variability in interface strength data obtained in earlier testing (see, for example, the exhaustive 1993 international round-robin tests[13]) is probably due to the lack of individual measurement (separately, for each specimen) of some of the relevant physical parameters (fibre radius, matrix modulus, radius ratio), and of an appropriate theoretical model to explain the source of that variability.

CONCLUSIONS

An energy-based interpretation of the single-fibre fragmentation technique may be utilized as a quantitative measurement of the interfacial toughness in composites that exhibit some degree of interfacial debonding. Measurements must be performed in the linear elastic region of the stress–strain trace, at low strain levels. This has the advantage that it is possible to assess the interfacial toughness of composite systems that never reach saturation in a fragmentation test. The present approach incorporates the debonding length as a necessary measurable parameter. We suggest that the commonly observed variability in interface strength data is probably due to the lack of individual measurement (separately, for each specimen) of some of the relevant physical parameters (fibre radius, matrix modulus, radius ratio), and of an appropriate theoretical model to explain the source of that variability. This is especially true in view of the relatively poor quality (spreading non-uniformity, thickness variability, etc.) of the commercially available fibre sizing agents, which leads to variability in surface and geometrical properties along individual filaments, and between filaments. Many more experimental data need to be generated to verify the extent of validity of the model, and the relative roles of the physical parameters.

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