

ANALYSIS OF THE SINGLE-FIBRE FRAGMENTATION TEST

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Abstract

This work investigated the analysis and experimental protocol of the single-fibre fragmentation test. The possible interpretations of this test were first reviewed, several strength or energy-based models being available in the literature. An extensive screening study was carried out to determine the optimal geometry and test conditions of single-fibre fragmentation testpieces. The results of these tests, together with a modelling of the fragmentation process, revealed that the widely used interpretation of this test is inappropriate when describing the essential features of interface failure. Instead, local stress or energy analysis should be used. One particular limitation of existing models is their poor description of the stress field at low volume fractions. In this work, a simple, closed-form, shear-lag based model was chosen to describe the distributions of the axial stress in the fibre and the interfacial shear stress. It simplified to a great extent the modelling and simulation work. While approximate, this method enables a quantitative assessment of the quality of the interface in carbon fibre and glass fibre epoxy single fibre composites. Finally, limitations and possible extensions of this study were outlined.

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1 INTRODUCTION

The properties of the fibre/matrix interface in any fibre reinforced composite material are crucial with regard to the resultant properties of the composite itself. For most polymer composites the requirement is for a very strong interface so that the properties of the composite become susceptible to microstructural damage formation only at relatively high loading. All unidirectional fibre reinforced composite materials exhibit significantly different properties when comparing axial properties to those measured transversely. During loading the transverse properties are degraded more than axial properties because of interface failure and its consequences, and this arises also in the 90° plies of laminates leading to ply crack formation. The development of a strong fibre/matrix interface helps to maintain transverse properties during loading, and minimise the differences between axial and transverse composite properties, and the rate of accumulation of damage during loading. It is clear that the overall composite properties, whether in unidirectional or laminated form, are significantly affected by the strength properties of the fibre/matrix interface. Various methods of measuring these properties have been developed most of which require the use of a micro-mechanical model to interpret the results of the experiments in terms of so-called interface properties [1]. Fibre/matrix interfacial failure has received considerable attention because it is one of the principal micro-level damage modes in laminates. These are conventionally:

- fibre/matrix interfacial failure;
- fibre failure;
- matrix failure.

Most failure properties of laminates can be obtained from the adhesion test results by the use of semi-empirical approaches [2,3], which in general lack physical justification. Since the importance of fibre/matrix adhesion for various polymer composite systems has been quantified, numerous tests have emerged [4,5], among which single-fibre composites are often employed. The use of well defined geometry and loading was essentially motivated by the need to obtain the interface characteristics free of the influence of neighbouring fibres. Among fibre-matrix adhesion tests, the most popular are the fibre pull-out test [6-8], the fibre push-out test [9,10], the microbond test [8,11-16], also referred to as the microdrop test, and the fragmentation test [17-21]. The latter technique was introduced in the following simulations because it offers advantages over alternative methods of being applicable to a wide variety of systems, including brittle fibres. It is generally preferred to other test methods in the industry because little equipment is required (a microscope and a tensile tester), as well as the simple preparation of specimens.

This work explores the conventional analysis of the fragmentation test by using a strength-based approach, derived from a modified shear-lag model. First, the currently available models are briefly reviewed, and the choice of the model is discussed. Second, a simulation of the fragmentation process was introduced, and it is shown that global fragment analysis failed to reveal most fibre and interfacial features. An extensive screening study allowed the determination of the optimal geometry and test conditions of the single-fibre fragmentation testpieces. It was further decided to conduct local stress analysis on two different systems with several interfacial strengths. The first system consists of carbon fibre-epoxy testpieces, with an intact or a greasy fibre surface, or immersed in boiling water for 10 hours. The second system consists of glass fibre-epoxy testpieces with different sizings. In both systems, it was possible to assess the quality of the interface using this test in combination with this analysis. The interfacial strength obtained could not be formally related to the thermodynamic work of adhesion, due to the lack of a suitable theory of solidifying thermoset resins.

The advantages, as well as the drawbacks of this test are finally summarised, with particular emphasis put on the application of this test in an industrial environment.

2 STATE OF THE ART

2.1 Defining Interface Properties

A key problem regarding the failure of interfaces between fibre and matrix in unidirectional fibre reinforced composites is defining exactly what is meant by the term 'interface failure'. The following concepts need to be addressed, namely, interface strength and interface fracture energy [22].

2.1.1 Interface strength

The strength of an interface is often regarded as being a key parameter characterising the failure of an interface. The term 'strength' implies a critical value of a component of stress - but which one? Thinking of a simple composite having a single fibre perfectly bonded to a surrounding hollow cylinder of matrix, it is easy to envisage changing the temperature in such a way that a uniform tensile radial stress is induced at the fibre/matrix interface. At a critical temperature the interface fails and the critical uniform interfacial normal stress can be thought of as being the interface strength in tension. However, in many interfacial tests the loading of the interface is dominated by shear deformation that leads on to the concept of an interfacial shear strength. Indeed it is often assumed, that at the point of shear dominated interfacial failure, the shear stress on the interface has reached a critical value known as the shear strength. Frequently it is assumed that the shear stress on the interface is uniform enabling the easy calculation of an apparent interfacial shear strength. For all experimental configurations of interface tests the shear stress will not be uniform so that an assumption of uniformity (as in the interpretation of micro-drop test) will introduce errors and lead to interfacial strengths that may not be relevant in other test configurations. Thus using interface data from a particular test in other loading situations can introduce a significant degree of uncertainty.

The interesting question is: what happens if the interface is subject to combined tensile and shear loading? This is often the case in interfacial test method configurations. There is no easy answer to this question using strength concepts.

2.1.2 Interfacial fracture energy

Interfacial failure is also characterised by an interfacial fracture energy that quantifies the degree of bonding between fibre and matrix. This concept is ideal for the consideration of the propagation of a debonded zone in the interface due to progressive axial loading. Such a phenomenon occurs in fibre pull-out tests, and fibre fragmentation tests. A physically based approach will attempt to balance the energy available from the fibre and matrix, taking account of the potential energy of the loading and of energy dissipation due to friction at the interface in the debonded region, with the energy required to overcome cohesive interface forces during the creation of new areas of debonded interface. The beauty of this technique is that it solves the problem of taking account of the effects of combined tensile and shear loading of the interface. The difficulty is that it is not yet clear how the initiation of debonding can be considered. One viable approach is to assume that the interface in a composite either has a distribution of small defects in the form of local debonds, or that a stress singularity promotes fibre/matrix debonding near the singularity at very low loads. Such singularities occur, for example, where fibres enter the matrix in pull-out tests, or where fibre fractures occur in fragmentation tests. The initiation of interfacial debonding is thought to

arise from the initiation of the growth of the defects governed by fracture mechanics principles. The initiation of debonding may be such that the initial stages of debond growth may be highly unstable so that a significant portion of the interface suddenly debonds followed by arrest. Subsequent debond growth then requires additional loading. Another difficulty is that debond growth involves mixed mode fracture.

The criterion for fracture in mode I (only tensile opening of a crack) involves the mode I fracture toughness K_{Ic} such that fracture occurs when the mode I intensity factor K_I attains the critical value K_{Ic} . The fracture toughness is related to cohesive forces (characterising adhesion) acting at the debond tip, and to small scale yielding plasticity mechanisms that must occur in the debond tip region. Similarly the criterion for fracture in mode II (only in-plane shear opening of a crack) involves the mode II fracture toughness K_{IIc} such that fracture occurs when the mode II intensity factor K_{II} attains the value K_{IIc} . The measurement of K_{IIc} for a material involves difficulties of attaining pure mode II conditions which are not usually realised in practice. For mixed mode opening where the crack faces simultaneously exhibit in-plane and shear opening, the correct fracture criterion to use does not seem to be decided. An approach often made (see for example Reference [23]) is to assume that mixed mode fracture is governed by a relation of the form

$$\left(\frac{K_I}{K_{Ic}}\right)^2 + \left(\frac{K_{II}}{K_{IIc}}\right)^2 = 1 \quad (1)$$

which has no physical justification except that it is one method of interpolating between the limiting cases of pure mode I and mode II fracture. The following equivalent relation involving strain energy release rates is also used

$$\frac{G_I}{G_{Ic}} + \frac{G_{II}}{G_{IIc}} = 1 \quad (2)$$

Many other variations of this theme have been proposed

2.2 Principle of the Fragmentation Test

The single-fibre fragmentation test involves a resin dogbone testpiece in which a single brittle fibre is embedded along the testpiece's axis. While the specimen is loaded in tension, the fibre breaks repeatedly until all the fibre fragments are shorter than a critical length l_c . It is then impossible to reach the failure strength of the remaining fragments, as they are too short to transfer enough axial stress through the interface for another break to occur. Therefore, a saturation state is reached. The distribution of fragment lengths is analysed through stress transfer model(s), and a measure of interface performance is derived. Very few models are capable of accurately accounting for the effect of interactions between fibre breaks. The very popular constant interfacial shear stress model is often used to predict the onset of saturation as the assumption of a uniform shear stress in the interface requires that cracks must not be closer than a particular distance that is predicted by the model. This distance depends upon the value of the interfacial shear stress and thus measurements of average crack spacings at the saturation condition can be used to estimate the average interfacial shear strength. The problem with this approach is that the model of stress transfer on which it is based is rather approximate for one to have total confidence in the methodology. As a matter of fact, the model predicts that all fragment lengths are between $l_c/2$ and l_c , which is not the case in the experiment.

Much discussion has been carried out during the past two decades on the nature of the result obtained. A definitive treatment of experimental data is still awaited regarding the following aspects of fragmentation testing:

- inaccurate modelling of the interfacial stress state
- poor reproducibility of the experiments
- paradoxically, lack of validated relations between the interface toughness and the work of adhesion.

These three points are addressed in detail in the following. Note that the fragmentation process involves, as stated in the introduction, multiple damage modes. In general, the three micro-level damage modes are present in a fragmentation test, thereby rendering the modelling an intricate task. Most analyses are simplified to a considerable extent, and still, describe perfectly well some, but not all, of the features of this test.

2.3 Model Requirements

The requirements, as well as the possible stress transfer models when the interface is intact are addressed in the two following sections [22]. Prior to that, it is useful to clarify the notion of intact or debonded interface.

2.3.1 Interface characterisation

Fragmentation tests involving the sequential fracture of a single fibre embedded in the matrix do not lead to many of the difficulties of analysis that are encountered when using other test methods, e.g. the microdrop test. The geometry and mode of loading are such that the models used to interpret the results can be assumed to be axisymmetric and easily modelled in principle. This is because the fibre is embedded in a large volume of matrix whose external boundary is remote from the interface itself, and which does not need to be axisymmetric in the tests being modelled. A great deal of modelling has been devoted to analysing the stress distribution associated with fibre fractures. If the interface is very strong and remains perfectly bonded even when the fibre has fractured, the shear and normal stress tend to infinity as the fibre fracture is approached. The normal stress is again often tensile, promoting the debonding of the interface even for very small applied loads. As the axial load is increased the region of debonding will increase as the tip of the debond propagates along the fibre/matrix interface away from the fibre fracture. The major problem encountered with the technique is the uncertainty regarding the nature of contact between fibre and matrix after the interface has debonded. It is often assumed that the interfacial shear stress in the debonded region is uniform having a value that can be regarded as a material constant. This does not make sense as the value of the shear stress is highly likely to be dependent upon the local normal stresses, as would occur for frictional effects. The thermal and moisture/shrinkage residual stresses can in some situations increase the value of the normal stress because of fibre clamping effects arising from thermal and moisture expansion mismatch between fibre and matrix. Another problem with the assumption of a constant interfacial shear stress is that the shear stress will have a non-zero value in the interface at the location of the fibre fracture, while it must have a zero value as the same point is approached along the broken fibre surface. This situation can be modelled only if a suitable type of singularity is allowed to exist at the point in the interface at the location of the fibre fracture. Such an approach does not seem to have been used in the modelling field. Most models predict that there is a non-zero shear stress on the fibre fracture surface which is physically impossible in the absence of a singularity.

An alternative method of modelling stress transfer in the debonded region assumes that stress transfer in the debonded region is governed by a friction law that is sometimes modified to take account of surface roughness effects. The advantage of this approach is that it allows the interfacial shear stress to tend to zero as the fibre fracture is approached (so that

the fibre fracture surface is stress free as required), and leads to a non-uniform interfacial shear stress that is affected by the degree of fibre clamping arising from thermal and moisture expansion mismatch effects. The Coulomb friction law is often assumed for the debonded region introducing a friction coefficient that characterises interface behaviour. This leads to the problem of determining its value from the results of fragmentation experiments.

2.3.2 Fibre fracture

The fragmentation test is the only test that involves fibre fracture when assessing interface properties. It is useful to consider in more detail the modelling of fibre fracture and its effect on the behaviour of interface debonding. For unidirectional composites loaded in the fibre direction, the first damage mode encountered is the fracture of fibres. The strength of fibres can be subject to significant scatter and this leads to fibre fractures that are distributed throughout the composite. The objective here is to consider only isolated fibre fracture events in order to discuss the role of the interface during such a process. There are two distinct ways of approaching the prediction of the initiation of a fibre fracture. To discuss these methods it is useful to focus on an idealised fragmentation test where a single fibre is embedded in matrix material which is assumed to have the form of a cylindrical tube surrounding the fibre which is perfectly bonded to the matrix at all points on the interface. The concentric cylinder system is loaded axially and can be subject to thermal residual stresses.

