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Review
Single fibre fragmentation test for assessing adhesion in fibre reinforced composites

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The single fibre fragmentation test for measuring the properties of the fibre–matrix interface in fibre-reinforced composites is reviewed. Special emphasis has been paid to the recent stress transfer models in single fibre composites and its application to the development of a suitable data reduction technique for the fragmentation test. The complexities of the correlation of the micromechanical results to the properties of the macrocomposites have been highlighted.

1. Introduction

Fibre-reinforced composites of superior mechanical properties are made by combining fibres into matrix material of similar/dissimilar nature in various ways, whereby interfaces are inevitably created. In composites, both fibre and matrix retain their physical and chemical identities, yet they produce a combination of mechanical properties that cannot be achieved with either of the constituents acting alone. For example, fibres have very high strength and modulus but are developed only as very fine fibres. Plastics have considerable resistance to chemical environments. By combining fibres and resin, a material (composite) can be produced which has the strength and stiffness close to that of the fibres and with the chemical resistance of the plastic. To achieve an outstanding performance, these fibre-reinforced composites need to exhibit optimum adhesion between the fibres and the matrices. To promote the fibre–matrix adhesion, different surface treatments/coupling agents are applied to the fibre surfaces. Silane type coupling agents are normally applied to glass fibre surfaces during manufacture [1] while carbon fibres are usually surface treated oxidatively [2] and aramid fibres are usually treated with epoxy finish [3].

The complexity of the interface is further increased by the use of sizing agents which are applied to the fibre surface to protect the fibre surface from damage and to bind the fibres together for ease of processing [4–7]. Hence, an interface can be more correctly termed an interphase where it represents an interfacial region of finite volume where the material properties vary either continuously or in a stepwise manner between those of the bulk fibre and matrix material [8]. Fibre–matrix adhesion can occur through five basic mechanisms: adsorption and wetting; mechanical interlocking; interdiffusion; dipolar and/or ionic interactions (e.g. acid–base); and covalent bonding [9, 10]. It is believed that chemical bonding will lead to strong and environmentally stable interfaces. Because the properties of the interface, which are governed largely by the chemical/morphological nature and physical/thermodynamic compatibility between the two constituents, most often limit the overall performance of the composite, a thorough knowledge of the microstructure–property relationship at the interface region is an essential key to the successful design and proper use of composite materials. A number of experimental techniques have been developed to characterize the fibre–matrix adhesion or interfacial properties in fibre-reinforced composites. The test methods using microcomposites include single fibre fragmentation, fibre pull-out, indentation and micro-debonding of a droplet [11].

The single fragmentation technique is reviewed in this paper because it represents a convenient and reproducible test method where the fibre–matrix interface is subjected to pure shear. However, its use is limited by the lack of a data reduction methodology for an interfacial parameter which can be correlated to changes in fibre surface chemistry and overall mechanical properties of a composite. A further complication is the presence of sizing resins on the commercial fibres which modify the matrix resin within the stress transfer region. This review attempts to address these concerns. In particular, consideration is given to the relative merits of quoting an interfacial shear strength (τ) when under the conditions of strong fibre–matrix adhesion, yielding within the matrix or interphase region is being examined. A brief mention is made to the correlation of single fibre fragmentation test results to the mechanical properties of high fibre volume fraction composites in order to put the current status of fragmentation test for the measurement of interfacial properties in perspective.

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2. Single fibre fragmentation test

The fragmentation test is now widely used for measuring the effect of different surface treatments on the interfacial shear strength of glass and carbon fibres because of its simplicity in specimen preparation, ease of testing and wealth of information obtained in terms of damage processes [4–8]. When an external stress is applied to a single fibre embedded in a matrix, the tensile stress is transferred to the fibre through an interfacial shear stress. As the tensile load increases, the tensile strain in the fibre will eventually exceed the failure strain of the fibre, and the fibre will fracture. The fibre continues to fracture into shorter lengths as the load increases, until the fragment length becomes too short to break, as illustrated in Fig. 1. This situation is defined as the saturation in the fibre fragmentation process. The shortest fragment length which can break on application of stress is defined as the critical fibre length. Due to the statistical nature of fibre strength, a single fibre does not break into the fragments of equal size and a wide variation in fragment lengths is observed. The fragment lengths for transparent matrix composites can be measured using a conventional optical microscope. An average interfacial shear strength at the interface τ can be estimated on the basis of the constant shear model proposed by Kelly and Tyson in 1965 [12]. It has been widely assumed that at saturation all the fragments are debonded/yielded providing for constant shear at the interface so that the following analysis can be employed

$$\tau = \frac{\sigma_{fu} d}{2 l_c} \quad (1)$$

where d is the fibre diameter and σ_{fu} is the fibre strength at a length equal to the critical fibre length l_c. Early work by Ohsawa *et al.* (1978) provided the background for the semi-empirical analysis of the test data [13]. The critical fibre length is calculated by

$$l_c = \frac{4}{3} \bar{l} \quad (2)$$

where $\bar{l}$ is the average fragment length.

Adding the photoelastic technique to the optical microscopy under polarized light allows the spatial distribution of stresses to be evaluated in the matrix around the fibre and near its broken end. The acoustic emission technique is also becoming increasingly popular for non-transparent matrix materials particularly for metal and ceramic matrix composites [14, 15] which allows calculation of number of fragments based on acoustic measurements. The popularity of the fragmentation technique results from the fact that data can be collected over a large area of interface and the process itself replicates the *in-situ* events in an actual composite material to a certain extent. In a variation, the single fibre fragmentation specimen loaded under tension perpendicular to the fibre direction has been used for measuring the compressive strength of the fibre [16]. Poisson's ratio-induced compressive strain causes multiple fracture of the fibre which can be used to calculate compressive strength of the fibre using Weibull statistics. Berglund and Varna have extended this to measure the growth of a debond at a fragment end [17].

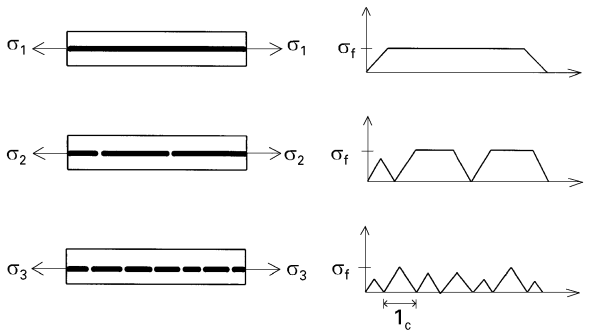

Figure 1 A schematic drawing of the fragmentation test.

2.1. Critical study of the fragmentation test

The use of the constant shear model to calculate the value of interfacial shear strength from the fragmentation test requires the input of fibre strength at the critical fibre length (Equation 1). The prediction of the fibre strength at the critical fibre length is normally done in air or *ex-situ* which itself does not replicate the situation in the fragmentation test specimen where the fibre is enclosed in a matrix. The fibre strength is a function of the gauge length and is controlled by the flaw size distribution present within the fibre. The extrapolation of the fibre strength from the gauge length to the critical fibre length requires some form of statistical analysis. A review of the existing theories to calculate the Weibull statistics from the experimental data is given in References 18 and 19. The extrapolation of fibre strength results at the gauge length to the fibre strength at the critical fibre length remains highly controversial. Many researchers have used three or four gauge lengths for extrapolation of the fibre strength results [20, 21]. In addition, the statistical strength of a fibre may change upon processing, and so the Weibull parameters determined *ex-situ* may be irrelevant for describing fibre performance *in-situ*. Several authors have presented models to calculate the fibre strength *in-situ* during the fragmentation test along with the interfacial shear strength measurement [22–26].

As the interest in the measurement of interfacial shear strength from the fragmentation test has increased, several attempts have been made towards the development of sophisticated statistical techniques to characterize the fibre fragment length distribution as well as the relationship between the length and tensile strength of the fibre. The relationship of Ohsawa *et al.* (Equation 2) is based on the fact that the fibre fragment length distribution is symmetrical. However, such a distribution of fragment lengths uniformly distributed around $\bar{l}$ and limited by l_c and $l_c/2$ is seldom observed. Some authors do not accept this relationship [24, 27]. Drzal *et al.* [27] have demonstrated that the fibre fragment length distribution, at saturation, can be fitted with a two-parameter Weibull distribution. α is the shape parameter and β, the scale parameter. These parameters do not correspond to those of the fibre

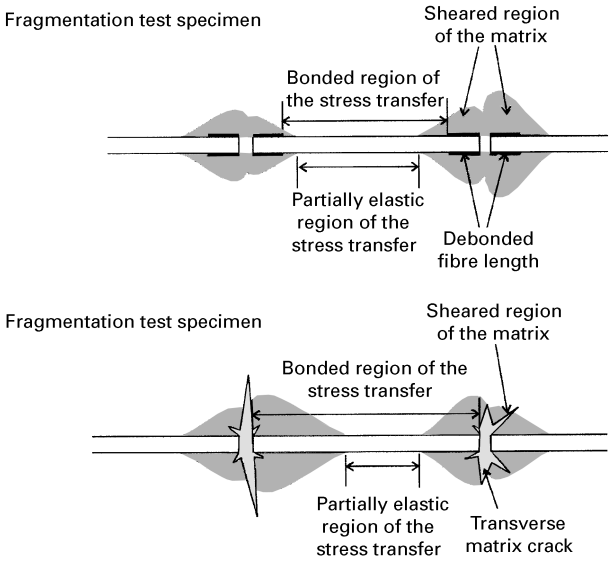

Figure 2 Different micromechanical damage events and stress transfer regions observed during the fragmentation test [65].

tensile strength. According to Drzal *et al.* [27]

$$l_c^* = \frac{\beta d}{\Gamma\left(1 - \frac{1}{\alpha}\right)} \quad (3)$$

where l_c^* is the most probable fragment length and Γ is the gamma function. Henstenburg and Phoenix [24] have proposed a modified version of the Kelly–Tyson equation (Equation 1) as follows

$$l_c = \left(\frac{2}{\Lambda}\right)^{1+\frac{1}{m}} \bar{l} \quad (4)$$

where Λ is a parameter depending on the fibre Weibull modulus m.

