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Stress transfer by shear in carbon fibre model composites

Part 1 *Results of single-fibre fragmentation tests with thermosetting resins*

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The fragmentation of an embedded single filament has been carried out for a number of carbon fibres and resins in order to determine the ability of the interface to transfer shear stresses at the fibre–matrix interface. Modifications have been introduced in the shape of a two-step embedding process so as to extend the standard method to brittle resins. The fibre surface treatment as well as the resin are conclusively shown to have a definite influence on the stress transfer.

1. Introduction

After a period of time since Fraser *et al.* [1] first reported data on glass fibres, the recent literature offers a great deal of fragmentation testing on various fibres. A decisive impetus was given by Drzal and co-workers [2, 3] who demonstrated that the method is likely to clear up some effects of the carbon fibre surface treatment or sizing. Ohsawa and co-workers [4, 5] similarly took the first steps with glass fibres in a series of four papers whose first three seem to ignore Fraser *et al*'s early work.

In spite of the unrealistic stress state, since the surrounding material is made of homogeneous matrix instead of the average reinforced matrix, the embedded filament tensile test has been commonly considered as a good means to study the stress transfer between fibre and matrix, particularly when there is a need for comparing fibres which were given various treatments or sizings.

Some of the earliest theoretical models attempt to determine how stress is transferred to a fibre when the surrounding matrix is loaded in tension: the Cox analysis is usually taken as the basic description of that transfer whenever organic thermosetting resins– which are at first sight linear-elastic up to the breaking point– are considered. Discussion of the theoretical aspects is put aside in the present paper and will be introduced in Part 2 [6].

As a reminder of what the fragmentation test consists of, it is useful to look at Fig. 1. When a resin sample, with a single continuous fibre embedded in it, is loaded in tension, the same deformation is experienced by the fibre and the matrix assuming that no frozen-in residual thermal strains are present as pointed out by Galiotis and co-workers [7, 8]. Now, if $\varepsilon_{fR} < \varepsilon_{mR}$, the fibre breaks when the specimen deformation reaches ε_{fr}. Further loading of the resultant two pieces is accomplished by shear through the interface, and the progressive rupture of the fibre may follow until the fragments are too short to be stressed to rupture ("saturation"). Measuring the average fragment length gives the critical length l_c of fibre and the critical aspect ratio l_c/d. Values of l_c/d are a function of the fibre tensile strength and the interfacial shear strength for the fibre–interface–resin system under consideration according to the so-called Kelly equation

$$l_c/d = \sigma_{fR(l_c)}/2\tau_m \quad (1)$$

With the fragmentation test, the above expression is used to get τ_m which can be taken as a valuable indication of the ability of the interface to transfer stresses. When l_c/d ratios from various sources are to be compared, one has to be cautious that the average fragment length very often stands for l_c in Equation 1.

Behind the apparent simplicity, the right-hand side terms of the above expression conceal a great deal of complexity since $\sigma_{fR(l_c)}$ refers to the ultimate strength of very short lengths of fibre and τ_m covers in fact a combination of different modes of stress transfer. Moreover, as will be discussed in Part 2 [6], there is an obvious difference between the strength of carefully selected fibres of very short gauge length and the corresponding characteristics of a fragment of the same length in which the fragmentation process statistically results at the end of the test.

In the same situation, Bascom and co-workers [9, 10] preferred to discuss fragmentation data in terms of l_c/d without converting into τ_m. However, comparing only the critical aspect ratios for a range of fibre–matrix systems is hazardous as interference of the fibre properties with the interface characteristics is just ignored.

This paper first presents the conditions which are required to get unquestionably sound τ_m values; then follows the application to some experimental and commercial carbon fibre–epoxy systems, with emphasis on pendent problems. In part 2 [6] a statistical model

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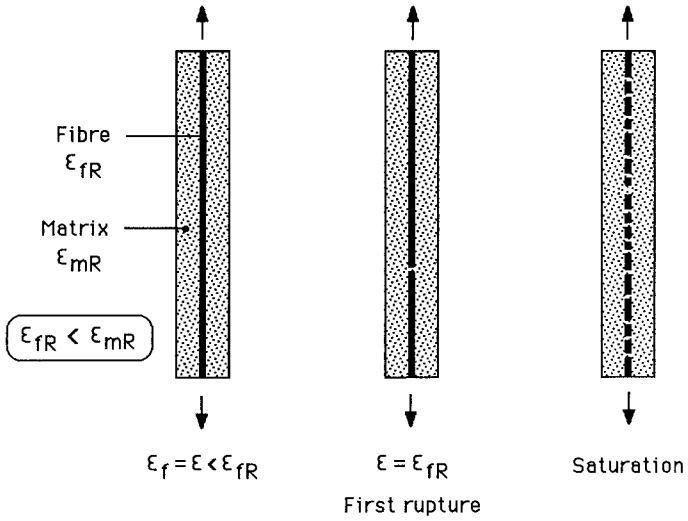