One approach, that is usually not considered in practice, is to assume that during the axial loading of the system the fibre/matrix interface remains intact for all fibres in the composite. Thus at the point of fibre failure the interface is perfectly bonded so that the tractions and displacements are continuous at all points on the interface. Immediately following fibre fracture the system responds elastically (and instantaneously if inertia effects are neglected) and an equilibrium stress field arises that can be predicted by solving the elasticity problem for a single fibre fracture in a matrix where the interface is perfectly bonded. The fibre fracture event is controlled by an energy balance where the energy required to form the crack in the fibre (characterised by a suitable fracture energy) is balanced by an availability of energy arising from a change in the elastic stored energy and the work done by the applied tractions. If the fracture event is assumed to occur under a fixed load then the energy availability can be calculated directly from the change in complementary energy (or the Gibbs free energy which is the negative of the complementary energy). Using a multiple cylinder representation for the fibre and matrix it is possible to solve the elasticity problem very accurately [24,25], and accurate estimates can be made of the energy availability for fibre fracture. A characteristic of the stress distribution, arising for a fibre fracture with a perfectly bonded interface, is that the stress field is singular at the location of the intersection of the fibre break and the fibre/matrix interface. The radial stress is tensile and this will lead directly to the initiation of debonding along the interface, and eventually an equilibrium configuration will be achieved.

The second approach considers that fibre fracture and interface debonding occur simultaneously so that when considering the energy balance the energy availability is calculated from the energy difference between the state of the system just before fracture occurs with the equilibrium state that arises following fibre fracture and the consequential fibre/matrix debonding that also occurs. When performing such calculations account must be taken of the energy required to debond the fibre/matrix interface and the energy dissipated by friction as a result of the relative motion at the interface of the fibre and matrix within the debond zone. The final equilibrium state may be difficult to determine as dynamic effects in the form of stress waves may lead to more than one fibre fracture at a given load, and to

stick/slip friction effects occurring in the debonded zones of the interface. These effects may have an influence on the resulting equilibrium state of the system.

The two methods described for predicting fibre fracture will lead to differing estimates for the stress at which a fibre fracture will initiate. It is of interest to know which will give the more reliable estimate. To attempt an answer to this question requires the consideration of the effects of defects which are distributed at random along the length of the fibre. In order to focus on key aspects, while enabling a tractable approach to be made, it is useful to regard the defects as small penny shaped cracks or external cracks which are normal to the fibre direction and whose centres lie on the axis of the fibre thus maintaining the axi-symmetric geometry. The size of the cracks will be variable and their maximum size is expected to be much smaller than the fibre radius. The fibre fracture event will be initiated by the growth of the defects (possibly unstably) so that the entire cross-section of the fibre is cracked. The condition for such defect growth can be determined using fracture mechanics concepts. As an embedded defect grows across the fibre cross-section (assumed to be in an axisymmetric manner) it might be possible for the interface to debond before the fibre defect has grown to the interface. Thus both fibre fracture and interface debonding could possibly occur simultaneously, although the failure initiating event (the initial growth of the defect) would not be influenced in any way by the properties of a perfectly bonded interface.

Background on Strength and Energy Methods

It is clear from the above discussion that the accuracy of interpretation of the results for interface testing depends upon the realism and accuracy of the stress transfer model used to represent the behaviour of the interface. It is appropriate now to identify those models that are recommended for use in this manner. It is recognised that many other approaches have been taken in the literature that would require a great deal of time and space to discuss fully in this report. First, the most popular models are introduced, then the most accurate models are reviewed.

2.4.1 Shear-Lag Models

Stress transfer analysis originated with the work of Cox [26], in which the so-called shear lag analysis was described for the case of fibre reinforced materials. The elastic solution to the stress field in the reinforcing phase and at the interface found significant success for studies of single fibre composites. This approach was limited by the one-dimensional analysis where the radial and hoop stresses were not determined, and the stress state was not entirely known. Moreover, it was necessary to determine an interfacial shear parameter H , for which Cox proposed a solution in the limit of high fibre volume fractions; this solution includes an effective matrix radius. However, single fibre composites have inherently low fibre volume fractions and the Cox approach may describe their stress field poorly. One complete analytical derivation of shear lag analysis was given in the work of Piggot [27], with an estimate for the interface parameter H for the case of hexagonal and square fibre packing. Cox analysis has been shown to be robust where R_m/R_f is far from 1 and ∞ , where R_m and R_f denote the effective matrix radius and fibre radius respectively.

The traditional shear lag analysis [26] assumes:

- perfectly elastic and isotropic matrix and fibre properties
- proportionality between interfacial shear force and the difference between the displacement in the matrix and the displacement that would exist if the fibre were absent
- perfect bonding between matrix and fibre
- same lateral stiffness of fibre and matrix

- no residual stresses.

Additionally, the stress is taken as uniform through a radial section of fibre, and the stress is entirely transferred from matrix to fibre by shear at the interface. This leads to the well-known determination of the axial stress σ_f in the fibre:

$$\sigma_f = E_f \epsilon_\infty \left(1 - \frac{\cosh(\beta z)}{\cosh(\beta t)} \right) \quad (3)$$

where ϵ_∞ is the far-field applied strain, E_f is the Young's modulus of the fibre, z is the axial coordinate, t is the fibre half-length, and:

$$\beta = \left[\frac{H}{\pi R_f^2 E_f} \right]^{1/2} \quad (4)$$

with

$$H = \frac{\pi E_m}{(1 + \nu_m) \ln(R_m / R_f)} \quad (5)$$

where E_m and ν_m are the matrix's Young's modulus and Poisson's ratio, R_m and R_f are the matrix and fibre radii, respectively.

The correlation between experimental data and Cox based models was generally good, but use was limited by the introduction of the effective radius of matrix R_m whose physical existence has not been defined clearly in the case of single fibre composites. As a consequence, these models are often used for comparative rather than predictive purposes. Typical profiles of axial stress in fibre and shear stress at the interface are represented schematically in Figure 1.

Refined models were developed to account for the radial displacement and realistic boundary conditions. The first one was presented by Whitney and Drzal [28], based on the superposing of an exact far field solution and an approximate local transient solution. The method

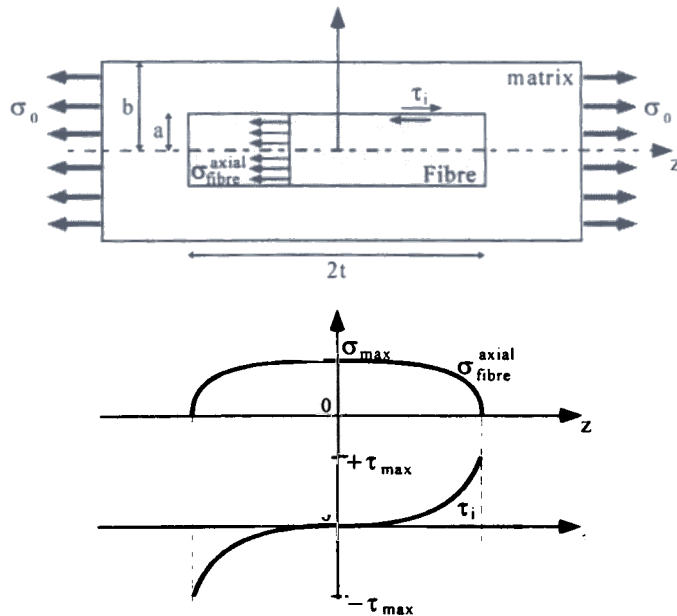


Figure 1 Schematic fibre axial stress and interfacial shear stress as determined by Cox model

was hampered by the use of a shear lag based stress function, although this solution has shown satisfactory experimental agreement for high fibre length per radius aspect ratios.

2.4.2 Variational Methods

An attractive approach was proposed by Nairn [21], where the problem of axially symmetric stress transfer was solved using the variational method and minimisation of the complementary elastic energy, with only one assumption: the axial stress in both fibre and matrix depended only on the z -coordinate. His approach used a generic stress potential as a first approximation. Whitney and Drzal [28] outlined the major advantage of the stress potential formulation, where all boundary conditions were satisfied and a zero shear stress at the ends was obtained, as depicted in Figure 2. One of the limitations of this analysis, similarly to Cox model, was the introduction of an equivalent matrix radius R_m .

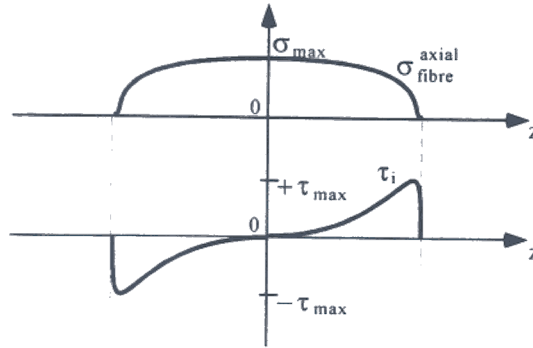


Figure 2 Schematic fibre axial stress and interfacial shear stress as determined by Nairn model

2.4.3 Review of the more accurate stress transfer models

The subsequent discussion will concentrate only on the models thought to be the best candidates for use in the interpretation of fragmentation and pull-out tests. The discussion will address three aspects of the modelling of interface behaviour, namely, perfectly bonded interfaces, constant shear stress behaviour, frictional slip behaviour.

2.4.3.1 Perfect interface bonding

There are essentially three different approaches that have been made to the accurate modelling of the stress distributions associated with perfectly bonded interfaces in fragmentation tests. With the emphasis on accurate stress analysis Nairn [29] following Hashin [30], has developed a stress transfer model for an imperfect interface where the interface imperfection is described by interfacial boundary conditions of the form

$$w_f - w_m = \frac{\sigma_{rz}}{D_s}, \quad u_f - u_m = \frac{\sigma_{rr}}{D_n} \quad (6)$$

where w_f and w_m denote the axial displacement in the fibre and matrix respectively, where u_f and u_m denote the radial displacement in the fibre and matrix respectively, and where σ_{rr} and σ_{rz} denote interfacial normal and shear stresses respectively. The parameters D_s and D_n are interface parameters associated with shear and normal deformations. By letting D_s and D_n tend to infinity the interface conditions reduce to those for perfect bonding where the displacement components must be continuous across the interface. Nairn [29] has applied the model for the case of perfect bonding comparing predictions with experimental results, obtained for relatively low applied loads using the Laser Raman technique, where a reasonable correlation has been obtained as there was very little interface debonding. For higher applied loads significant interface debonding occurs where use must then be made of

the above conditions for an imperfect interface. The experimental data indicate two distinct types of interfacial stress transfer behaviour (a debonded region near the fibre fracture, and a perfectly bonded region) that are not represented by the model for an imperfect interface. This difficulty could be overcome if the mathematical problem were to be split into two zones, one having imperfect bonding while the other has perfect bonding. It is concluded that Nairn's model [29] provides an accurate description of the stress transfer associated with a perfectly bonded interface but has the draw-back that the maximum interfacial shear stress does not occur at the location of the fibre fracture where it should be singular rather than zero. Shear-lag models lead to a maximum but finite value of the interfacial shear stress occurring at the location of the fibre fracture.

Pagano [31] has developed a powerful axisymmetric stress transfer model for a set of perfectly bonded concentric cylinders that can take account of hygrothermal effects. A representation is proposed for the stress field in each cylinder and it is shown how weighted averages of the displacement components may be calculated. The representation involves various stress transfer functions whose values are determined from sets of differential equations that are derived by imposing the Reissner variational principle. A key assumption is that in each cylindrical shell the stress components $\sigma_{\theta\theta}$ and σ_{zz} are linear in the radial coordinate. It is noted that for an isolated matrix crack or fibre fracture the form of the stress field assumed does not reduce to the Lamé solution at large distances from the fracture plane. The model has been used to predict the interfacial axial, shear and normal stresses for a composite subject to thermal residual stresses in order to check predictions with the known elasticity solution. A key characteristic of the method is that the fibre can be represented by a series of cylindrical shells having the same property, and similarly for the matrix. Increasing the number of shells to represent the fibre and matrix leads to more accurate predictions of the stress field. Pagano and Brown [32] have extended the model so that it can deal with stress transfer arising from matrix cracks having planes normal to the fibre direction, and imperfect interfaces.