However, the basic form of the relationship between the critical fibre length and the shear strength of the interface remains virtually unchanged from the original relationship given by Kelly and Tyson [12]. In reality, the fragmentation test is a very complex single embedded fibre test and several micromechanical phenomena observed in the real-life composites other than fibre fracture are also observed during the fragmentation test. Shear yielding of the matrix, interfacial debonding and transverse matrix cracking have been widely reported (Fig. 2) [20, 28]. In fact, the occurrence of these damage events during the fragmentation test makes the conventional data reduction technique based on the constant shear model invalid. The limitations of the constant shear model as well as data reduction techniques for the fragmentation test based on the constant shear model have been the subject of several studies [20, 28–31] and some of the major reasons are discussed above. At this point, it will suffice to say that the use of the constant shear model to calculate interfacial shear strength from the fragmentation test is highly inaccurate.

The validity of the constant shear model for the calculation of interfacial shear strength from the fragmentation test data requires that saturation in the fibre fragmentation process has occurred. It is widely assumed that the strain required for the saturation is at least three times the fibre failure strain [20, 30–33]. This means that the fragmentation test cannot be used with the matrices of low failure strains. A technique has been devised to circumvent this problem; in that a thin layer of the brittle matrix material is coated onto the fibre which is subsequently embedded in a ductile resin. This particular variation of the test specimen preparation is called the bimatrix fragmentation test [33–36]. Further, it has been reported that the strain required for the saturation of the fibre fragmentation process depends on the yield strain of the matrix rather than the fibre failure strain. A matrix with low yield strain and preferably with cold draw yielding will help to achieve saturation at a lower applied strain during the fibre fragmentation process, especially for glass and Kevlar fibres [37, 38].

The value of the interfacial shear strength obtained from the fragmentation test depends on the quality of the interface as well as the matrix properties [23, 39, 40]. At present, the square root relationship between the interfacial shear strength and the elastic modulus of the matrix (derived from the shear lag model [41] or finite difference method [42, 43]) is commonly used for the normalization of interfacial shear strength values obtained from the fragmentation test of a single embedded filament in different supporting matrices [23, 39, 40], as given below

$$\tau \propto \left[\frac{E_m}{E_f}\right]^{1/2} \quad \text{or} \quad l_c \propto \left[\frac{E_f}{E_m}\right]^{1/2} \quad (5)$$

where E_m and E_f are Young's moduli of fibre and matrix respectively. However, this relationship only considers the elastic properties of different matrices while the conventional data reduction technique assumes constant shear at the fibre–matrix interface (either through a perfectly plastic matrix or friction caused by interfacial debonding). Thus, the assumptions for calculating and for normalizing the value of interfacial shear strength from the fragmentation test data are contradictory. A new method for the normalization of fragmentation test results which is based on the effect of matrix plasticity (tensile yield strength and tensile yield strain) on the stress fields associated with a short embedded fibre, of the form given below has been proposed [44, 45].

$$\tau_2 = \tau_1 + \left(\frac{\sigma_{y_2} - \sigma_{y_1}}{\sqrt{3}}\right) + f(\Delta\varepsilon_y) \quad (6)$$

where τ_1 is the interfacial shear strength of a fibre embedded in a resin of tensile yield strength σ_{y_1} and τ_2 is the interfacial shear strength of the same fibre embedded in another resin of tensile yield strength of σ_{y_2}. Further, $f(\Delta\varepsilon_y)$ is an unknown function of the difference in yield strains of the two matrices.

In a significant development over the data reduction technique based on the constant shear model, Lacroix *et al.* [21, 46] developed a simulation for the fragmentation test based on the partial debonding model of Piggott [47, 48]. The mechanical properties of the interface are characterized by two independent parameters; the interface bond strength, τ_{deb} and

interface friction resistance, τ_{fri}. The simulation predicts the evaluation of the fibre fragment aspect ratio and debonding ratio as a function of applied strain. The derived interface properties, τ_{deb} and τ_{fri}, are then computed such as to obtain the best possible agreement between the experimental and simulated results for the fragment aspect ratio and debonding ratio. In a variation of the above simulation process, Favre and co-workers [20, 28, 32] used the interface debond strength derived from pull-out testing and the appropriate value of fibre–matrix friction coefficient. This computer simulation predicted the critical aspect ratio with good accuracy. However, both of these techniques, which are based on the partial debonding model, have the same inherent limitations as the partial debonding and the shear lag models [41, 47]. These limitations are discussed elsewhere in detail [29, 31, 49].

Because of limitations in the conventional data reduction techniques, the results from the fragmentation test cannot be used with confidence for the selection of suitable surface treatments for either glass, aramid or carbon fibres [50]. A clear understanding of the damage and failure processes associated with micro- and macrocomposites is necessary for the design of optimum interfaces for strong and reliable fibre reinforced composites. Hence, the critical requirements of an accurate data reduction technique for the fragmentation test will be:

1. An accurate stress transfer model for a single fibre composite which takes materials properties and different damage events during the fragmentation test into account.
2. An interface characterization parameter which may be based on the interfacial shear strength, fracture energy for crack growth along the interface or the efficiency of stress transfer between matrix and fibre.

3. Stress transfer in a single fibre composite

In the fragmentation test once the single embedded filament has fractured, the factor which determines the interfacial quality is (a) the rate of reloading the broken fragment and (b) the maximum stress transferred to a fragment. When the latter reaches the strength of that fragment, fracture occurs. Saturation occurs when the fragment cannot be reloaded sufficiently to fracture. Therefore, the primary requirement for an accurate data reduction technique for the fragmentation test is an accurate stress transfer model for the prediction of stresses in a single fibre composite. The first attempt to predict the stresses in a single fibre composite was made by Cox, known as the shear lag model [41]. In this model, it is assumed that both the fibre and matrix are elastic and perfectly bonded. Because of the large difference in moduli of components, stress transfer occurs through shear at the interface. This model was later modified by Piggott to include interfacial debonding at the fibre–matrix interface [47, 48]. These models have been widely discussed and used in interfacial studies. The major limitations of the one-dimensional shear lag type stress transfer model are that the interfacial shear stress is maximized at the broken fibre end and the improper consideration of the non-linear behaviour of the matrix [29, 31, 37, 49]. Stress transfer studies involving photoelasticity, laser Raman spectroscopy and finite element analysis have highlighted these limitations in the prediction of the stress profile in the fragments in the single fibre fragmentation test specimen (Sections 3.1–3.3). Of particular relevance is the exponential decay in interfacial shear stress from the fibre end so it is often assumed that the maximum value can be identified with as interfacial shear strength. However, at saturation in the fragmentation process, it is assumed that the fragments are completely debonded/yielded providing for a constant shear stress at the interface (i.e. an interfacial shear strength). This is not always observed, so that the final fragment length distribution is a function of a more complex stress transfer function. Thus a better model of the stress transfer process is needed. Therefore, we will concentrate on recent stress transfer models only.

3.1. Photoelasticity

A leading technique for the study of the stress fields around a single fibre filament embedded in a birefringent matrix is photoelastic analysis. Tyson *et al.* [51] demonstrated that the magnitude of the peak shear stresses was significantly larger than that predicted by the shear lag analysis [41]. However, divergence from the theoretical values was only marked over a distance from the fibre-end equivalent to two fibre diameters because of stress concentrations at the sharp corners of the fibre-ends. Later, a more detailed photoelastic analysis by Allison and Hollaway [52] showed two interesting features of the stress distribution around a blunt fibre-end composite:

1. The interfacial shear stress is zero at the fibre-end and reaches a maximum value at a small distance from the fibre-end, before finally decaying to zero towards the middle of the fibre length. This observation confirms the inaccuracy of one of the major assumptions of the shear lag analysis that the shear stress is maximized at the fibre-end.
2. The principal stress in the above mentioned composite was at a maximum at a point near the fibre-end at the fibre–matrix interface and at a point near the fibre-end cross-section in the matrix.