Figure 1 Schematic diagram of the fragmentation process.

of fragmentation will be presented with a thorough analysis of the contributions to τ_m.

2. Method requirements

To use Equation 1 properly, three conditions are to be wholly satisfied:

(i) A basic requirement to perform the test up to the critical length is that the matrix is able to sustain enough deformation before breaking. On the authority of past practice for the fragmentation test, a ratio of about 3 : 1 of the matrix rupture strain to that of the fibre appears to be necessary to achieve the fragmentation test without trouble. With carbon fibres in view, this ratio was satisfied with the first generation of epoxy resins of DGEBA type, but is no longer satisfied with the high-performance systems currently used in structural applications where rupture strains as low as 1.8 (Ciba 914C), 2.0 (Narmco 5245) and 2.4% (Narmco 5208) are measured on neat resin samples [11]. This is a very critical point in so far as high-elongation carbon fibres are now supplied with a strain-to-rupture in excess of 2% at practical lengths. Clearly, fragmentation data have up to now been collected with matrix systems which are no longer used with carbon fibres.

(ii) When performing the test, one has to make sure that attainment of saturation is effective and thus the fibre cannot break any longer. Thus the common practice of straining the resin to a prescribed value ought to be discontinued. Rich and Drzal [12] recently reported the variations of τ_m computed throughout the mechanical test from zero load up to the final load (saturation). It is apparent from these authors' data [12] that, for a given matrix, the rate of shear stress increase provides no indication of the final value of τ_m, which specifically depends on the interface quality.

(iii) Finally, the fibre tensile strength has to be known at the critical length, which ordinarily calls for a careful handling of the Weibull statistics in order to extrapolate to very small lengths the strength data obtained at experimentally feasible lengths.

Other considerations have to be kept in mind to give a complete description of the fragmentation process. Discarding them does not affect the significance of the τ_m data comparison between fibre–resin systems, but they cannot be ignored when tentative scenarios of the process will be put forward (see Part 2 [6]):

(i) Initial stress state: the residual thermal stresses are to be accounted for as for any composite material. Before mechanical loading of the specimen the fibre is axially under compression, which apparently defers the first fracture and the subsequent ones until a high specimen strain, reducing again the available matrix strain.

(ii) Complexity of the rupture mode: as outlined before, τ_m is nothing more than a rough parameter resulting from the combination of the various mechanisms operating at the interface, like elastic shear loading, debonding, friction and plastic yield. All of them must be identified to put the proper values into a model and thus get valuable information.

3. Experimental procedure

3.1. Materials

Table I gives a description of the carbon fibres we used and the mechanical properties we obtained by tensile-testing single fibres with a gauge length of 12 mm.

TABLE I Carbon fibre properties (12 mm gauge length)

Fibre	Origin	Surface conditions*	Mechanical properties		
			E (GPa)	σ_{fR} (GPa)	ε_{fR} (%)
Grafil HT (staple)	Courtaulds	UT	248	2.77	1.10
Torayka T300 6 K	Soficar	UT, ST, STS (50B)	231	3.83	1.65
Torayka T800 12 K	Toray	ST, STS (40B)	294†	5.10†	1.70†
AS4 6K	Hercules	ST	220	4.45	2.01

*UT: untreated, ST: surface treated, STS: surface treated and sized.
†Manufacturer's data (impregnated strand test).