McCartney [24,25] has also developed an axisymmetric model of stress transfer using multiple cylinders to represent the fibre and matrix in a unidirectional composite subject to thermal residual stresses. The stress field representation differs from that assumed by Pagano [31] in that its form is fully consistent with the exact Lamé solution at large distances from an isolated fibre fracture. A key characteristic of the model is that it is shown how the displacement field may be calculated from the stress field providing a complete solution to stress transfer problems. In contrast Pagano's model provides only weighted averages for the displacement field. The model has been used to predict the interfacial shear and normal stresses, together with the axial stress distribution. Increasingly accurate solutions have been derived by using multiple cylindrical shells to represent the fibre and matrix. Calculations have also been carried out to investigate the effects of surrounding composite material on the local stress transfer associated with an isolated fibre fracture. It has been shown that stress transfer occurs over a much larger distance when the broken fibre and associated matrix are embedded in an infinite medium having the effective properties of the fibre reinforced composite. A characteristic of the model is that two of the stress-strain relations have to be averaged across each cylindrical shell. The model has recently been improved (McCartney [24]) so that it becomes necessary only to average the axial stress-strain relation. This introduces negligible approximation when a number of thin cylindrical shells are used to represent the fibre and matrix. A highly desirable characteristic of the modified model is that the stress and displacement fields can be shown to minimise the Reissner energy functional when carrying out a variational calculation (c.f. model of Pagano [31]).

2.4.3.2 Imperfect interface bonding

For some years models have been available (McCartney [33], Hutchinson & Jensen [34]) that attempt to predict stress transfer between fibre and matrix in interfaces on which frictional slip zones can occur as well as perfectly bonded regions. Frictional stress transfer was assumed to be governed by the Coulomb friction law. McCartney [33] assumed that the composite was frictionally bonded so that there was no adhesion between fibre and matrix; the interaction between fibre and matrix occurring through the action of frictional forces arising from thermal expansion mismatch effects that cause the matrix to be clamped onto the fibres. The models are essentially concentric cylinder models where one solid cylinder represents the fibre and a surrounding cylinder represents the matrix. The axial stress in the fibre is assumed to be independent of the radial coordinate, and similarly for the axial stress in the matrix. Thermal residual stresses are taken into account. The assumption that the axial stresses are independent of the radial coordinate is a rather restrictive assumption which will certainly be in significant error if the models are applied to low volume fraction composites (as in a fragmentation or pull-out test) or to bonded interfaces where a stress singularity must exist in the interface at the boundary between the debonded slip zone and the perfectly bonded region.

As the constant shear stress model of interface debonding has attracted a great deal of interest in the literature, the concentric cylinder model has been applied also to this case using the stress transfer model (McCartney [33]) that provides the stress and displacement fields that are consistent with Reissner's variational principle. In all shear-lag models the interfacial shear stress is set to a fixed value everywhere in the region of fibre/matrix debonding. This means that the models will lead to a shear stress on the surface of the fibre fracture, as the shear stress must be symmetric unless a singular term is included in the analysis. A radial dependence of the axial stresses is essential if using the model to interpret the results of fragmentation and pull-out tests.

As already mentioned above, Nairn's model [29] was developed to deal with imperfect interfaces where the interfacial shear and normal stresses are proportional respectively to the discontinuities in the axial and radial displacements at the interface. The model requires the specification of two interface parameters namely, D_s and D_n as described above. Pagano's model [31] has been used by Tandon and Pagano [35] to investigate the behaviour of debonded interfaces where stress transfer occurs arising from frictional slip governed by Coulomb's law where there is just one interface parameter; the coefficient of friction. The model has been used to consider frictional effects for fibre/matrix debonding associated with matrix cracks having planes normal to the fibre direction. The model has been used to indicate for matrix cracking the linear relationship of the debond length and the remote stress for various values of the coefficient of friction. Very good agreement is shown between model predictions and the earlier calculations of Kaw et al. [36] using a different modelling method. Stress transfer arising from fibre fracture can also be treated using the model.

2.4.3.3 FEA modelling

There has been extensive FEA modelling of interfacial behaviour. For example, in the case of the fibre pull-out test where interfacial debonding is regarded as being governed by energy release rates, references [37-40] describe some contributions that have been made. While the great majority of fibre fragmentation modelling has considered only elastic matrix behaviour, there are polymer composite systems for which matrix plasticity plays an important role. Unfortunately, the methods used to analyse the fragmentation problems with elastic matrices cannot be used when the matrix undergoes non-linear deformation (non-linear viscoelastic, plastic), arising from stress intensification caused by fibre fracture. Finite element methods have to be used (see for example [41]) where plastic flow has been shown to lead to interfacial shear stresses that are more or less uniform in the region of plastic deformation.

These results indicate that the constant interfacial shear stress model has some potential for being used to predict the effects on stress state of plasticity in the matrix.

The conventional fragmentation test can only be used to assess interface properties if the strain to failure of the matrix is at least three times that of the fibres [42]. In order to extend the range of materials for which the fragmentation test can be used, a new type of fragmentation test has been developed [42,43] where the interface between the fibre and a relatively brittle matrix can be assessed by embedding the two phase system in a third more ductile phase. The analysis of the system becomes more complicated and FEA has been used to understand the characteristics of the new test, and to derive interfacial properties by suitably using the model to interpret the results of fragmentation tests.

2.4.3.4 Energy Methods

While the level of fibre-matrix adhesion was traditionally evaluated by means of strength based approaches, an alternative consisting of the analysis of the energy release rate of a debonding interface was introduced recently by Nairn and Wagner [44-46]. This energy-based approach is strongly dependent on the stress analysis, and has consequently been upgraded several times, as the modelling effort constantly increased in the field. The main steps of the energy analysis were:

- shear-lag (Cox) based analysis [44]: neglected internal stresses and friction of the debonding interface, which resulted in a large underestimate of the interfacial toughness;
- Bessel-Fourier analysis [45]: the stress state was given by Bessel-Fourier series in a closed form which satisfies all boundary conditions. This model included internal stresses and friction, and is probably the most accurate up to now, provided the number of terms of the series expansion is large enough. However, the complexity of the analysis has limited its use in practical applications;
- shear-lag (Nayfeh) based analysis [46]: the Nayfeh shear-lag model was extensively reviewed by McCartney [47] and Nairn & Wagner [48] recently. These authors proved that it overcame most limitations of the Cox approach, particularly for low fibre volume fractions. As a result, the energy analysis was simpler, included internal stresses and friction. The model was successfully tested on several types of fibre-matrix systems [48]. One advantage was that reaching the saturation state was no longer required, as the model should be applied in the elastic range only. The major weakness of this method concerns the experimental determination of debond growth, usually an intricate task. Nevertheless, this method is probably the only energy-based approach that has any potential for application in an industrial environment.

2.5 Summary on Modelling Issues

The major conclusions to be drawn from the discussion on fragmentation modelling are as follows [22]:

Most methods currently used to interpret the results of interface tests in terms of interface parameters are based on simplistic stress analysis models that can at best lead only to nominal values for the interface properties. Such interface data should not be expected to show consistency when comparing results from different types of test, when non-consistent models are used. In addition such data should not be reliable when attempting to predict the behaviour of the fibre/matrix interfaces within a typical composite material;

As an example of conclusion 1, most analyses of fragmentation test results assume that the interfacial shear stress is uniform in the debonded (or yield) zones associated with

fibre fractures, and is a material constant. This assumption cannot be rationalised with constraints that are placed by the principles of mechanics. The technique for estimating the critical shear stress from fragmentation lengths is very dependent upon the model characteristics which cannot be justified in general because of the inconsistencies regarding the principles of mechanics;

- The fragmentation test is the only interfacial test technique that involves fibre fracture. The energy released during such fracture processes is such that dynamic effects may be important where stress waves may initiate more than one fracture at a given load, and lead to stick-slip processes within the interface debonding zone;
- Interface performance is best characterised by the consideration of interfacial fracture energies rather than interfacial strength, i.e. using fracture mechanics principles rather than a strength of materials approach. The issue of mixed mode loading arises and requires thorough investigation as currently used criteria for predicting mixed mode interface failure are empirical without any theoretical justification;
- Modelling stress transfer during interfacial failure needs to be accurate if the results of the tests are to have potential for use for predictions of behaviour in other situations, e.g. in a composite, or when analysing the results of different types of interfacial test method. Accurate analysis is best achieved by using axisymmetric uncomplicated geometries so that fragmentation testing and pull-out/push-through tests would seem to be preferred.

The model used in the following essentially contradicts some of these recommendations. The analysis, introduced as an 'Engineering Approach', is used because it is an approximation providing a useful alternative to the exact solution to the stress transfer problem, which remains unknown to this day. Furthermore, this approach has proven to be robust in the analysis of another test geometry, that of the microbond test.

3 ENGINEERING APPROACH: A MODIFIED SHEAR-LAG MODEL

3.1 Basic Model

In this work, a stress transfer model is used which was introduced recently [49]. Although approximate in common with all shear-lag models, it results in simple analytical solutions, gives an excellent determination of axial stress in the fibre, and has proven to be robust by comparison of predictions with other adhesion experiments. The cylindrical system used to model the stress transfer in the fibre geometry, and its main geometrical features, are represented in Figure . It involves a fibre of diameter $2a$, with Young's modulus E_f , embedded in a matrix having an external surface of diameter $2b$. The length of the fibre is $2t$. The r and z axes denote respectively radial and axial directions, oriented as shown in the figure. Stress transfer is considered as being two-dimensional, with symmetry about the plane $z = 0$. The complete derivation is reported in Reference [49]. To summarize, the hypotheses for stress transfer over a cylinder are:

- perfectly elastic and isotropic matrix and fibre
- perfect bonding between matrix and fibre
- constant stress through a radial section of fibre, and integral stress transfer from matrix to fibre by shear at the interface
- incompressible fibre compared to the matrix

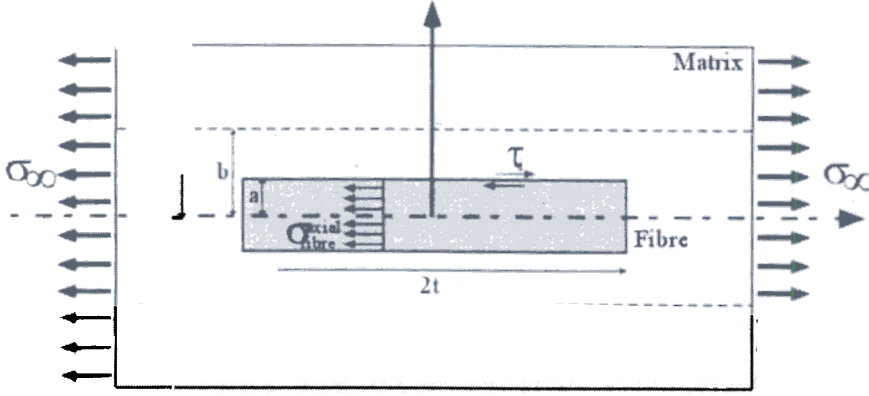


Figure 3 Geometry of the stress transfer model

- axial derivative of the axial stress in the matrix is graded in the radial direction by a function of $1/r$

elastic properties define the matrix boundaries a and b related to the stress transfer phenomenon, where a is the fibre radius and b satisfies $(b-a)E_m = aE_f$ if the sample radius b^* is greater than b , otherwise $b = b^*$.

It was shown that the average axial stress in fibre σ_f must satisfy the following differential equation:

$$\frac{\partial^2 \sigma_f}{\partial z^2} = \frac{a^2 E_f + (b^2 - a^2) E_m}{a(1 + \nu_m)} \left[\frac{\sigma_f - \sigma_\infty}{E_f} \right] \quad (7)$$

where σ_∞ is the applied stress (Figure), and κ is given by:

$$\kappa = \frac{12[2(b-a) + \zeta(b^2 - a^2)]}{a \left\{ 24a(b-a) - 16(b^3 - a^3) - 3\zeta(b^2 - a^2)^2 + 6b(2 + \zeta b)[2b^2 \ln(b/a) - (b^2 - a^2)] \right\}} \quad (8)$$

where

$$\zeta = \frac{E_m}{aE_f} \quad (9)$$

In the case of the cylindrical geometry depicted in Figure boundary conditions are given by

$$\sigma_f|_{z=\pm t} = 0 \text{ and } \tau = 0 \quad (10)$$

where τ_i is the interfacial shear stress, the solution to Equation (7) becomes:

$$\sigma_f = \frac{b^2 E_f \sigma_\infty}{a^2 E_f + (b^2 - a^2) E_m} \left[1 - \frac{\cosh(\beta z)}{\cosh(\beta t)} \right] \quad (11)$$

where

$$\beta = \left[\kappa \frac{(a^2 E_f + (b^2 - a^2) E_m)}{a E_f (1 + \nu_m)} \right]^{1/2} \quad (12)$$

Equation (11) provides a fully determined expression for axial stress along the whole fibre length. This equation, which resembles that proposed by Cox, is, however, free of any

adjustable parameter. Instead, it includes a structural parameter, κ , which is a function of the elastic moduli of the constituents and of the system geometry. The interface shear stress τ_i is derived using the force equilibrium on a dz element of fibre:

$$\frac{\partial \sigma_f}{\partial z} = -\frac{2}{a} \tau_i$$

and

$$\tau_i = \frac{a\beta}{2} \frac{b^2 E_f \sigma_\infty}{a^2 E_f + (b^2 - a^2) E_m} \frac{\sinh(\beta z)}{\cosh(\beta t)} \quad (14)$$

Figure 4 gives a representation of the two stresses σ_f and τ_i . This model was found to reproduce accurately several results reported in the literature [49].