3.2. Laser Raman spectroscopy

Over the last few years, laser Raman spectroscopy (LRS) in combination with mechanical measurements has been used to study the micromechanics of discontinuous fibre composites by measuring the tensile strain distribution along a reinforcing fibre. This technique employs a laser Raman microprobe to measure the strain in an individual fibre at the microscopic level. This technique was first used to study a model polydiacetylene fibre composite [53]. The shift in

strain dependent Raman bands of high performance fibres in air is used to measure the deformation of a fibre embedded in resin matrix. The Raman technique is now commonly used to monitor the strain profiles in carbon [30, 54–56] and Kevlar fibres [57–59] embedded in epoxy resin matrix. Recently, laser Raman spectroscopy technique has been extended to glass fibre composites which was not possible previously due to the amorphous nature of glass fibre [60].

Studies of the deformation of the reinforcing fibres in an epoxy resin matrix have shown that the strain distribution along the fibre is in qualitative agreement with the shear lag model at low matrix strains [54, 61]. The fibre strain measured by LRS along the individual fibre is converted into an interfacial shear stress (ISS) profile using a simple balance of forces argument. A comparison of the value of interfacial shear strength obtained from the fragmentation test using the conventional data reduction technique (Kelly–Tyson model) and the maximum shear stress at the fibre–matrix interface measured using LRS has shown that the interfacial shear strength prediction from the fragmentation test using the constant shear model is lower than the maximum ISS inferred from LRS measurements by a factor of at least two [30]. Furthermore, it was shown that the basic assumption of the Kelly–Tyson model that the interfacial shear stress is constant over the fragment length in the fragmentation test specimen is fundamentally wrong even at high values of the applied strain [30]. The stress profile in a single embedded fibre composite depends on the fibre and matrix properties and the quality of the interface.

The effect of the fibre surface treatment on the strain profile in a short embedded fibre was monitored using laser Raman spectroscopy [62]. The maximum interfacial shear stress was observed to be considerably higher for the surface treated fibres compared to the untreated ones. Some of the important conclusions from laser Raman spectroscopy of the carbon fibre/epoxy composites are:

1. The value of the interfacial shear stress increases with applied strain despite the presence of interfacial debonding [30].
2. The length of interfacial debonding at the point of the fibre fracture is driven by the strain energy released by the fractured fragments [30].
3. Debonding accompanies fibre fracture even at low applied strains [30].
4. After a certain value of applied strain, further fibre fractures do not occur and the strains in the fragments starts to decrease [30, 56].
5. The fibre strain at very high applied strains is much lower than that at lower applied strains [30, 56].
6. The interfacial shear stress increases with applied strain and then decreases to a plateau value [54].
7. At low applied strains, the interfacial shear stress is maximum at the fibre-end. As the applied strain increases, the maxima start shifting inwards along the fibre fragment [54, 56].
8. A typical stress profile in an embedded fibre consists of three regions; (a) a region of the ISS fluctuations around a constant quotient, (b) an area of ISS built-up to a maximum value and (c) a region of ISS decay to zero value (Fig. 3) [54].
9. At higher applied strains, the three regions of the stress transfer can be identified in all the cases, regardless of whether these regions are activated from a fibre-end or a fibre break [54].

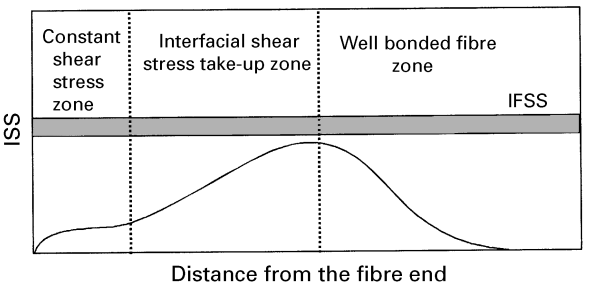

Figure 3 Three zones of stress transfer observed during laser Raman spectroscopy of single fibre composites [54].

Most commercial grades of aramid fibres are treated with a sizing resin to improve their handling characteristics. The effect of a proprietary epoxy resin based fibre surface treatment in a Kevlar fibre/epoxy resin system was investigated by Andrews *et al.* [61]. The important conclusions were as follows:

1. At low applied strains, the distribution of the stress or strain along the fibre appeared to be defined using the classical shear lag theory. As can be seen in Fig. 4, this is a consequence of the resolution of the technique. Indeed at these low strains, the maximum shear stress occurs very close to the end of the fibre.
2. The maximum value of interfacial shear stress was higher for the pretreated fibres. This was attributed to a change in the matrix properties in the vicinity of the fibre/matrix region. The deformation which occurred around the fibre fracture was predominantly shear yielding of the matrix. The stress transfer mechanics of this composite could be described by the shear lag analysis up to a higher value of applied strain in comparison to the untreated fibre.
3. In the case of untreated fibres, interfacial failure was initiated by shear yielding of the matrix followed by debonding at the fibre-end.

Several studies have compared the measured fibre strains from laser Raman spectroscopy with the estimates of interfacial shear stress obtained from the prediction of finite element analysis and a close agreement between the two techniques has been shown [63, 64]. Some of the above mentioned observations cannot be explained on the basis of elastic theories of stress transfer in single fibre composites. However, an elasto–plastic stress transfer model can explain some of these observations [65].

3.3. Finite element analysis

The first attempt to use finite element analysis to predict the stress fields in a single fibre composite was

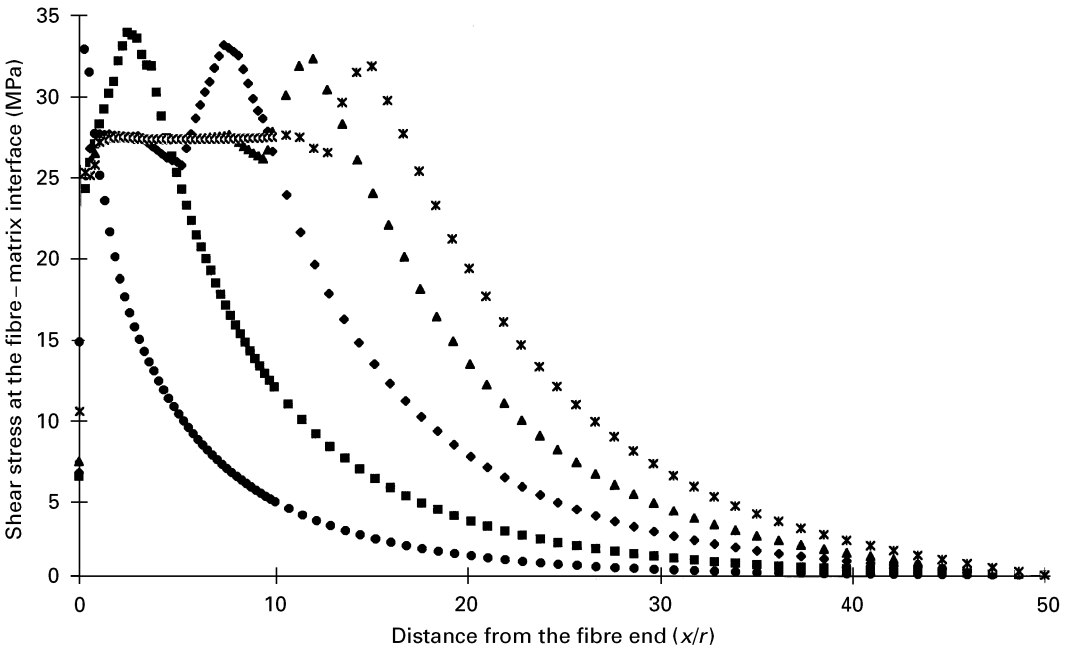

Figure 4 Shear stress at the fibre–matrix interface at different applied strains predicted by finite element (FE) method [37]. Strain: (●) 0.6%; (■) 1.2%; (◆) 1.8%; (▲) 2.4%; (✳) 3.0%.

made by Carrara *et al.* [66] in 1968. The effect of stress concentration at the fibre-end and stress transfer across the fibre-end cross-section was demonstrated. A good agreement with the shear lag model away from the end of the fibre was shown. However, the shear stress at the fibre-end was twice that predicted by the shear lag model because of a stress concentration or a singularity in the model which lead to an inaccuracy. This analysis considered only elastic deformation of the matrix and was further limited by the computing power available at that time [66].

Guild *et al.* [64] compared the strain profiles from the laser Raman technique of a short Kevlar fibre embedded in an epoxy resin matrix with the predictions from an elasto–plastic finite element model. The experimental tensile stress/strain curve was used for the modelling of the non-linear behaviour of the matrix. A good agreement between the two predictions was shown. However, discrepancies were observed at the end of the fibre. The finite element (FE) predictions were dominated by the presence of a singularity at the fibre-end. The other important conclusion from this study was that the interfacial shear stress is maximized at a point very near to the fibre-end (contrary to the shear lag model which predicts that the interfacial shear stress is maximized at the end of the fibre and increases with applied strain). A typical value of the interfacial shear stress at the plateau for a short single fibre reinforced Kevlar/epoxy system was 37 MPa.