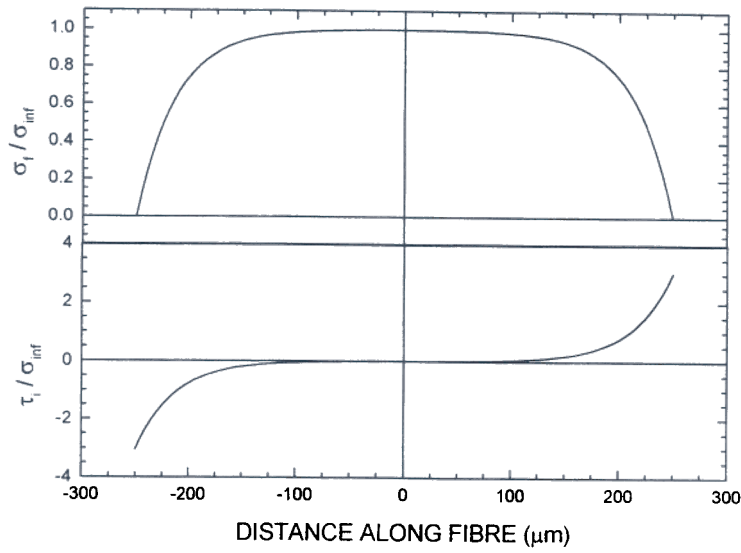


Figure 4 Fibre axial stress and interfacial shear stress resulting from the stress transfer model. Glass fibre/model epoxy network [49]. Stresses are normalised versus σ_∞

3.2 The Effect of Internal Stresses

It should be noted that the above derivation ignores the internal stress state of the fibre/matrix assembly, which would result from cure shrinkage of the resin, and also from the difference between thermal expansion coefficients (CTE). However, it was shown that residual stresses have to be taken into account in the analysis [6,50,51]. To do so, in shear-lag problems, the easiest way is to include internal stresses through the far-field displacements. Equation (11) is also written as:

$$\sigma_f = E_f \epsilon_\infty \left(1 - \frac{\cosh(\beta z)}{\cosh(\beta t)} \right)$$

In the simple case of elastic thermal stresses, the far field shrinkage is symmetric with regard to the centre of the fibre, and equals $(\alpha_g - \alpha_f) \Delta T$, where α_f and α_g are the coefficients of thermal expansion of respectively the fibre and matrix below its T_g . The far-field shrinkage, resulting from the contributions of ϵ_∞ , the applied strain, ϵ_i , the tension applied to the fibre prior to the cure, and thermal shrinkage, is introduced in Equation (15) by replacing ϵ_∞ by the sum of these contributions:

$$\sigma_f = [E_f(\epsilon_\infty + \epsilon_i) + K] \left(1 - \frac{\cosh(\beta z)}{\cosh(\beta t)} \right)$$

where $K = E_f(\alpha_g - \alpha_f)\Delta T$ is the internal stress parameter associated with this geometry. The interfacial shear stress thus becomes:

$$\tau_i = \frac{a\beta}{2}[E_f(\varepsilon_\infty + \varepsilon_i + K)] \frac{\sinh(\beta z)}{\cosh(\beta l)} \quad (17)$$

Therefore, a typical non-uniform interfacial shear stress profile is obtained, as described by Equation (17) and represented in Figure 5. It is evident that, upon introduction of the internal stress term K , the interfacial shear stress τ_i is increased at the fibre break.

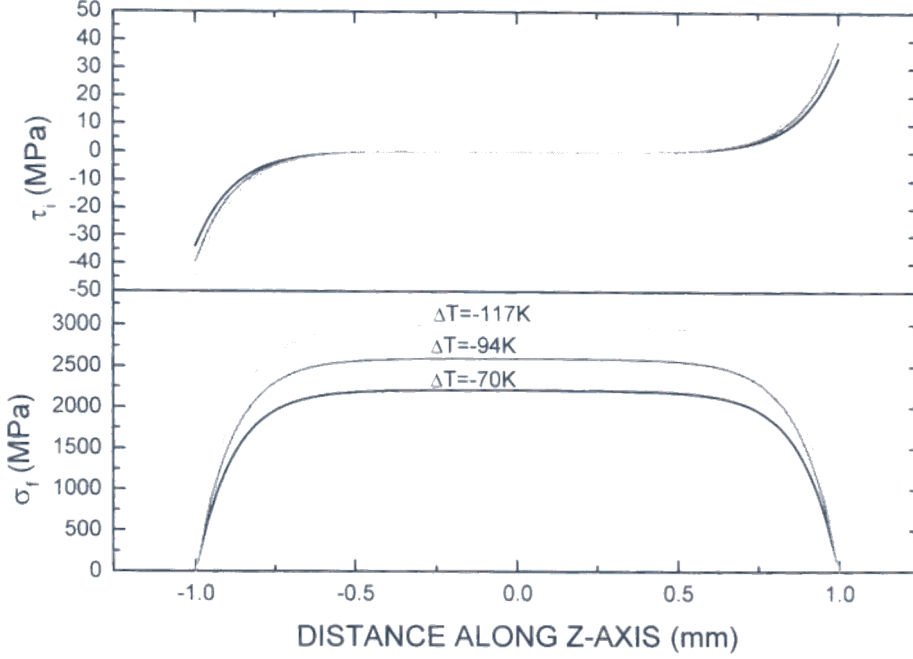


Figure 5 Theoretical interfacial shear stress obtained through the modified shear-lag model. The shear stress profile results from superposing the shear stress produced by external loading and internal stress. (Glass fibre / TGMDA-based epoxy [52], with $\Delta T = -117^\circ\text{C}$, -94°C , -70°C and $\varepsilon_0 = 0.02$)

3.3 Stress Transfer with Partial Debonding

During the fragmentation test, the most obvious failure process is fibre fracture, determined essentially by fibre properties. However, the interfacial failure plays a role too, since the ability to transfer stress is considerably reduced over a failed interface. For modelling purposes, a uniform friction is supposed here. In that sense, the geometry now involves two zones of debonded lengths m_l and m_r , respectively left and right, which are both smaller than the half length of the fibre. The uniform friction in these zones is assumed to be equal to $\pm\tau_{fric}$, taken as negative in the left part and positive in the right one. The axial and shear stress states are shown in Figure 6.

It is now necessary to solve the differential equation (7), with internal stresses, and with new boundary conditions, defined by:

$$\sigma_f|_{z=l-m_r} = \frac{2}{a}\tau_{fric}m_r \text{ and } \sigma_f|_{z=m_l-l} = \frac{2}{a}\tau_{fric}m_l \quad (18)$$

After calculation, the result is written as:

$$\sigma_z = \varphi_1 \cosh(\beta z) + \varphi_2 \sinh(\beta z)$$

with

$$\varphi_1 = \frac{\frac{2\tau_{fric}m_r/a + \psi_\infty}{\cosh \beta(t - m_r)} - \frac{2\tau_{fric}m_l/a + \psi_\infty}{\cosh \beta(t - m_l)}}{\tanh \beta(t - m_r) - \tanh \beta(t - m_l)}$$

and

$$\varphi_2 = \frac{\frac{2\tau_{fric}m_r/a + \psi_\infty}{\sinh \beta(t - m_r)} - \frac{2\tau_{fric}m_l/a + \psi_\infty}{\sinh \beta(t - m_l)}}{\frac{1}{\tanh \beta(t - m_r)} - \frac{1}{\tanh \beta(t - m_l)}}$$

where

$$\Psi_\infty = E_f(\epsilon_\infty + \epsilon_i) + K$$

As shown in Figure 6, the maximum interfacial shear stress is considerably reduced by debonding. As such, it is supposed in this work that the interface fails when its intrinsic shear strength, that is the local shear strength of the interface, is reached. A uniform friction zone is obtained over the failed region. Following this analysis, the closed-form obtained is easily implemented in the following simulation of the fragmentation process, using a Monte-Carlo method.

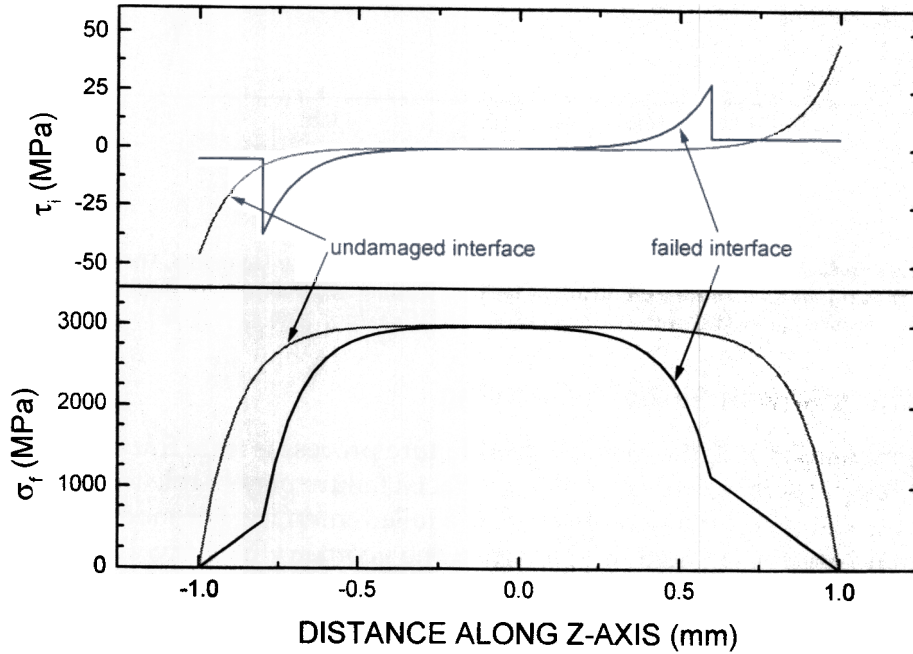


Figure 6 Stress transfer along fibre with debonded ends: shear and axial stresses. Glass fibre / TGMMA-based epoxy [52], thermo-elastic approach with $\Delta T = -70^\circ\text{C}$, $\epsilon_0 = 0.02$, and $\tau_{fric} = 5\text{MPa}$, $m_l = 0.2\text{mm}$, $m_r = 0.4\text{mm}$

4 A MONTE-CARLO SIMULATION OF THE FRAGMENTATION PROCESS

4.1 Principle

Figure 7 shows the flowchart used to devise the program for Monte-Carlo modelling of fibre fragmentation. The process is classical, as described in the following. First, the fibre is discretised in a certain number of sites of equal length (10000 in the example), which are all attributed an ultimate strength, selected at random from a two-parameter Weibull distribution, where the strength is a function of the length of the segment l as given by:

$$\sigma_{ult}(l) = \sigma_0 l^{\frac{1}{m}} \Gamma\left(1 + \frac{1}{m}\right) \quad (23)$$

where σ_0 and m are respectively the scale and shape parameters of the Weibull distribution, and Γ is the Gamma function.

Initially there is just one fragment of length L_0 , and the load is increased until either interfacial failure at the ends of the fragment is reached, or the ultimate axial strength of one site is reached. In the latter case, generally several sites have a lower strength than the local axial stress resulting from loading. A list of all these sites is built, from which an element is

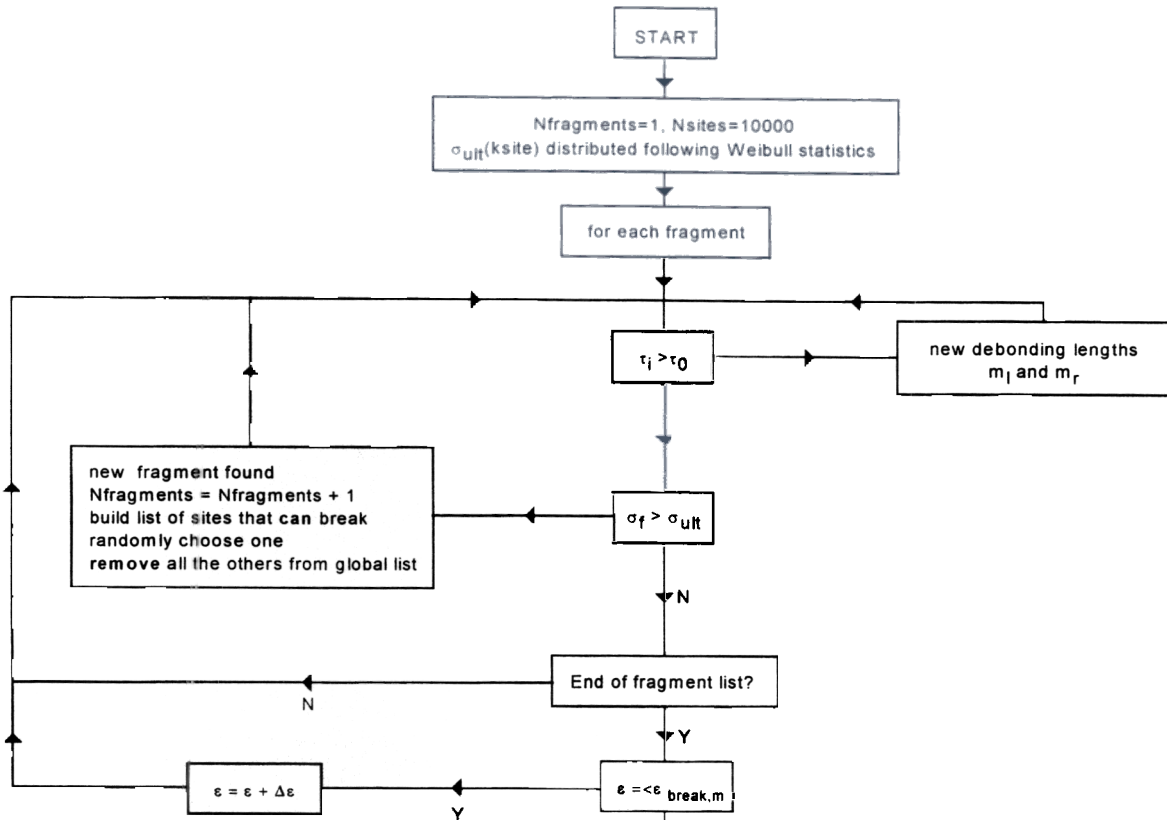


Figure 7 Fragmentation simulation flow chart

picked randomly. Two fragments are formed, and all the sites from the list having a strength lower than that of the site randomly selected are deleted from the list (by giving them an infinitely large strength). The two fragments formed are tested again versus Weibull statistics, and the process is iterated until the fragments formed cannot fail any more. If the strain is lower than the strain at break of the resin, it is incremented by a strain step $d\epsilon$ and the whole process is iterated.