Using an axisymmetric finite element model, Tripathi *et al.* [37] concluded that the stress profile in and around a fibre is strongly influenced by the plastic or non-linear behaviour of the matrix. Shear yielding of the matrix occurs near the end of the fibre at very low applied strains. Two critical regions of interfacial shear stress corresponding to the matrix tensile yield and cold draw strengths are observed (Fig. 4). It is possible to theoretically calculate the matrix shear yield strength from the matrix tensile yield strength and predict the maximum possible shear stress at the interface in the case of a perfect interface, assuming the matrix at the interface experiences conditions of pure shear. The maximum shear stress at the interface is limited only to a very small portion of the fibre which is not the fibre-end. However, at higher applied strains, a major portion of the fibre is subjected to a plateau shear stress which is lower than the maximum interfacial shear stress and is given by $3^{-1/2}$ times the cold draw strength of the matrix, assuming the von Mises yield criterion. A significant region of a fibre fragment will be subjected to the plateau interfacial shear stress at the last stages of the fragmentation test because of the high applied strains. However, the shear stress profile even at high applied strains significantly deviates from that predicted using the constant shear model, particularly in the mid-region of the fibre fragment (Fig. 4). Interfacial debonding, if observed, causes further deviation from the idealized situation predicted by the constant shear model.

The effect of matrix elastic modulus, tensile yield strength (or cold draw strength in the case where it is less than matrix yield strength) and tensile yield strain on the fragmentation test has been investigated using finite element analysis [37]. The elastic modulus of the matrix controls the rate of development of tensile stress from the fibre-end but the maximum tensile stress at the middle of the fibre length is dependent on the matrix yield strain. Furthermore, the use of a matrix of low yield strength and/or high yield strain for the fragmentation test can lead to an incorrect estimation of the interfacial shear strength by the constant shear model. In certain cases, the predicted value of interfacial shear strength from the fragmentation test data can exceed the shear yield strength of the matrix

which illustrates the inadequacies of the constant shear model for estimating fibre–matrix adhesion. It is argued that in certain cases where the researchers have proposed the presence of a ductile or a stiffer interphase, it is possible to explain the results, completely or partially, on the basis of matrix plasticity. Furthermore, it is shown that the interfacial shear strength is dependent on the matrix yield strength rather than the matrix modulus [37].

The value of interfacial shear strength obtained from the fragmentation of a single fibre composite with a poor interface (water-sized, uncoupled fibre) calculated using the conventional data reduction technique based on the constant shear model is higher than the maximum interfacial shear stress predicted by finite element analysis [37] which assumes a perfect interface. This raises serious reservations about the conventional data reduction techniques for the fragmentation test. Although the finite element model employed assumed a perfect interface, incorporation of a debonded region is a complex issue. Nedele and Wisnom [67] have attempted to simplify the problem of a poor interface in a two-zone stress transfer problem using a so-called debonded and a bonded perfect region. However, a rigorous analysis will require the relaxation of the assumption of perfect stress transfer over the entire interface.

The stress transfer in single fibre composites was examined by Termonia using a finite difference approach [42]. A single fibre embedded in an infinite three-dimensional matrix was considered to evaluate the effect of fibre-to-matrix modulus ratio and adhesion parameter on the stress transfer. An elastic stress analysis was performed. The importance of the end-adhesion, neglected by Cox [41] and Dow [28], was demonstrated in the total stress transfer to the reinforcing fibre. Although, in the fragmentation process, the fibre is continuously broken and end-adhesion is not important, an isolated fibre behaves more or less the same as a fragment except for a localized region near the fibre-end [35, 36, 54]. The adhesion factor was modelled by breaking bonds at the fibre-matrix interface. A decrease in the adhesion was observed to increase the critical fibre length, particularly when the adhesion is less than 30% of the perfect adhesion.

Finite element analysis has also been used for validation of the data reduction technique based on the constant shear model for the measurement of interfacial shear strength of glass fibre phenolic composites using the bimatrix fragmentation test [35, 36]. It is shown that the inclusion of a thin coating of the phenolic resin on the fibre surface in the fragmentation test specimen does not cause any additional limitation to the use of the constant shear model for the bimatrix fragmentation test.

3.4. The improved stress transfer models

3.4.1. The axisymmetric model

More sophisticated models have been put forward by considering the longitudinal as well as lateral displacements in the fibre–matrix system. The model proposed by Whitney and Drzal in 1987 [49] is based on the superposition of an exact far-field solution and an approximate local transient solution. The stress is represented by a decaying exponential function multiplied by a polynomial. Equilibrium conditions and the boundary conditions of classical theory of elasticity are exactly satisfied. The far-field solution away from the broken fibre-end exactly satisfies all the equations of elasticity. The model includes the effects of expansional strains as a result of moisture and temperature. The model considers the orthotropic behaviour of the fibre.

The axial normal stress in the fibre is given by

$$\sigma_f = \left[1 - \left(4.75\left(\frac{x}{l_c}\right) - 1\right)e^{-4.75(x/l_c)}\right]A_1\varepsilon_c \quad (7)$$

Interfacial shear and radial stresses are as follows

$$\tau = -4.75\,\mu A_1\varepsilon_c\left(\frac{x}{l_c}\right)e^{-4.75(x/l_c)} \quad (8)$$

$$\sigma_{rr} = \left\{A_2 + \mu^2 A_1\left[4.75\left(\frac{x}{l_c}\right) - 1\right]e^{-4.75(x/l_c)}\right\}\varepsilon_c \quad (9)$$

where A_1, A_2, and μ are constants based on the material and geometry of the test specimen. According to the axisymmetric model, the interfacial shear stress is not maximized at the end of the fibre as given by the classical shear lag analysis of Cox [41]. This is because of the fact that the free end boundary condition which requires τ to vanish on the broken end of the fibre and is exactly satisfied in the axisymmetric model [49].

3.4.2. The variational model

In another model presented by Nairn in 1992 [31], the axisymmetric stress fields in the fibre as well as the matrix were resolved using variational mechanics. The analysis begins with an admissible stress state that obeys equilibrium and traction boundary conditions exactly. The solution considers the entire fibre length and thus accounts for the interaction of fibre breaks. The variational mechanics includes the residual thermal stress and, therefore, can be used to study the effect of radial compressive stress on the interfacial shear stress. However, the analysis assumes a perfect interface and, the matrix and fibre behave elastically. The variational mechanics model is also applicable for orthotropic fibres.

According to the variational method, the stress state in the fibre can be represented as

$$[\sigma_f] = [B_1][\Psi] \quad (10)$$

where

$$[\sigma_f] = (\sigma_{rr}, \sigma_{\theta\theta}, \sigma_{zz}, \tau_{rz}) \quad (11)$$

$$[\Psi] = [\sigma(\rho), T, \Psi, \Psi', \Psi'', \sigma_\infty] \quad (12)$$

and

$$[B_1] = \begin{bmatrix} -\frac{V_m A_4}{V_f A_0} & -\frac{V_m A_5}{V_f A_0} & -\frac{V_m A_3}{V_f A_0} & 0 & f_1 & V_f\left(1-\frac{V_m A_2}{V_f A_0}\right) \\ -\frac{V_m A_4}{V_f A_0} & -\frac{V_m A_5}{V_f A_0} & -\frac{V_m A_3}{V_f A_0} & 0 & f_2 & V_f\left(1-\frac{V_m A_2}{V_f A_0}\right) \\ 0 & 0 & 1 & 0 & 0 & 0 \\ 0 & 0 & 0 & -\frac{1}{2}\xi & 0 & 0 \end{bmatrix} \quad (13)$$

where σ_{zz}, σ_{rr}, $\sigma_{\theta\theta}$ and τ_{rz} are the tensile stress in the fibre and radial, hoop and shear stresses at the fibre–matrix interface respectively. T is the cure temperature and the other unknowns in Equations 12 and 13 are the complex function of material, geometry and stress state parameters of the test specimen. Similarly the stress state in the matrix can be represented in a matrix form.

3.4.2.1. Limitations. The finite element predictions for a short embedded fibre have been compared to those from the shear lag model of Cox [41], the axisymmetric model of Whitney and Drzal [49], and the variational model of Nairn [31]. The superiority of the variational model of Nairn over the other stress transfer models was observed (Fig. 5). However, Nairn compared the predictions from the variational mechanics and the finite element analysis at very low applied stress and showed good agreement between the two predictions. Since both the fibre and the matrix behave elastically at very low strains, a good agreement could be understood. However, the behaviour of the matrix during the fragmentation test at high strains ($> 8\%$) is essentially plastic. At these strains, the mechanical behaviour of the matrix severely deviates from linear elastic behaviour and cannot be accounted for in the variational model. This is one of the serious limitations of the variational model. Furthermore, the variational model cannot predict the shear stress transfer across the interface due to frictional forces and does not take into account an imperfect interface. Furthermore, in the variational model of Nairn [31], two of the four compatibility equations are not exactly satisfied [28, 37]. Hence, the stress field solution around a fibre fragment embedded in the matrix requires the volume of the near-field matrix cylinder, or in other words, the area of influence of the fibre on the matrix. The area of influence is difficult to measure. However, a typical value of area of influence or R/r for glass fibre/epoxy system obtained from the birefringence has been reported as 10–15 [28]. Tripathi *et al.* [37], using a finite element model, have shown that the above value for the area of influence is valid only at low applied strains. At higher applied strains, the prediction of the area of influence is very complicated because of localized shear yield of the matrix at the fibre-end and global plasticity effects