Validation checks are conducted on the results obtained:

- for a given gage length L_0 , the final number of fragments and the average crack length are independent of the number of sites if it is greater than a certain value. In practice, N_{sites} was fixed by $L_0/N_{sites} = 10^{-3}$. This empirical equation, determined by trial and error, was proven to give a sufficient accuracy;
- when the Weibull shape parameter is given a very large value, i.e. all the sites have the same strength that is equal to the Weibull scale parameter, one failure is observed, while the $(N_{sites} - 1)$ remaining sites are all given infinite strength

All the implementations were realised using Python2.1, which allows the use of a standard random number generator based on the standard Wichmann-Hill algorithm, combining three pure multiplicative congruential generators of modulus 30269, 30307 and 30323. Its period (how many numbers it generates before repeating the sequence exactly) is 6,953,607,871,644 [53]. This algorithm is used both for distributing strengths along the fibre sites, and selecting the failure location from the list of possible sites failing.

Although relatively basic, this simulation is consistent with the experiment, where a step-wise loading is imposed by increasing the strain of the testpiece. Provided the fibre failure is governed by axial strength only, this should reproduce the fragmentation process accurately.

For each computer run, the evolution with applied load of the number of fragments and of the mean fragment length $l_{c,av}$ are obtained. The distribution of fragment length at saturation, if reached, is also recorded. By doing so, the objectives are:

- to examine the validity of several strength based methods of data reduction. In particular, the reduction algorithm from Hui, Shia and Berglund [54] (denoted HSB in the following) is analysed in detail;
- to compare the results of the simulation to those of experiments realised in the laboratory. The main interest here is the reproducibility of the test.

The results are examined in the next section.

4.2 Results

The parameters used in the simulations of a typical CFRP are presented in Table 1

Table 1 Parameters used in the simulation of the fragmentation test

	Simulation 1 (S1)	Simulation 2 (S2)
N_{sites} (-)	25000	10000
L_0 (mm)	50	20
W (mm)	4	4
t (mm)	1	
a (mm)	3.5E-3	3.5E-3
E_f (MPa)	208E3	208E3
α_f (1/K)	-1.10E-6	-1.10E-6
E_m (MPa)	3200	
ν_m (-)	0.35	
α_g (1/K)	7.90E-5	7.90E-5
ΔT (K)	-100	-100

For this study, the only parameters changing are the gage length and the number of sites. Additionally, the Weibull parameters of the fibre were taken from reference [55], with $\sigma_0 = 3.3$ GPa and $m = 7$. This corresponds to a brittle behaviour, which is generally observed with carbon fibres.

4.2.1 Strength Based Data Reduction Scheme

Figure 8 presents the results of 5 runs of the simulation with the parameters S1 in Table 1

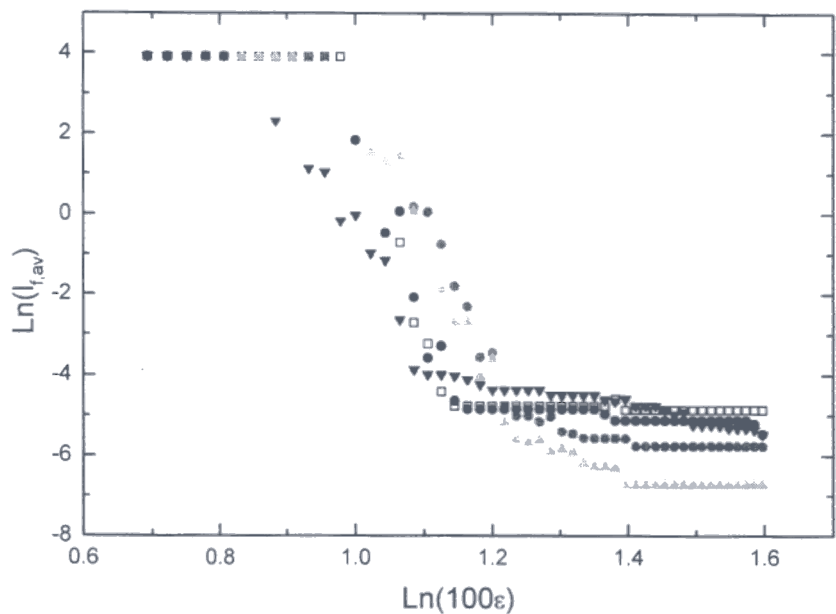


Figure 8 Mean fragment length vs. applied strain. Data are plotted following HSB recommendations

One immediate remark is that the curves do not superpose. To understand this result, one must bear in mind that the simulation is based on a fully stochastic process. This means that after the run the distribution of fragment lengths should be stochastic too. As a result, five runs give five widely different results. It will be shown in next section that experimental results show exactly the same trend. Remember also that in this simulation, contrary to others, the number of sites chosen is large, and the fragment is not forced to fail at its mid-point. This last restriction is present in most works in the literature.

Before drawing any conclusions on this test, it is useful to discuss the HSB method. The data reduction scheme proposed by these authors is based on a linear regression of that part of the data in Figure 8 in the small strain fragmentation regime. Effectively, taken on a small scale, the simulation indicates that the slopes are quite close. In fact, from the linear regression, it is claimed that the Weibull parameters can be obtained. Plots of the results for the pseudo-linear region are presented in Figure 9. This plot allows us to establish that the HSB reduction scheme is inadequate when obtaining the Weibull parameters in the pseudo-linear regime. The major reason for this is that it is not possible to obtain such parameters from this type of plot. Effectively, one set of parameters generates five completely different results, so that the process cannot be inverted. In experimental work, it is very tempting to attribute the deviation from the linear behaviour to experimental variability, and obtain a best fit. However, the results of the simulation clearly show that **it is not possible to reduce fibre parameters from the average fragment length**. It is therefore strongly recommended to obtain the Weibull parameters of the fibre by other means, for example testing in air at different gage lengths. The best solution is nevertheless to test fibres in the matrix, using the single fibre composite, but to keep only the first break as characteristic of the Weibull plot. This unfortunately needs an extensive number of experiments.

Moreover, the HSB plots shown in Figure 8 are used by these authors [54] to obtain, after translation, master curves. One justification is that there are different internal stress levels or

gage lengths etc. requiring such translations. This is not the case here, so that the curves cannot be superposed. Finally, one common interpretation of such a plot is to say that the lowest curve, presenting the smaller fragment length at saturation, corresponds to the best adhesion, while the highest $l_{c,av}$ represents a poorest adhesion. This statement is also completely false, as the five runs were generated using the same interfacial shear strength. Therefore, although adhesion plays an important role in determining the final value of the average fragment length at saturation, it is impossible to determine an adhesion value from this information only.

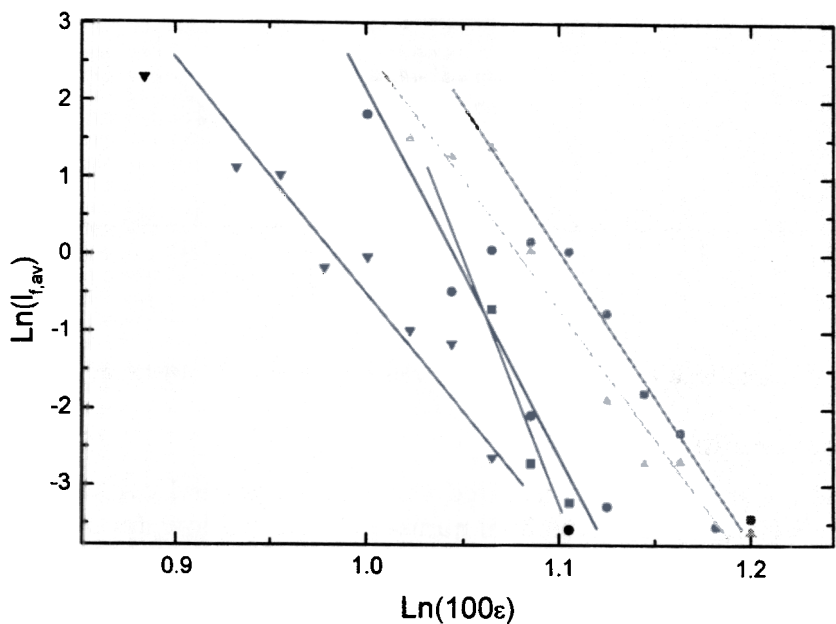


Figure 9 Mean fragment length vs. applied strain, magnification of the pseudo-linear regime presented in Figure 8

4.2.2 Simulated Influence of the Gage Length

Figure 10 shows the results obtained for the short gage length simulations (S2). As stated above, there is no influence of the gage length on the simulation results, provided the number of sites is large enough. This is clearly confirmed by Figure 10, which presents the same features as observed previously. When the two plots are superposed, the values, once the fragmentation process is started, are in the same range. The envelope of the fragmentation curves in Figure 8 and Figure 10 match closely. This is particularly true for the mean fragment length at saturation. Notice however that the low strain plateau values are different, since they represent the length of the initial fragment, which is equal to the gage length.

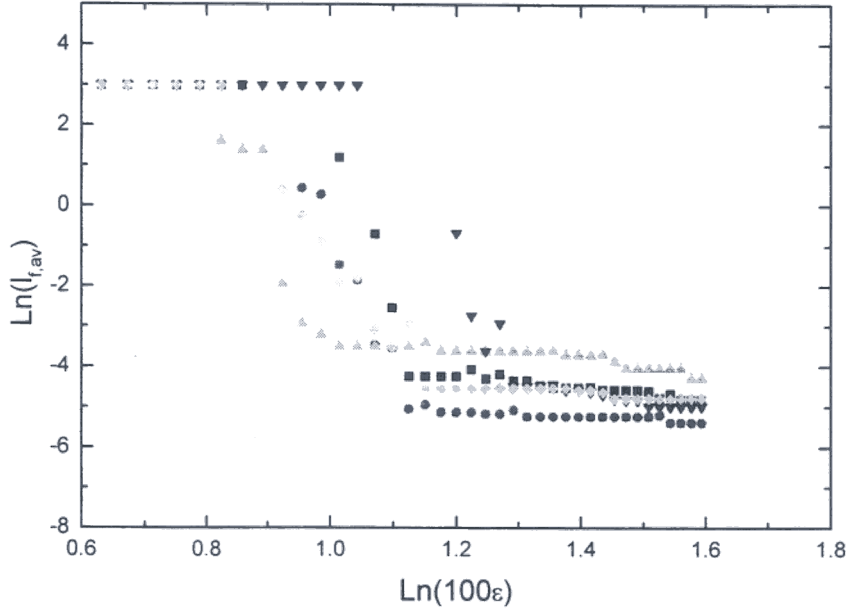


Figure 10 Mean fragment length vs. applied strain, for the S2 simulation

4.2.3 Experimental Results vs. Simulation

Figure 11 presents a comparison of simulated versus experimental curves. A good agreement is obtained. The dispersion of the final number of cracks is low, for both experimental and simulated results. However, this is not the case when the number of fragments starts to increase, where fairly different behaviours between samples are obtained, in both cases. In addition, the simulated curves have been obtained by trial and error on material properties and internal stress level, trying to reproduce at best the behaviour of real specimens. The result is satisfactory, with the result that this method can be used to characterise the Weibull parameters of the embedded fibre, as well as the internal stress level. These are the parameters that were varied to obtain the best fit here. Since the simulation is based on an axial strength method, with a satisfactory model describing the axial stress in fragments, information can be obtained **only** on axial properties of the fibre/matrix assembly.