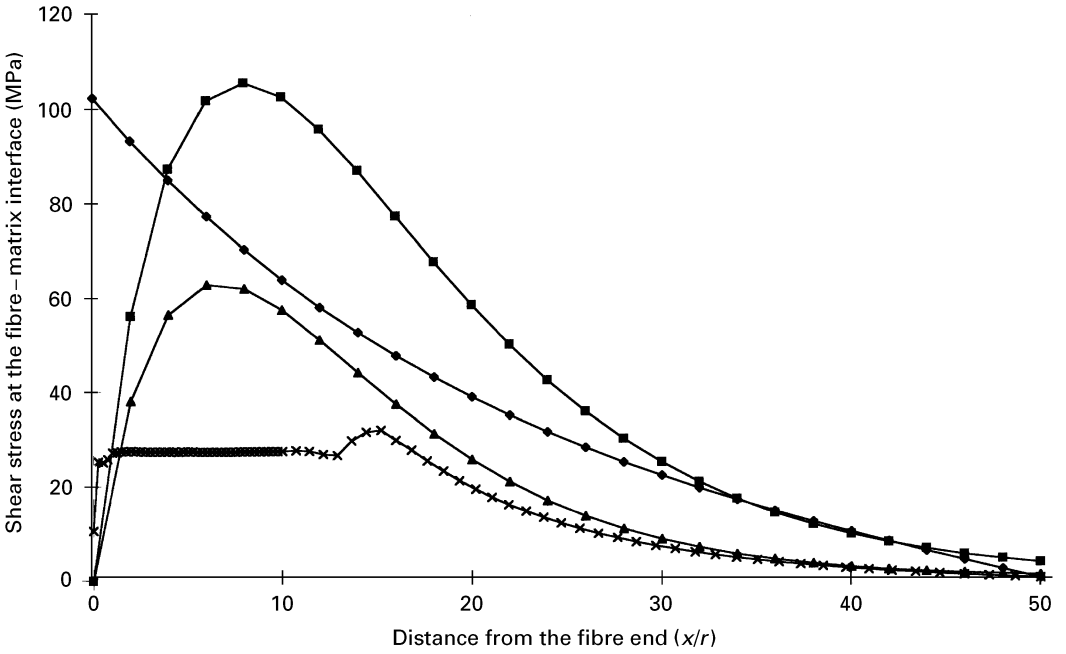

Figure 5 Comparison of shear stress at the fibre–matrix interface by different micromechanical models at 3% applied strain [37]. (◆) shear lag model; (■) axisymmetric model; (▲) variational model; (×) FE model.

[37]. Furthermore, the stress induced by thermal contraction on the fibre can be overestimated by the use of the cure temperature (T). In fact the relationship between the unknown stress free temperature, matrix glass transition temperature and cure temperature needs to be addressed.

3.4.3. Bessel function approach

Nairn *et al.* [68] have recently proposed a new stress analysis model for the fragmentation test using a Bessel–Fourier series stress function with some additional polynomial terms. The solution satisfies equilibrium and compatibility at every point in the model and satisfies most boundary conditions exactly. The only approximation is that the axial stress in the fibre at a fibre break is taken to have an average value of zero instead of zero at every point. The shear stress at the fibre–matrix interface and tensile stress in the fibre can be given as follows

$$\tau_{rz,1} = \sum_{i=1}^{\infty} \sin k_i\zeta \left[b_{1i}\left(\frac{1}{s_1^2} - a\right)\frac{I_1(\beta_{1i}\xi)}{s_1} + b_{2i}\left(\frac{1}{s_2^2} - a\right)\frac{I_1(\beta_{2i}\xi)}{s_2}\right] \quad (14)$$

$$\sigma_{zz,1} = B_2 + B_3 d\xi^2 + \sum_{i=1}^{\infty} \cos k_i\xi \left[b_{1i}\left(\frac{c}{s_1^2} - d\right) I_0(\beta_{1i}\xi) + b_{2i}\left(\frac{c}{s_2^2} - d\right) I_0(\beta_{2i}\xi)\right] \quad (15)$$

where I_0 and I_1 are the modified Bessel functions of the first kind. The other parameters in Equations 14 and 15 are the complex function of material, geometry and stress state parameters of the test specimen. The complete description of these parameters will be out of the scope of this review paper and can best be found in the original paper of Nairn and Liu [68]. Similarly, all the relevant stresses in the fibre and the matrix can be given in terms of modified Bessel functions of first and second kinds. The major advantage of this approach is that it can handle the imperfect interface/interphase, using the interface parameter D_s, originally proposed by Hashin for laminates [69]. The applicability of the interface parameter D_s as measured from the fragmentation test to D_s for the laminates is, although possible, not yet proven. If a correlation between the values of D_s measured from the two test methods can be found, this could overcome the present problem of the correlation of micromechanical test results to the macromechanical tests. However, a complete solution to the problem of correlation in micro- and macromechanical properties cannot be achieved without solving the underlying problems with stress transfer models, as discussed earlier. Nairn *et al.* [68] have proposed two methods to deduce interfacial properties from the fragmentation test. First, the value of D_s can be calculated using the fibre strain data obtained from laser Raman spectroscopy at low applied strains. Secondly, the total strain energy of the fibre fragment can be used for developing the fracture mechanics model for the fragmentation test.

The Bessel function approach, although superior to the variational approach, still does not account for the yielding of the matrix at the fibre interface. However, this model can be modified using the approach developed by Tripathi *et al.* [65] for incorporating the effect of matrix plasticity in the variational model (Section 3.4.4). Another limitation of the variational model is its complexity. However, it still remains a very attractive model for predicting the interfacial shear strength from the fragmentation test.

3.4.4. Plasticity effect model

The major limitation of the existing micromechanical models to predict the stress transfer behaviour of the single fibre composites is their inability to consider the effect of matrix plasticity. The importance of the matrix plasticity on the stress transfer in single fibre composites have been highlighted using finite element analysis and laser Raman spectroscopy (Sections 3.2 and 3.3). Hence, it is of prime importance that the effect of matrix plasticity should be incorporated in these stress transfer models. The effect of matrix plasticity has been incorporated in the variational model by the plasticity effect model using von Mises yield criterion [65]. For the case where no debonding occurs, the plasticity effect model imposes a cut-off limit based on the shear yield and cold draw strengths of the matrix to the value of interfacial shear stress calculated from the variational model of Nairn [31]. A good agreement between the predictions of the shear stress at the fibre–matrix interface and tensile stress at the axis of the fibre from the plasticity effect model and finite element analysis has been shown (Figs 6 and 7) [65]. For Raman-sensitive fibre/matrix composites, laser Raman spectroscopic measurements agree well with predictions from the finite element analysis; hence, it is expected that the predictions from the plasticity effect model will agree with those from the laser Raman spectroscopy also.

For the case where debonding is observed at the fibre–matrix interface, the plasticity effect model is further combined with Coulomb's law to take the frictional stress into account [65]. The plasticity effect model is a three-zone stress transfer model and can predict the stresses in a single fibre composite where partial debonded, partial bonded and shear yielded regions of the matrix coexist (Fig. 8), contrary to the two-zone stress transfer partial debonding model of Piggott. The biggest limitation of the plasticity effect model is its inability to predict the initiation and propagation of the debonding at the interface.

4. Interface characterization parameters

As discussed in Section 2.1, that the second stage of development of an accurate data reduction technique for the fragmentation test is the development of an interface characterization parameter which could be an interfacial shear strength, a stress transfer function or an energy based criterion. So far, the interfacial shear strength calculated from the fragmentation test using the constant shear model has been the major

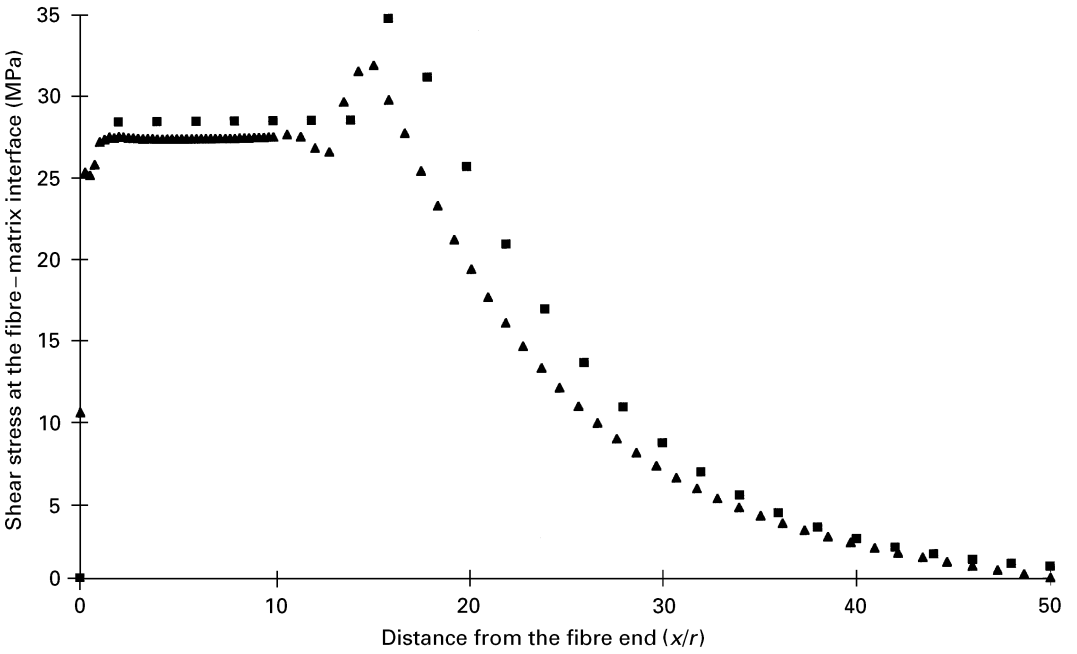

Figure 6 The interfacial shear stress predicted by the plasticity effect model (■) at 3.0% applied strain and that predicted by FE analysis (▲) [65].