The values obtained, $\sigma_0 = 3.25$ GPa and $m = 3.4$, are in good agreement with a previous study [55]. The Weibull shape parameter $m = 3.4$ is a quite low value for HTA carbon fibres, it is usually supposed to be in the range of 5. It is smaller than the reference value, obtained in air, of 5.3. This strengthening of individual fibres when they are embedded in a matrix, characterised by a decrease of the Weibull shape parameter, is commonly observed for a wide range of systems. It is thought to arise because of the elimination of surface defects when the fibre is surrounded by a matrix. One must nevertheless be careful when drawing such conclusions, because the test does not allow the accurate determination of the Weibull shape parameter. For a very brittle fibre, there is little influence of the adhesion parameter on the test. This was verified by varying the values of τ_0 and τ_{fric} between 40 and 70 MPa, and 5 and 20 MPa, respectively. When the Weibull parameters were estimated, it was decided to fix these values at $\tau_0 = 50$ MPa and $\tau_{fric} = 10$ MPa, following results of other studies. The method used does not provide an absolute value for the shear parameters anyway. An extended modelling is needed, as described in the remainder of this report.

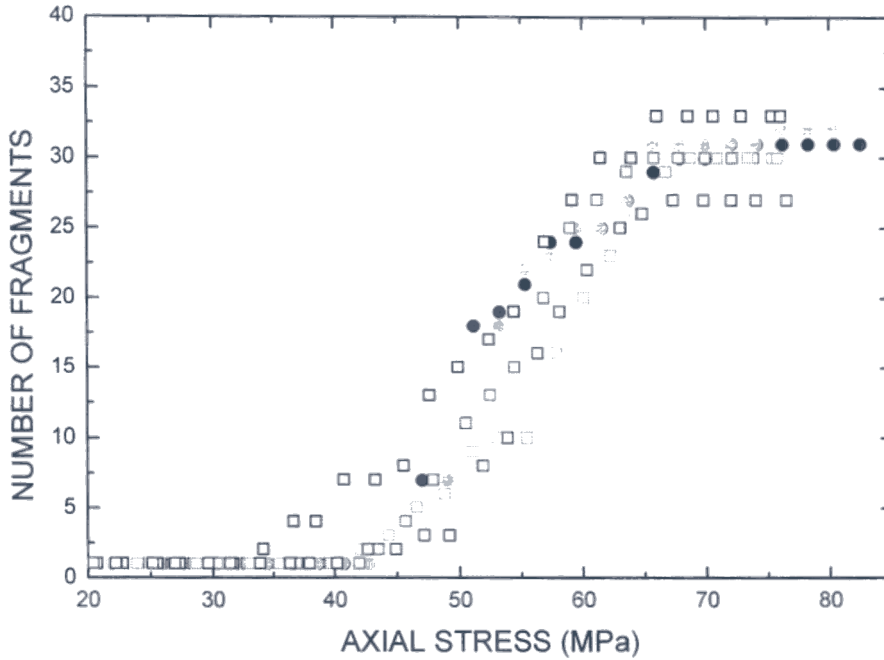


Figure 11 Simulation gauge length =45mm, high strain, circles are simulations and squares experiments.

All the input parameters are coupled and it is difficult to establish their effect when they are varied independently. Nevertheless, the influence of internal stresses on the value of the shape parameter is dominant. By omitting internal stresses one would obtain a higher shape parameter (7 in the present case).

A last question can be answered here: can the data simulation scheme be used to obtain systematically the fibre characteristics? As shown here, the answer is possibly. With some effort, one can devise parameters that allow the matching of the behaviour of the embedded fibre when fragmenting, as illustrated here. However, the nature of both simulations and experiments is statistical, and statistical tools should therefore be used to prove that this method is valid. In this sense, we propose that a Savitsky-Golay filter is used as a figure of merit to test whether the distributions of fragment sizes are equivalent or not, and whether the number of fragments versus axial stress curves are equivalent or not.

To do so, about 5-10 simulations per sample are needed. A figure of merit is calculated for each simulation. The best fitting values are then sought within a certain possible range by means of a factorial design algorithm. This process is iterated until a certain accuracy is reached, which is evaluated using a Student t-test for example. To summarise:

- the fixed inputs are: $N_{sites}, L_0, t, W, a, E_f, E_m, \nu_m, \alpha_f, \alpha_m, (\tau_0, \tau_{fric})$;
- the variables are: $\sigma_0, m, \Delta T, (\tau_0, \tau_{fric})$.

This should provide an automated way to obtain at least σ_0 and m . The internal stress characterisation and its main parameter ΔT can be obtained by a separate method anyway.

Based on these theoretical analyses, it is possible to discuss several features of the test in terms of local stress analysis. The experimental study conducted includes the study of the effects of geometry of the testpiece, strain rate, pre-load applied to the fibre. Based on the

results of this screening study, two systems, with carbon fibres or glass fibres, were studied in detail. The results are presented in the next section.

5 EXPERIMENTAL RESULTS

5.1 Screening Study

The first set of experiments involved one screening study, from which the optimum experimental conditions were determined. The parameters being varied were:

- geometry of the testpiece: the gage length, the width W and thickness t ;
- strain rate: stepwise loading with increments of 0.1%, 0.2% strain, and two delay times before observation, 10min or 5min. Constant strain rate measurements were also performed, at 0.1 and 0.01 mm/min;
- load F_0 applied to the fibre prior to curing the testpiece: 0.2g and 0.8g.

The results for the screening study are presented in Table 2. It was decided to consider the influence of external parameters on the average fragment length, the onset of fragmentation and the stress needed to reach saturation. The most classical methods to analyse the test data use only the average fragment length, but the onset and saturation stresses can be used to calibrate the model in order to fit the results of the stochastic simulation, as explained above.

The first important remark concerns the way the mean fragment length $l_{c,av}$ is obtained. A relatively small difference is shown between the two columns of Table 2, where column **a** is the exact result and column **b** is the usual approximation made in the literature, which is to divide the gage length by the total number of fragments. In all sets of testpieces, negligible differences were found, with $l_{c,av}^b$ approximately equal to the true value. However, the fragmentation process involves the formation of a fibre crack, which is accompanied by matrix cracking and/or yielding, between the two fragments formed. This phenomenon is time dependent and length dependent, and can be ignored when the length of each fragment is measured. Notice that an energy approach would have to account for these phenomena, by including them in the energy balance. Such an analysis has been carried out by Nairn and co-workers recently [45]. Following this approach, the energy release rate of a debonding interface should be obtained independently of the loading conditions and the gage length. For now, it is sufficient to remember that any strength-based approach based on the mean fragment length obtained by dividing the gage length by the number of fragments is useless, as will be shown in the following, since an analysis of local strength is required.

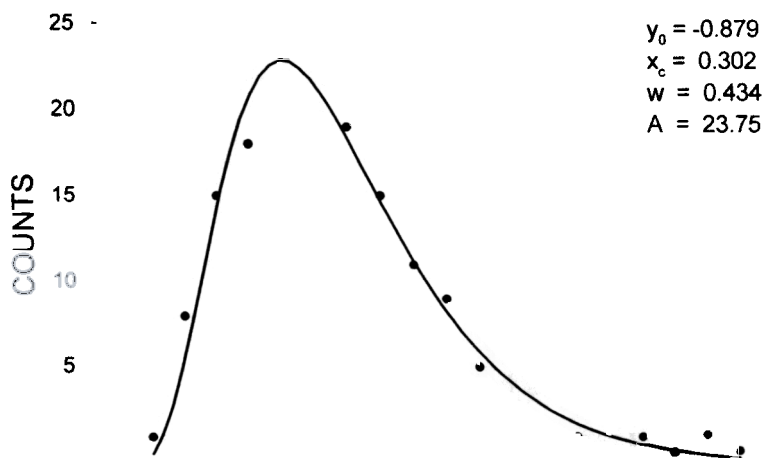
This is well supported by the fact that there is apparently no influence of the external parameters (geometry and load) on $l_{c,av}^a$, which is constant and equals approximately 0.39. The distributions of fragment lengths additionally present the same patterns, and can be approximated by a log-normal behaviour: Figure 12.

No influence of the thickness and width of the testpieces could be established from Table 2. Using the simple modified shear lag approach described in section 2, the dimension (either width or thickness) of the specimen that should be used to change the axial stress response should be such that $b \leq aE_f/E_m \approx 350\mu m$. The smallest dimension used here was a thickness of 1mm, which implies that $b = 500\mu m$. Therefore, the independence of the results upon the variation of dimensions is perfectly justified.

Table 2 Results of the screening study

	L_0 (mm)	W (mm)	(mm)	$\dot{\epsilon}$ (mm/min)	F_0 (g)	$l_{c,av}^a$ (mm)	$l_{c,av}^b$ (mm)	σ_{onset} (MPa)	σ_{sat} (MPa)
1	25	4	2	stepwise 0.2%, 10'	0.2	0.39 ± 0.07	0.40 ± 0.07	$37 \pm$	59 ± 4
	25	4	1	stepwise 0.2%, 10'	0.2	0.39 ± 0.02	0.40 ± 0.02	36 ± 5	61 ± 2
3	25	4		0.1	0.2	0.35 ± 0.01	0.36 ± 0.01	40 ± 6	69 ± 3
4	25	4	1	0.01	0.2	0.37 ± 0.06	0.38 ± 0.06	36 ± 6	61 ± 4
5	25	4	1	stepwise 0.1%, 5'	0.2	0.36 ± 0.01	0.36 ± 0.01	35 ± 4	60 ± 3
6	25	4	1	stepwise 0.1%, 2.5'	0.2	0.37 ± 0.08		37 ± 7	63 ± 4
7	25	2	1	0.1	0.2	0.35 ± 0.03		42	69 ± 3
8	25	3	1	0.1	0.2	0.33 ± 0.03		38	68 ± 1
9	25	5	1	0.1	0.2	0.39 ± 0.11		38	66 ± 6
10	35	4	1	0.1	0.2	0.33 ± 0.05		42	70 ± 1
11	15	4	1	0.1	0.8	0.35 ± 0.01		30	63 ± 1
12	45	4	1	0.1	0.8	0.35 ± 0.04		39	65 ± 1
13	15	4	1	0.1	0.2	0.36 ± 0.04		32	68 ± 2
14	45	4	1	0.1	0.2	0.40 ± 0.06		40	66 ± 2

a. each fragment length determined under the microscope, and averaged
b. determined classically by dividing the gage length by the number of fragments



On the other hand, an influence of the gage length was found on the onset and saturation stresses, when the gage length was equal to 15mm. All the testpieces had the same radius of curvature, i.e. at the section area change was not changed with the gage length, as defined in the standard NIST procedure proposal, modified in that work [56]. The difference in test-piece behaviour is clearly pointed out by the present study, and is due to geometrical effects. The necking zone of the testpiece is of the same dimensions, but the stress concentration is different, and when the gage length becomes small, it globally influences the stress state of the part of the testpiece under observation. For larger gage lengths, there is little influence of geometry but a large gage length is more suitable for statistical studies, the population of fragments being more representative of the interaction between defects.

The effect of the pre-load is not very marked, and a larger pre-load would be needed to see an effect on the mean fragment length or the onset stress. Such loads are experimentally out of reach, because of the brittle nature of carbon fibres. Nevertheless, applying a pre-load helps to maintain the fibre in place and keep it straight during testpiece manufacture. It possibly eliminates most unwanted changes in the alignment of the fibre, and therefore results in lower standard deviations in all measured quantities.

Finally, a remarkable outcome of the screening study is that it demonstrates the possibility of having continuous measurement of the fragmentation process. This study shows that it is completely impracticable to carry out the measurement using a stepwise method, with a time before observation of 5 or 10 minutes. First, this time allows for stress relaxation to occur, which further complicates the analysis. Second, the characteristic measurements carried out on the testpieces were not influenced by the change of strain rate. Notice that the viscoelastic behaviour of the resin, far from its glass transition temperature, is not a sensitive parameter in this study. A highly viscoelastic matrix would typically show different behaviours, because of the change of Young's modulus with the strain rate.

Following the trends obtained through this screening study, the following parameters were chosen for the quantitative analysis described in the next section:

- the gage length of 25mm was chosen, for practical reasons, although a gage length of 45mm would have been preferable, so that the distributions of fragment length were more statistically representative;
- the width and thickness of the testpieces were set to 4 and 2mm respectively, for practical reasons (ease of handling and moulding of the testpieces);
- the pre-load was chosen as high as possible. In the following it was set to 0.2g for carbon fibres and 1.6g for glass fibres;
- a constant strain rate of 0.1mm/min was chosen, as it led to an acceptable experimental time, which was not insignificant when a large series of testpiece measurements were involved.

5.2 Quantitative Analysis

5.2.1 Evaluation of the Internal Stress Level

The importance of internal stresses in terms of local shear stress has already been highlighted in Figure 5, where it was shown that the shear stress at the ends of the fibre is largely increased by internal stresses. Other studies have shown the importance of accounting for internal stresses correctly when analysing adhesion measurements [57, 58]. They recommend the decomposition of the local stress state into two terms, one representing the contribution of the applied load, to which is added the contribution of internal stresses. It was

further shown that practical adhesion, corresponding to the applied load term, could be doubled in certain systems by controlling the internal stress level. Although it is not the primary goal of this report to determine the effect of internal stresses on adhesion measurement by the single fibre fragmentation test, it is useful to identify the sources of internal stresses, prior to describing how they were accounted for in the analysis.