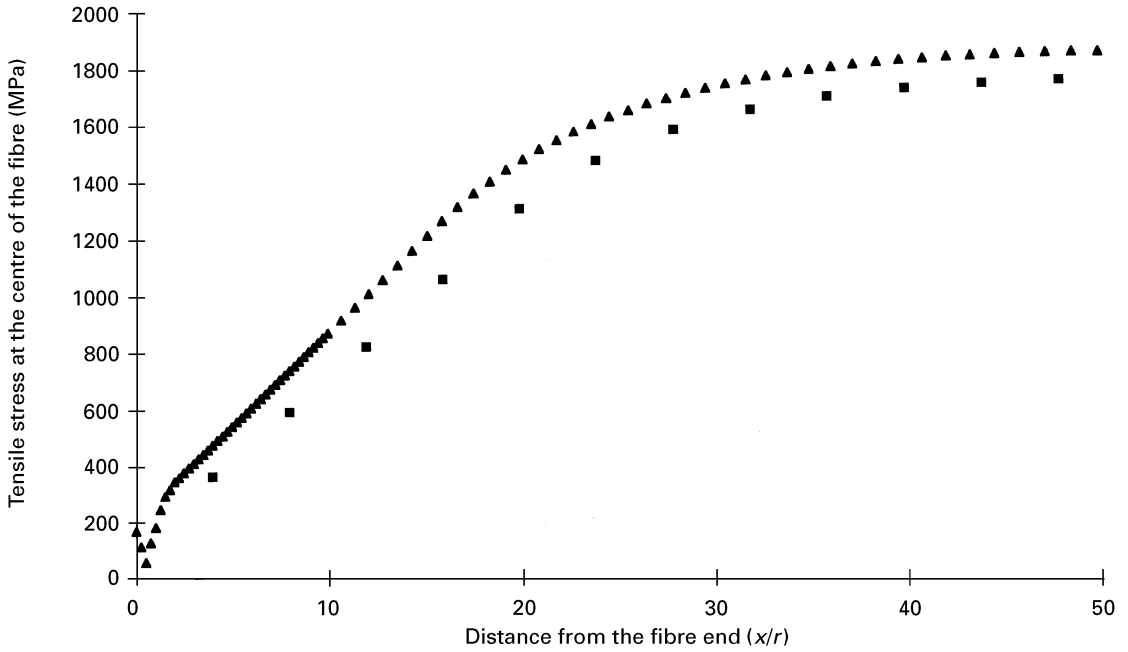

Figure 7 Tensile stress predicted by the plasticity effect model (■) at 3% applied strain and that predicted by FE analysis (▲) [65].

criterion for interface characterization. However, an accurate stress transfer profile in the fragmentation test specimen requires zero interfacial shear stress at the fibre-end. Furthermore, shear yielding of the matrix causes a constant plateau region of the interfacial shear stress near the fibre-end. Thus, the conventional definition of interfacial shear strength which is obtained from the fragmentation test and which predicts the maximum interfacial shear stress at the fibre-end can be questioned [65].

Recently, two data reduction techniques have been proposed for the measurement of interfacial adhesion from the fragmentation test. A new data reduction technique, known as cumulative stress transfer function or CSTF technique has been developed to obtain a fibre–matrix adhesion parameter from the fragmentation test [29, 70]. In this technique, the total stress transferred to an embedded fibre is calculated on the basis of the plasticity effect model which accounts for all the relevant tensile, shear and radial stresses in the single fibre composite and elasto–plastic properties of the matrix, as shown in Fig. 9. Since the CSTF value is a direct measure of the efficiency of the stress transfer to the fibre across the interface, the correlation of the fragmentation test results with those from macromechanical tests is expected to be better,

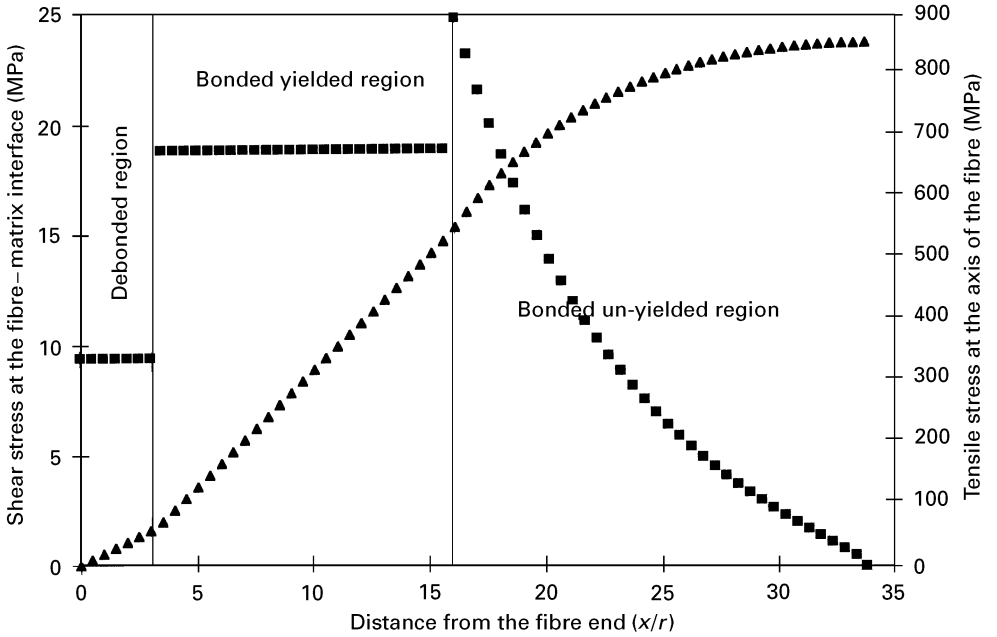

Figure 8 A typical shear stress profile (■) at the fibre–matrix interface and tensile stress (▲) at the axis of the fibre predicted by the plasticity effect model in the case of interfacial debonding [70].

although not proven. Furthermore, the CSTF technique is not sensitive to the fibre fragmentation process and, hence, a matrix similar to that used in real-life composites can be used for the fragmentation test provided a sufficient number of the fibre fragments is obtained. This technique has been used for the measurement of fibre–matrix adhesion in a glass fibre–epoxy resin system and so far the results agree well with the known surface chemistry [29]. The effect of carboxylic functionality deposited on the carbon fibre surface using plasma polymerization on the value of interfacial shear strength obtained from the Kelly–Tyson model and the CSTF value is shown in Fig. 10 [71]. It can be seen from the figure that the CSTF technique provides a smoother (compared to the conventional analysis) trend in adhesion over a range of expected chemical adhesion. It is also clear that the "degree of adhesion" varies between frictional and thermal clamping of the poorly bonded fibre and matrix yielding in the case of a well bonded fibre. It, therefore, becomes possible to identify an optimum surface treatment of chemical functionality. The CSTF calculation is statistically more satisfactory since each individual fragment, including both bonded and debonded regions, is recorded. The value of τ, obtained from the constant shear model, is in fact the reciprocal of the normalized average fragment length. The benefit of the CSTF methodology is that it should prove possible to compare tests done at constant strain, rather than at fragment saturation.

Recently, Wagner *et al.* [72, 73] have proposed an energy-based parameter for estimating the degree of adhesion between a single fibre and its surrounding matrix, using the fragmentation test. In this method, the energy before fibre fracture is transformed into the energy contributions necessary for the formation of a fibre break surface and interfacial debonding. Hence, interfacial debonding associated with the fibre break is measured. This method requires the measurement of the surface energy of the glass fibre surface Γ_f using the fracture energy concept [74]. The fracture energy of the interface Γ_i can be calculated from the expression

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

where L_d is the interfacial debonding length caused by the fracture of the fibre.

Because this method relies on the measurement of interfacial debonding which occurs well below the saturation strain, it does not require the saturation in the fibre fragmentation process. However, this technique, at present, has several limitations. The fracture energy of the interface is calculated from the shear lag model of Cox [41], which in itself is not very accurate. Further, this analysis does not account for transverse matrix cracking, matrix shear yielding and the energy lost during fibre fracture to form the acoustic waves, along with the pre-existing fibre stresses such as the thermal and fabrication stresses. Furthermore, the analysis assumes that no stress transfer occurs in the debonded region through the frictional stress transfer. Definitely, this analysis, as yet, is far from complete but has a strong potential for measuring the fibre–matrix adhesion by the fragmentation test.

5. The relevance of fragmentation testing to real composites

5.1. Role of the interphase

The above discussion assumes the formation of a distinct interface between fibre and matrix which can be characterized by an interfacial shear strength. In some cases, an additional parameter of frictional shear strength is used to characterize the interface

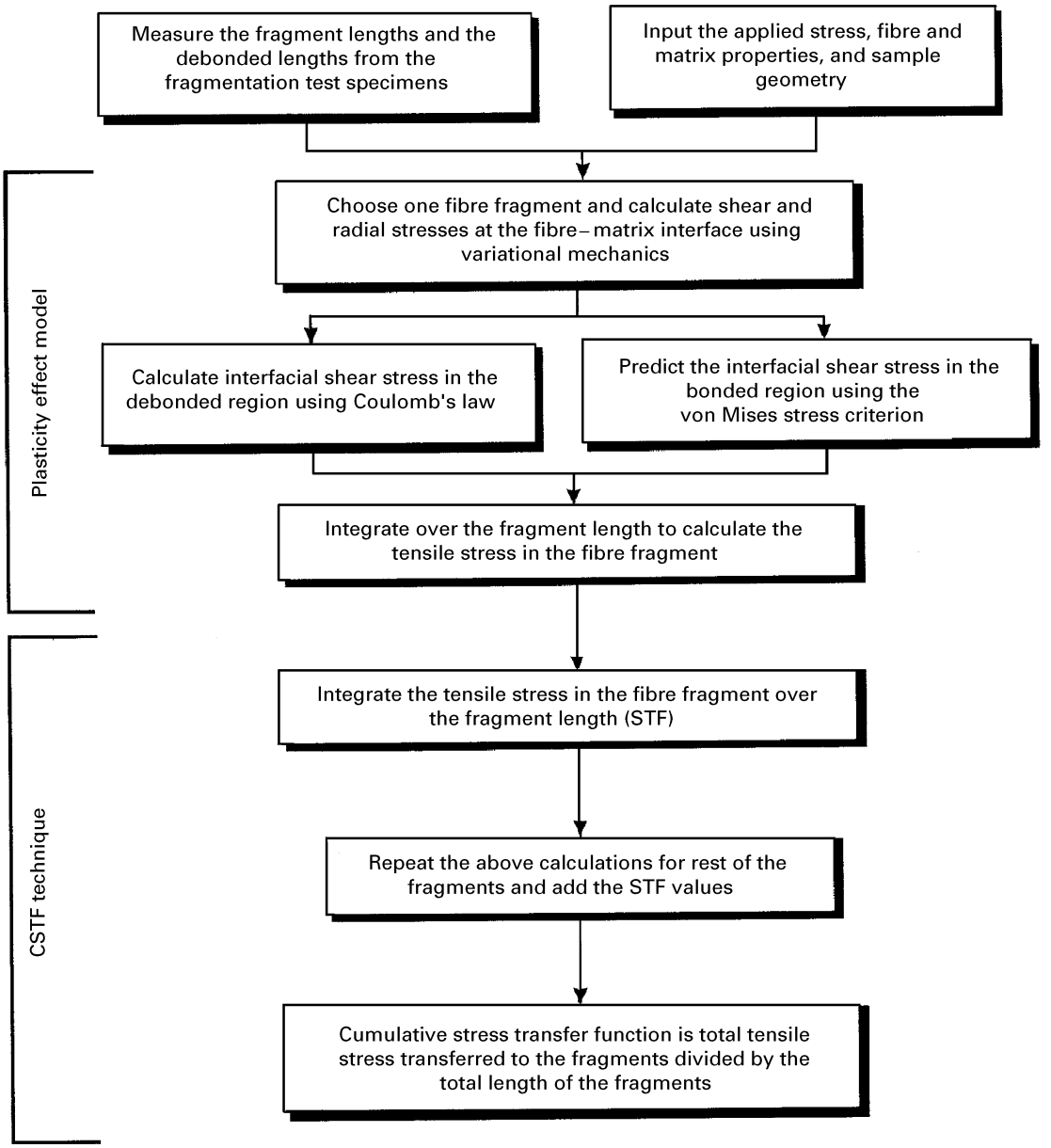

Figure 9 Schematic representation of the CSTF technique to calculate the fibre–matrix adhesion from the fragmentation test [70].

[20, 28, 46]. However, during spinning of commercial reinforcing fibres, the fibres are coated with a protective sizing resin which may, in the case of glass fibre, also contain coupling agents and handling aids. Many researchers have supported the concept that a finite volume of the material surrounding the fibre, defined as an interphase, is quite different in properties to either the fibre or the matrix. This interphase may be homogeneous or may exhibit a property gradient. The interphase region may be a diffusion zone, a nucleation zone, a chemical reaction zone or any combination of the above [75]. The chemistry involved in the formation and control of the interphase region present in glass and carbon fibre-reinforced composites has been reviewed elsewhere [76].

Although the existence of an interphase is now accepted, its properties are still far from clear. The interphase may be characterized by a full set of the mechanical properties including modulus and strength. This interphase may be either more rigid or more ductile than the bulk matrix. It is more likely that a graded region exists in current materials with an ill-defined set of mechanical properties. The evidence in favour of either type of interphase are circumstantial rather than evidential. Equally, the thickness of the interphase region is difficult to define.

Although the effect of an interphase region on the tensile properties of unidirectional composites may be small, an extensive set of investigations has shown that variations of as much as 50% in compressive strength and as much as two orders of magnitude in the notched fatigue life can be achieved by just altering the interphase which may be as little as 1% of the total composite by weight [75]. Furthermore, it is envisaged that the interphase region greatly influences the long-term performance of such systems, especially life and retained strength under cyclic loading and/or in the presence of aggressive environments such as high temperature and corrosive chemicals [77–79].

From the above discussion, it is clear that an interphasial region will significantly modify the stress transfer function. Thus the fragmentation test data

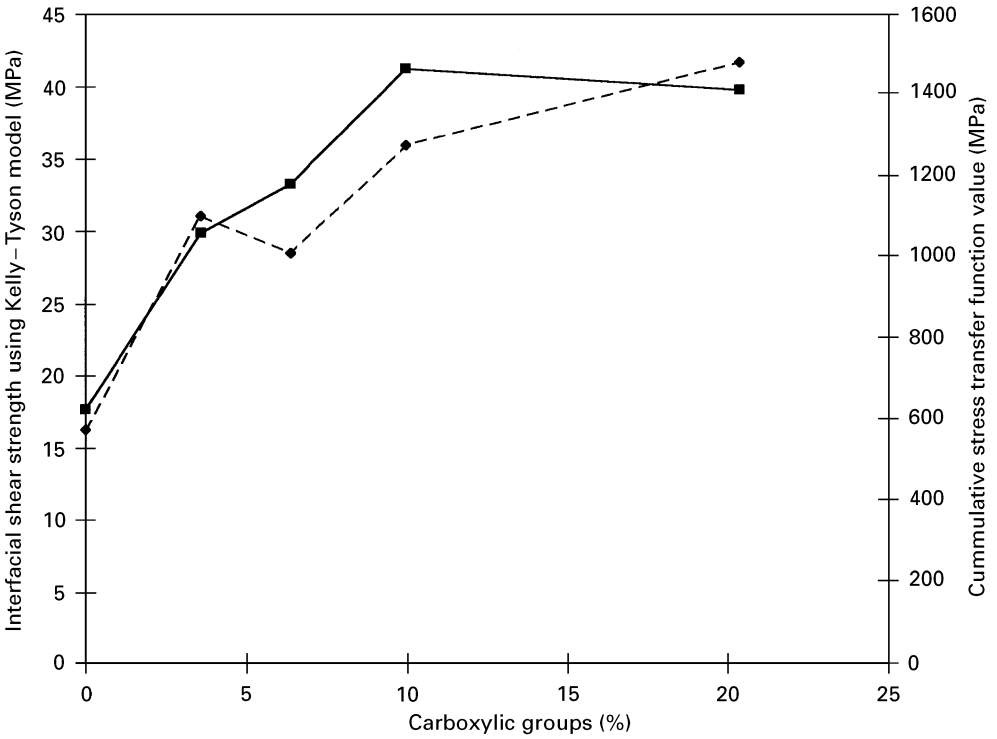

Figure 10 The effect of carboxylic acid functionality on the carbon fibre surface deposited using plasma polymerization on the interfacial adhesion of single carbon fibre epoxy composite [71]. (◆) ISS; (■) CSTF.

reduction methodology also needs to address the influence of these factors. Finite element analysis was used to study the stress transfer in bimatrix fragmentation test specimens [35, 36]. A corollary to the study will be to explore the influence of a distinct interphase on the shear stress transfer process. A three-zone concentric cylinder was used to model the reinforcing fibre, interphase and the matrix. The effect of resin matrix and the interphase was studied in detail. Three types of interphases were used to predict the role of hard, intermediate and ductile interphase. The following conclusions were drawn [76]:

1. The shear stress profile remained the same as that shown and discussed in Sections 3.3 and 3.4.4 in the broader sense. However, the following deviations were observed:
2. The overall shear stress profile is controlled by the more ductile phase which may be either the resin or the interphase. With a ductile matrix, yielding of the matrix dominates the stress transfer but when the situation is reversed and a ductile interphase region exists, then the yielding of the interphase dominates the process.
3. If a hard interphase is used, the shear stress between the fibre and the interphase is increased by few MPa depending on the modulus of the interphase. Consequently, the magnitude of the tensile stress transferred to the reinforcing fibre through a rigid interphase is increased, to a degree.