Internal stresses build up during processing of thermoset systems as a result of [52, 59]:

- chemical shrinkage of the resin during curing: the composite with resin in a liquid state is brought to an elevated temperature, above or below the bulk resin infinite T_g , which, while curing, builds up stiffness and shrinks at the same time. In the present work, the cure of the resin took place far below the ultimate T_g of the resin, without any post-cure. The resulting Young's modulus of the resin is high at the cure temperature, as well as the relaxation time. As a result, chemically induced internal stresses are not negligible for this particular system and geometry;
- thermal shrinkage during cool-down from the cure temperature to ambient or an aging temperature, since the resin and its reinforcement usually do not have the same coefficients of thermal expansion.

Depending upon the resin behaviour, the following evaluations of the internal stress level during cool-down of the composite are possible:

- (A) Elastic stress: as a first approximation, the internal stress level can be derived from a temperature-independent, thermoelastic stress analysis [60]. This simple case has been widely used in the literature, although it clearly corresponds to an upper bound to the real internal stress level. It assumes that the Young's moduli of the resin and fibre, and their coefficients of thermal expansion are constant over the temperature range ΔT . This gross approximation is particularly inaccurate close to the glass transition of the polymer, which generally involves an abrupt change in Young's modulus.
- (B) Thermo-elastic stress: it is important to note that Equation (17) includes the matrix stiffness through the parameter β . When the matrix stiffness depends on temperature, the evolution of E_m with temperature can be included by integration during cool-down. In most cases, the coefficient of thermal expansion of the resin below its glass transition temperature, α_m , can be considered constant within a few percent error. A typical result of this approach is to decrease the level of internal stresses. This approximation is, however, independent of the cooling rate, whereas most polymers are well known to exhibit viscoelastic behaviour beneath their glass transition temperature, with associated stress relaxation. Relaxation during cool-down, as well as at the equilibrium temperature must therefore be introduced in order to more accurately describe the internal stress level;
- (C) Thermo-viscoelastic stress: a classical mechanical model presented to describe the relaxation behaviour of polymers is based on a parallel combination of Maxwell elements, yielding the elementary relaxation response of an element i [61] as a function of time and a relaxation time λ_i . The relaxation time generally follows a time-temperature superposition following the Williams-Landel-Ferry (WLF) law [61];
- (D) Thermo-viscoelastic including aging stress: a simple linear viscoelastic law, i.e. an exponential retardation function is not sufficient to describe the salient features of structural recovery [62, 63], and the stress relaxation modulus $E(t)$ of a quenched and annealed amorphous glass changes not only with the load time t but also with the aging time t_e . If one cools an amorphous polymer from above to below

its T_g , and then keeps the temperature fixed, the volume of the material evolves spontaneously towards equilibrium [64]. Such behaviour is also obtained if the enthalpy instead of the volume is measured, so that the evolution of the glassy polymer's thermodynamic state has been termed structural recovery. This change in internal state is characterised by some physical changes, such as the engineering properties of the polymer [65-68]. The sum of these changes has been termed physical aging, to distinguish it from chemical aging, since all the changes are completely reversible when the polymer is reheated to above its T_g [62]. These are readily described using a multiple ordering parameter model to define the viscoelastic response function for the polymer in glassy state, and describe the volume departure from equilibrium. It was shown by Struik that physical aging involves a global shift of the spectrum of relaxation times towards longer times [62].

One important feature is the considerable difference between the thermo-elastic and thermo-viscoelastic approaches to thermal stresses. A thermo-elastic approach provides an upper bound to the internal stress level. Furthermore, large differences appear when the material is held at an aging temperature prior to quench to a test temperature.

A Diglycidyl Ester of Bisphenol A (DGEBA) epoxy resin, cured by a metaphenylene diamine (mPDA) in a stoichiometric amount, was used in this study of single-fibre fragmentation. The cure cycle was chosen as described in previous studies, i.e. 2 hours at 75°C , followed by 2 hours at 125°C . The curing agent is a tetrafunctional aromatic diamine, which are well-known to give rise to high internal stresses [69]. The resulting properties of the resin were taken from Refs. [70-72].

A complete study of cure kinetics and build-up of internal stresses was carried out by Gupta and Brahatheeswaran [70-72]. The internal stress after cure and cool-down to room temperature was obtained by the bi-layered beam bending technique [72]. This involves curing of a thin film of epoxy coated on a flat aluminium strip, and the measurement of the deflection at the centre of the beam after the processing cycle. These authors measured the internal stress level after 12 hours (cool-down time), and subsequently after periods of 24 hours, for 3 days. The results they obtained are reproduced in Figure 13.

A comment is first needed on the values presented in Figure 13, which shows results for both a standard cure and a standard cure followed by a post-cure. It is shown that the effect of the post-cure is not negligible and profoundly affects the relaxation behaviour of internal stresses. The resin, after standard cure, is not therefore completely cured. When stored at room temperature, the cure continues, and physical aging is present at the same time, because the resin is held below its T_g . Both these phenomena tend to increase the Young's modulus of the resin, and to slow the relaxation of internal stresses. Eventually, at longer times the trend may even reverse, i.e. additional internal stresses are generated. Thus, the behaviour of this resin is intricate, and although it has been used in several studies, the full characterisation of its long-term properties has not been achieved. Without more information, a reasonable assumption is to take the last point of the plot in Figure 13 (standard cure) to be a reliable value for the internal stress level at any longer time. The error should be kept within 10%, as the relaxation phenomena compete with aging and post-curing effects.

The next logical step is to render this information independent of the geometry, by obtaining an effective temperature difference ΔT_{eff} corresponding to this internal stress level for the particular beam bending geometry. This procedure has been validated recently for both cylindrical and planar geometries [57]. The internal stress level is calculated from:

$$\sigma_i = E_m(\alpha_m - \alpha_{al})\Delta T_{eff} \quad (24)$$

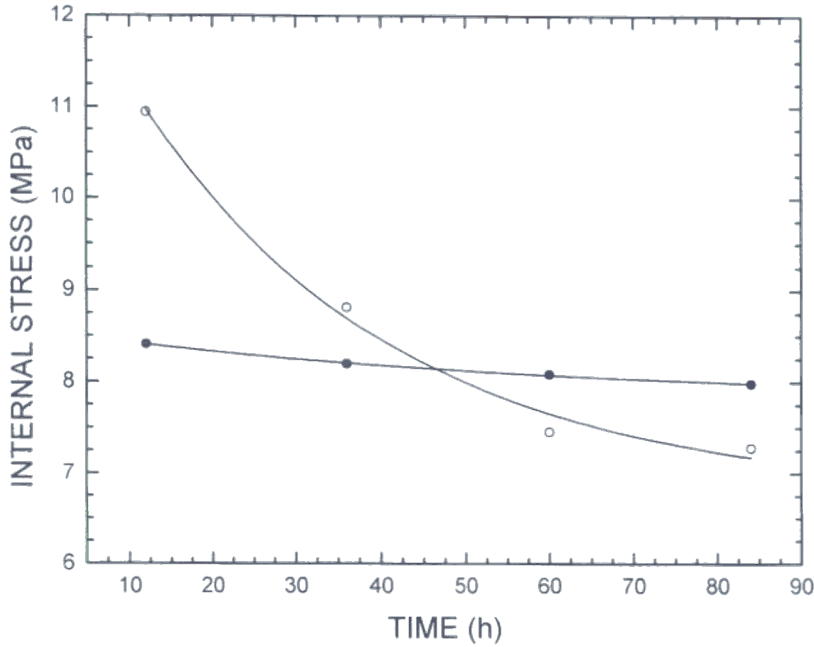


Figure 13 Internal stress data for standard cure (solid circle) and post cure (open circle) bi-layered beam bending specimen. The post cure was performed at 175°C for 6 hours (Reproduced from Ref. [71])

where $\alpha_m = 5.20 \times 10^{-5} \text{ cm/cm/K}$ and $\alpha_{al} = 2.30 \times 10^{-5} \text{ cm/cm/K}$ are the linear coefficients of thermal expansion of the resin and aluminium, respectively [71,72].

The Young's modulus of the resin was evaluated during the fragmentation experiments, and found to have the value $E_m = 2.61 \text{ GPa}$.

A value of $\Delta T_{eff} = -96.1^{\circ}\text{C}$ was obtained, a reasonable value compared to -105°C which is imposed during cool-down. In spite of the marked relaxation behaviour of the resin, The values are relatively close. However, it must be remembered that this internal stress level includes cure stresses, and that ΔT_{eff} is therefore **not** a stress-free temperature.

5.2.2 Effect of Surface Quality on Adhesion

The effect of surface quality on adhesion was investigated with carbon fibres single fibre fragmentation tests. Under the test conditions described in the previous section, 3 systems were tested: a reference system (A1), a system where the carbon fibre has been coated with a mould release agent to simulate the effect of grease deposition on the fibre (A2), and finally a reference system with the testpiece conditioned in boiling water for 24 hours (A3).

From the preceding discussions, results need to be analysed locally, i.e. only a local strength can be determined at that point, the average strength being meaningless. To do so, the following approach was adopted. The longest fragment resulting from each test was analysed, because it gives rise to the higher shear stress. For this fragment, the maximum load corresponding to the onset of saturation was used in the calculation and the debonded length was measured using an optical microscope. The length and diameter of the fibre were obtained through the procedure detailed in reference [73].

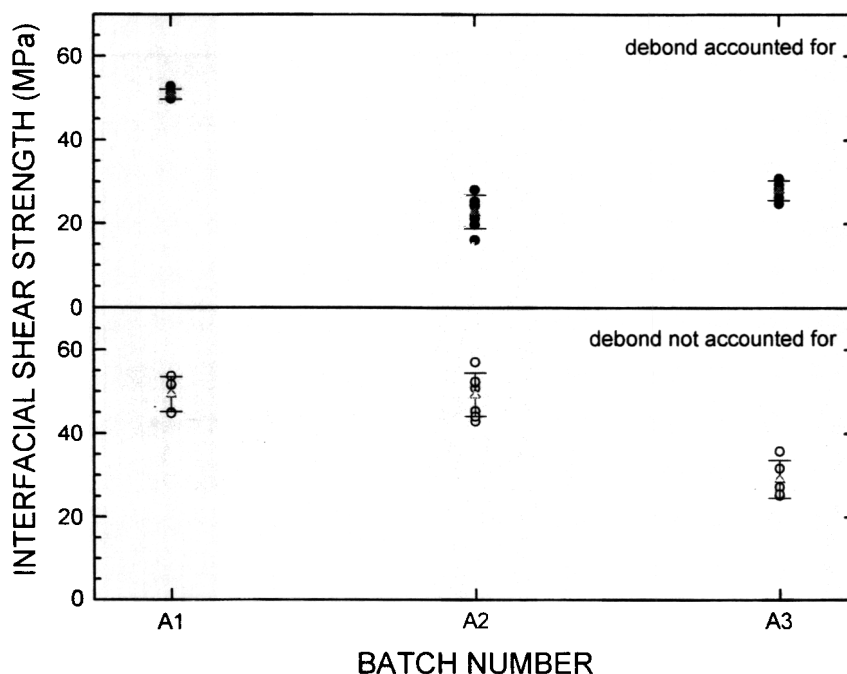


Figure 14 Interfacial shear strength for various treatment and conditioning of the carbon fibre - DGEBA/mPDA single fibre fragmentation testpiece

The maximum interfacial shear stress, arising at the extremity of the fibre, was calculated following two different methods, namely with or without accounting for debonding. The results are presented in Figure 14.

First of all, it is important to notice that if internal stresses were not accounted for, one would obtain interfacial shear strength of about 70MPa for the reference batch, which is greater than the matrix's shear yield stress. The values obtained here, of about 50 MPa, still are quite high, but are likely to be representative of this system, since the carbon fibres are sized to enable a good wettability by the resin, and this sizing (γ -APS) forms some covalent bonds with the resin. Moreover, the roughness of carbon fibres tend to promote adhesion by enabling mechanical interlocking, which is not present with smooth fibres, e.g. glass fibres.

It is shown that upon treatment by a release agent, the interfacial adhesion is decreased by more than a half in the upper plot. The influence of debonding is perfectly illustrated, with two completely opposing results if debonding is accounted for or not. This is due to the long debond length which promotes a long fragment length in this case. Thus, if debonding was not accounted for, the long fragment length would correspond to a high shear stress at the ends, and one would mistakenly conclude that adhesion is promoted by the use of a release agent.