5.2. Correlation with composite properties

Further complications arise when attempts are made to correlate the results of the fragmentation test with the mechanical performance of high fibre volume fraction composites (macrocomposites) since the stress profiles are quite different in each composite. During the micromechanical testing of fibre-reinforced composites, it is assumed that a particular trend in the values of interfacial shear strength of the fibre with different surface treatments reflected by the micromechanical testing will be observed during the micromechanical tests also. However, this correlation is difficult to prove and sometimes does not follow, as explained in the following paragraphs. Different macromechanical properties show differing sensitivities towards the fibre–matrix adhesion measured using the fragmentation test. The ultimate aim of interfacial studies is to provide the knowledge which enables the mechanical and/or hygrothermal properties and hence reliability of a composite structure to be ascertained.

Drzal and Madhukar [80] used a well-characterized experimental system consisting of an epoxy resin with three carbon fibres with different surface treatments. Single fibre fragmentation tests were conducted to quantify the level of fibre–matrix adhesion. Three different surface treatments produced three different failure modes in the fragmentation test specimens. The lowest level of adhesion produced a frictional debonding, the intermediate level produced interfacial crack growth and the highest level of adhesion produced transverse matrix cracking. High fibre volume fraction composites made from the same materials were tested for on- and off-axis properties. It was observed that the composite properties could only be explained when both the fibre–matrix adhesion and damage events are considered.

The effect of carbon fibre surface treatment on the unidirectional tensile strength of the composites was examined by Ivens *et al.* [81]. The carbon fibre strength is influenced by the degree of surface treatment. Two competing factors are reported to contribute towards the unidirectional fibre composite strength. First, an improved surface treatment results in a shorter ineffective length (i.e. the length over which a broken fibre will not carry the maximum load or the load transfer length). Secondly, an enhanced surface treatment results in a lower debonded length and in the limiting case can lead to transverse matrix cracks. The transverse matrix crack results in an increased stress concentration factor on the neighbouring fibres, causing brittle failure of the composite. As a consequence, it was shown that the tensile strength of a unidirectional carbon fibre/epoxy composite was maximized when 50% of the standard commercial fibre surface treatment was used. A method for the calculation of composite strength was proposed which combined these two limiting cases. This model includes the effect of strain reduction in the fibre, creation of the crack between the two fibre-ends and the increase in stress in the matrix as a result of fibre fracture.

Hoecker and Karger-Kocsis studied the effect of the interface on the transverse and impact properties of the carbon fibre/epoxy composites [82]. Fibre–matrix adhesion was characterized by determining the interfacial shear strength from single fibre fragmentation and micro-debond tests. The effect of two well-characterized interfaces on the transverse tensile, transverse flexural, interlaminar shear stress and the impact properties of the unidirectional and cross-ply laminates was studied. The effect of the quality of the adhesion was observed in the case of the transverse tensile test even though the loading direction in this test is very different from the single fibre fragmentation test. The improvement in properties was attributed to differing mechanisms of failure which changed from clean fibre surface fracture to the cohesive matrix failure as the interfacial adhesion improved. The interface relevance of the transverse flexural properties was not observed. Furthermore, short beam shear tests do not show the effect of the adhesion while the transverse isoipescu test shows an improved interfacial shear strength for the composite with the improved adhesion, as suggested by the micromechanical tests. The Charpy impact properties of the unidirectional laminates were a complex function of the interfacial adhesion. An improvement in impact properties of the composite with a "good" interface (as predicted by the micromechanical tests) was observed which is contrary to the popular belief that improved adhesion will cause brittle failure of the composite [82].

The effect of interfacial adhesion on the properties of laminates can be relatively small because of the long fibre length. However, the effect of interfacial adhesion will be substantial in the case of compresssion-moulded or injection-moulded short fibre composites because a major portion of the fibre will not experience the maximum load. The interfacial shear strength from short fibre composites can be indirectly calculated using a technique proposed by Bowyer and Bader [83]. The Bowyer and Bader methodology to calculate the interfacial shear strength from the strength of short fibre reinforced composites is essentially based on the Kelly–Tyson model and considers different fibre lengths present in a composite [83]. The condition required for the applicability of the Bowyer and Bader approach is similar to that required for the constant shear model i.e. shear stress remains constant at the fibre–matrix interface due to either the matrix yielding or interfacial debonding. Using the well known law of mixtures, Bowyer and Bader suggested that the composite strength is given by

$$\sigma_c = CX + CY + Z \tag{17}$$

where X, Y and Z are total contributions of the subcritical fibres, supercritical fibres and matrix as defined below

$$\sigma_c = C\left[\sum \frac{\tau l_i V_i}{2r} + \sum E_f \varepsilon_c \left(1 - \frac{E_f \varepsilon_c r}{2l\tau}\right) V_j\right] + E_f \varepsilon_c (1 - V_f) \tag{18}$$

Where V_i and V_j are the volume fractions of fibres with lengths l_i and l_j respectively and C is the orientation factor. The only unknowns in the Bowyer and Bader equations are τ and C which can be calculated using two values of tensile stress and strain from a tensile strength/strain curve of the composite material. It has been reported that the value of interfacial shear strength obtained from the bimatrix fragmentation test is not in agreement with that obtained from the strength of injection-moulded glass fibre phenolic composite using Bowyer and Bader technique, even though underlying assumptions in the two techniques are the same [1].

Based on the above discussion, it can be concluded that the macromechanical properties are a complex function of the interface properties as measured from the micromechanical tests, in particular the fragmentation test. A clear understanding of the translation of the micromechanical results to the macromechanical properties is still in the fluid stage and a proper consideration of the loading scenario of the ultimate composite application is very necessary for the design of the optimum interfaces for the improved mechanical properties of the fibre reinforced composite materials.

6. Conclusions

In this review, we have highlighted the complexities of interfacial characterization in the fibre–matrix composite using the single fibre fragmentation test. It has been concluded that the existing data reduction techniques are still unable to characterize the interface completely and accurately. Over the last few years, however, it has become clear that simple measurement of final fragment length distribution during the fragmentation test is not sufficient for the characterization of the interface. Additional measurements such as interfacial debonding caused by the fibre fracture,

growth of interfacial debonding, preferably at incremental values of applied strain, should be recorded and measured during the fragmentation test. Further, latest data reduction techniques to measure interfacial parameters from the fragmentation test, such as those discussed in Section 4, should be employed. Because of the complex nature of these data reduction techniques, a close co-operation between the theoreticians and experimentalist is necessary.

Furthermore, forthcoming data reduction techniques for the fragmentation test should take two major limitations of the fragmentation test into account. First, it should employ the same resin for the fragmentation test as used in the manufacture of laminates since the interface/interphase may be very different if the two resin matrices (one for the fragmentation test and another for the manufacture of the laminate) differ. Secondly, the data reduction technique should be applicable at lower applied strains than those presently used for the fragmentation test because real laminates never approach such high strains as used for the fragmentation test and the fibre fragmentation process never occurs in laminates. Further reliability/accuracy of the test and the time required for the test will be crucial factors for the application of fragmentation technique at the industrial level as a standard quality control tool. Over the last few years, in the absence of an accurate and reliable data reduction technique for the fragmentation test, use of laser Raman spectroscopy to directly measure interfacial shear stress have been proposed. However, it is still to be clarified how LRS can differentiate between two "good" interfaces which show yielding or different sizings which have unnoticeable effect on the interface as such. Further, it is still to be shown how LRS can handle the large number of fibre fragments which are formed as a result of statistical nature of fibre strength.

Needless to say, until a recognized technique is established, a scientific basis for the surface treatment of fibres, for optimizing the performance of high fibre volume composites, cannot be generated. In the absence of a direct correlation between the interfacial properties measured from the fragmentation test and the macroproperties obtained from the testing of high fibre volume fraction composites, it is likely that the characterization of the interface by the single parameter such as interfacial shear strength may not give the complete answer. Hence, a particular test procedure and/or a particular data reduction technique which may be directly relevant to the crucial laminate property may be necessary to get the complete answer for the interface/interphase engineering or in other words combinational interface engineering. The development of a comprehensive data reduction technique for the fragmentation test which will enable the relationship between the interfacial chemistry and composite properties to be studied directly with confidence will have very wide consequences. More specifically, the true role of surface chemistry and sizing resins, on the macro-properties of a composite can be understood. Furthermore, the identification of degradative mechanisms in the composites (i.e. matrix, interfacial or interphasial) will provide far more reliable structures. This will allow fibre manufacturers and designers of the fibre reinforced composites to accurately design the interfaces/interphases for the optimization and improvement of mechanical performance especially off-axis properties and those which determine reliability and durability, and hence, provision for lighter weight structures through more effective utilization of the fibre properties.

Acknowledgements

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