When the testpiece is exposed to boiling water, a classical result is obtained, that the interfacial shear strength is reduced by approximately a factor 2. This result must be interpreted very carefully, since the properties of the resin after being soaked by water are not entirely known. It is well known that water acts as a plasticiser with most epoxy resins, and tends to decrease their glass transition temperature. Moreover, it has been shown recently that the spectrum of relaxation times, hence the relaxation behaviour of epoxy resins, is profoundly

affected by the water content. This phenomenon can be dealt with by a formalism similar to that describing physical aging, i.e. by introducing a shift factor which depends upon the hygrometric ratio, although its coupling to other phenomena (physical aging, applied stress), is not known. It is nevertheless generally known that water diffuses faster at the interface, and weakens adhesion by hindering all weak interactions (Lipschitz-Van der Waals bonds) between the fibre and matrix, and possibly some covalent bonds too.

Finally, the values for both coated and conditioned testpieces are close, and it is therefore thought that most of the covalent bonds have been destroyed at the interface, and that the resulting strengths obtained are characteristic of the surface roughness of the fibre only, i.e. mechanical interlocking.

As a conclusion, these results using a proper analysis show that the test is suitable for surface quality assessment when the difference in surface quality is large. In the present case, the single fibre fragmentation test is, however, a sophisticated tool for obtaining a result which would be evident when using classical laminated testpieces, for example in a transverse tensile strength test. This is probably not the case when small differences between surface treatments need to be evaluated, as discussed in the following section.

5.2.3 Effect of Surface treatment on Adhesion

The effect of surface treatment on adhesion was examined with testpieces having glass fibre, and three different sizings: conventional (γ -APS sizing, B1), water-based (B2), and optimal (epoxy+polyester sizing, B3). The same hypotheses as in the preceding section were made, and shear strength results obtained with or without accounting for debonding are presented in Figure 15.

The same difference as noted previously is observed upon the introduction of debonds in the analysis. Again, far too high shear strengths would be obtained by neglecting debonding of the interface. In the B2 experiments, debonding was hard to observe and the error involved was large compared to other batches. This explains the higher standard deviation of this experiment.

The B1 experiment gives a very interesting result, comparable to previous experiments involving the same fibre and a pure DGEBA with a comparative chemistry, cured by a diamine (Jeffamine D400). These experiments used the microbond test rather than the single fibre fragmentation test, and the same shear lag model. An interfacial shear strength of $22.1 \pm 0.2 \text{ MPa}$ was found, which is comparable to the $22.8 \pm 1.2 \text{ MPa}$ found here. This unexpected outcome is fully justified by the chemistry of interfacial reactions: supposedly, the resin only (DGEBA) forms bonds with the sizing of the fibre, and the hardener does not have any effect in the interfacial properties resulting from these reactions. This is exactly the result obtained, since the two resins have exactly the same chemistry, the degree of purity of the DGEBA being the only difference. Therefore, the two tests are consistent provided the analyses are. This seems obvious, but it should be emphasised at this point that most studies from the literature have presented different results with the two tests, which were due to the use of inappropriate models, and not to the experimental results.

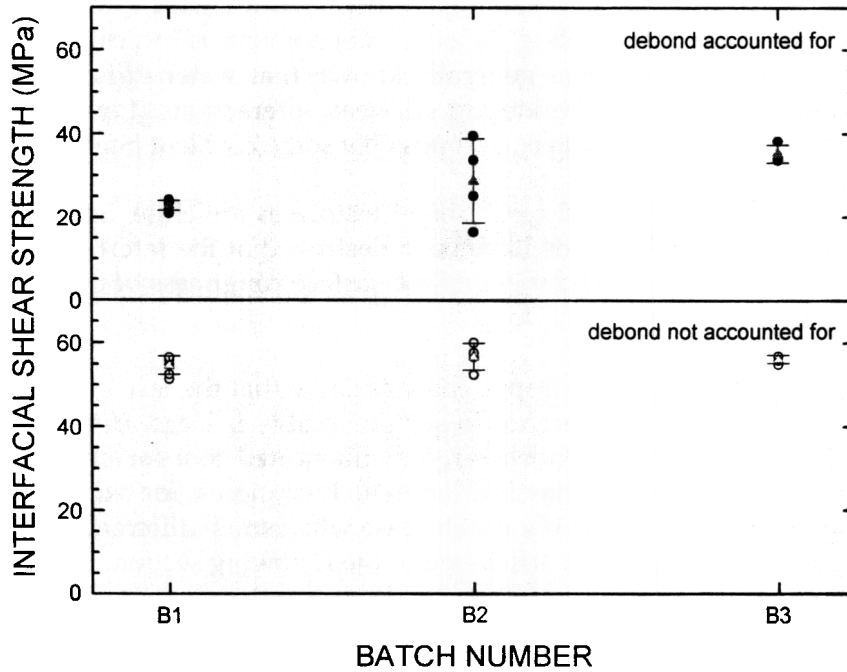


Figure 15 Interfacial shear strength for various surface treatments of glass fibre - DGEBA/mPDA single fibre fragmentation testpiece

The validity of the interfacial shear strength measurement having been checked, it is interesting to compare the results obtained in this study to those of surface energy measurements which were obtained in previous studies [74,75].

Following the theory developed by Nardin and Schultz [76,77], the interfacial shear strength τ_0 should be proportional to the work of adhesion W_a , so that:

$$\tau_0 = \delta^{-1} \sqrt{\frac{E_m}{E_f}} W_a \quad (25)$$

where δ is defined as a characteristic intermolecular distance of adhesion. The work of adhesion is classically obtained from contact angle measurements on the fibre, as:

$$W_a = \gamma_L (1 + \cos\theta)$$

where γ_L is the liquid's surface tension in which the fibre is dipped, and θ is the contact angle between the fibre and liquid.

The results of contact angle measurements, together with the calculated work of adhesion and the interfacial shear strength obtained through the resent model and experiments are given in Table 3, and shown in Figure 16. Although the data seems to follow a linear trend, one must be very careful when drawing such conclusions about these experiments. The major reason is the small variation of the contact angle, which, when taken as a cosine, involves very small variations of the work of adhesion. In most of the studies available in literature, models used to calculate the interfacial strength are inappropriate by different degrees -- by not accounting for internal stresses, or debonding, or using the simplified Kelly-Tyson approach (which was the method devised by Nardin and Schultz) -- but still, they satisfy quite well the relation (25). So do the present results, but it must be kept in mind

that some other determinations of the work of adhesion were carried out in a former study, and yielded different results [74]. In this work, the information concerning the contact angle was used because it is generally considered as the easiest way to obtain information on the work of adhesion.

Notice also that there is a good correlation for the advancing contact angle only, and large deviation from the linear behaviour of the receding contact angle. The errors involved are important, particularly on the water-sized glass fibre testpieces. Moreover, the work of adhesion represents only a part of the adhesive strength, and is measured in a liquid state. It should therefore first be established that the work of adhesion increases linearly when curing proceeds, to conclude that Equation (25) is true [69]. Some measurements have been carried out on neat epoxy resins that tend to prove this fact, but it is still not formally established.

As a conclusion, the intrinsic shear strength was found to be affected to a great extent by the interface chemistry, and the single fibre fragmentation test was efficient in monitoring these changes. On the other hand, it is still not possible to predict the interfacial shear strength from thermodynamic data of the interface.

Table 3 Contact angles versus work of adhesion and interfacial shear strength for Vetrotex glass fibres with 3 different sizings

Sizing	Advancing Contact Angle	Receding Contact Angle	W_a/γ_L (advancing)	W_a/γ_L (receding)	Interfacial Shear Strength	δ (advancing)	δ (receding)
			(-)	(-)	(MPa)	($^{\circ}$ A)	($^{\circ}$ A)
epoxy com- patible size	53.3 ± 0.9	45.8 ± 0.5	1.598±0.007	1.697±0.006	22.8 ± .2	4.6 ± 0.4	2.1 ± 0.5
water sizing	53.0 ± 1.7	45.2 ± .0	.603±0.014	.705±0.012	28.7 ± 10	3.7 ± 0.	2. ± 0.5
water+silane sizing	50.0 ± 2.2	44.4 ± 1.8	1.643±0.024	1.714±0.02	35.2 ± 2.0	3.1 ± 0.1	2.3 ± 0.7

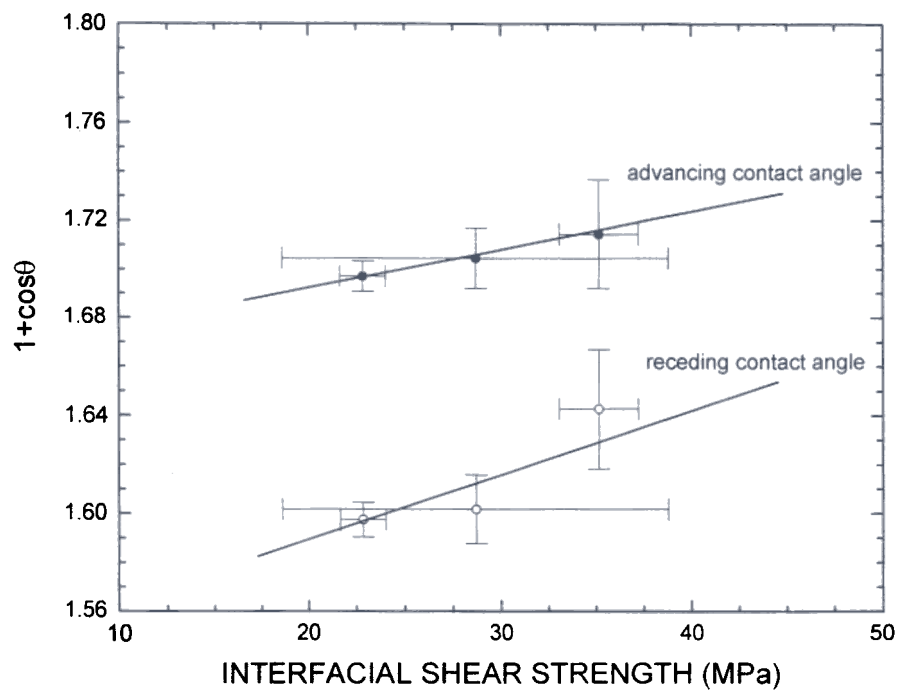


Figure 16 Relation between the work of adhesion and the interfacial shear strength

6 CONCLUSIONS

This work investigated the analysis and experimental protocol of the single-fibre fragmentation test. After a review of the possible analyses of this test, an extensive screening study was carried out to determine the optimal geometry and test conditions of single-fibre fragmentation testpieces. The results of these tests, together with a modelling of the fragmentation process, revealed that the widely used interpretation of this test is inadequate when describing the essential features of interface failure. Instead, local stress or energy analyses should be used. In this work, a simple, closed-form, shear-lag based model was chosen to describe the axial stress in the fibre and the interfacial shear stress. While approximate, this method allowed the assessment of the quality of the interface in carbon fibre and glass fibre epoxy single fibre composites. It was possible to discriminate between different degradations of the fibre surface (tests on carbon fibre testpieces), as well as different surface sizings (glass fibres). The good quality of the results should not, however, disguise the following drawbacks of this test:

- the information obtained on fibre properties is still subject to a large standard deviation, which should limit its use. A method based on statistical description of the variability of data was proposed to overcome this inconvenience in this work;
- the existing stress transfer models are still unable to solve all the equations of equilibrium with all boundary conditions satisfied. As a consequence, the estimated interfacial strength or toughness values are, at best, good approximations. They should be therefore used with care, as comparative rather than quantitative information;
- another consequence of the previous point: because the stress state is not fully known, and because of the uncertainty arising from it, it is meaningless to extrapolate thermodynamic quantities from these values. This step will require much more work to be carried out;
- the results prove that the fragmentation test is a stable method for quality control. However, the same results can be obtained by testing composite testpieces, which are easier to manufacture, and to test;
- this test cannot be performed on all systems of materials, and the strength analysis needs the saturation state to be reached. On the other hand, some energy methods are applicable in the small strain regime, which should enable more systems to be tested;
- and finally, although quality control can be performed using the single-fibre fragmentation test, the rigorous application of the results of this test as a design parameter for composites is still out of reach. The advancement of the research at NPL suggests this could be achieved within 2 years, by the use of energy-based methods.

For these reasons, it seems wise to recommend continuing the testing of representative composite testpieces in industrial environments. The extra-cost of materials is largely compensated by the time savings involved in the preparation, testing of the testpieces, and also the interpretation of the test. The interpretation of test results should be simplified in the near future, and can certainly be fully automated, but the need to manufacture the specimens will always limit to a large extent the use of this test.

7 FUTURE WORK

Several interesting aspects were highlighted by the present study of fibre fragmentation, which have lead to possible modifications of the test analysis. From what has been said, it is of primary importance, in future studies, to:

- carry on the analysis of the test by the energy release rate method. While this should extend the range of possible systems to be tested, some modifications of the test method seems unavoidable. The positive point is that the existing experimental set-up do not need to be modified and can be used directly. The methodology needs to be modified by carrying out a local debonding analysis;
- modify the test geometry. Both the fragmentation test and the microbond test have limitations. It is proposed to mould specimens whose size is between those testpiece geometries considered here, i.e. specimens smaller than the millimetre. Additionally, they should be moulded in-situ, i.e. inside the mechanical tester, to avoid handling and preparation difficulties. To do so, MEMS technology could be used;
- perform a complete statistical study of the test, if the current geometry continues to be used (c.f. section 3).

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