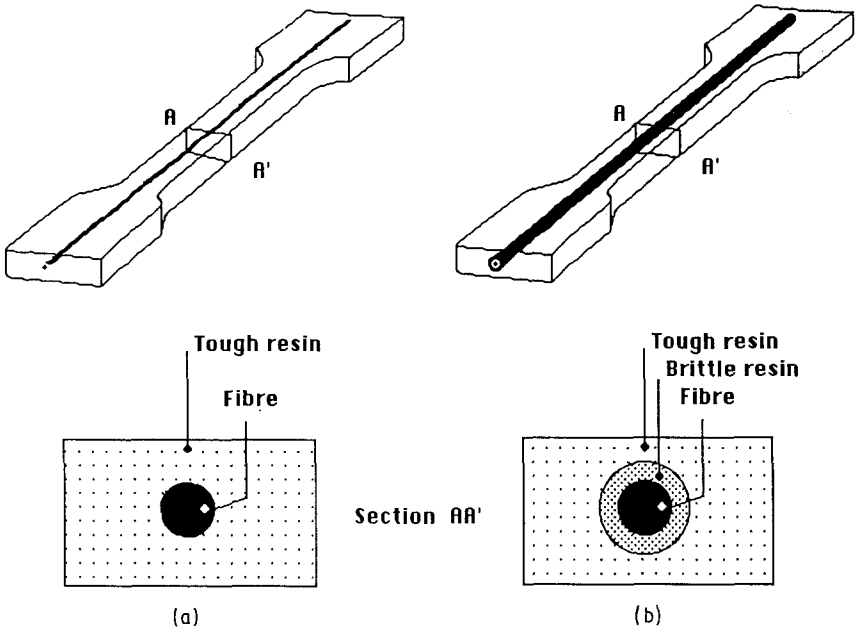

Figure 2 Dog-bone specimens for fibre fragmentation in (a) tough resin ("plain resin" geometry) and (b) brittle resin ("coaxial" geometry).

Table II shows the resin systems: as mentioned above, the first system is quite convenient for moulding single-filament specimens and matches very well with the strain-to-failure requirement for carbon fibre fragmentation. The three others are by no means tough or extensible enough to allow the test to be performed. A "coaxial" specimen was especially designed to get round the difficulty [13].

3.2. Specimens

Dog-bone specimens (Fig. 2a) were obtained by casting the hot liquid Araldite resin into silicone moulds with single carbon filaments mounted along the cavity centre lines. A two-step curing cycle was adopted to get bubble-free specimens and prevent premature fibre fractures due to the interaction at high temperature between the mould and the freshly hardened resin; after a short period under a mild vacuum to de-aerate the resin, hard-setting proceeds at 60° C; then the specimens are taken away from the mould and transferred to an oven for completion of the curing cycle (2 h at 140° C).

When necessary (for acoustic emission counting-up, see Section 3.3 below), the parts of the fibre between the end of the flat section and the fillet on each side were removed by precision machining and the resulting grooves filled up with resin. A length of 30 mm of fibre in the calibrated flat part of the specimens is left for fragmentation.

With brittle resins, a modification was introduced: the fibre is first coated with a thin sheath of the resin by using a miniature coating device where a drop of hot resin moves about along the fixed fibre. Curing follows according to the specifications in Table II. The coated fibre is then embedded into the deformable and crack-resistant Araldite resin, giving the so-called coaxial specimen (Fig. 2b).

When the coaxial specimen is loaded in tension, each fibre rupture induces a fracture of the brittle resin sheath but the penny-shaped crack is now stopped by the tougher supporting resin, thus preventing the fracture of the whole specimen. The mechanical implications of the coaxial specimen were presented elsewhere [13] and will be referred to again later.

3.3. Tensile testing

Specimens were tested in a loading system (Fig. 3) consisting of a home-built motorized straining stage with the moving crosshead driven at a constant rate of 0.5 mm min^{-1} and equipped with an LVDT extensometer. The straining stage is supported by a X–Y platform which makes centering on the observed area easier. The equipment is set under an optical microscope operating in transmitted light between polarizing filters. The fragmentation can be continuously monitored by a video camera and recorded on video cassettes for later replay and analysis.

A PZT transducer coupled to the specimen collects the acoustic emission (AE) associated with the fibre fractures and AE, analogically recorded as counts per event, is simultaneously plotted as a function of time or displacement. This gives readily the cumulative number of fibre fractures against time, up to the end of the tensile test, as presented in Fig. 4. With AE, the breaking process can easily be supervised and the test stopped before specimen failure as soon as a long

TABLE II Matrix properties

Resin system	Max. temp. cure	Tensile properties		
		E (GPa)	σ_{mR} (MPa)	ε_{mR} (%)
Araldite LY556/HT972	2 h/140° C	2.70	94.1	5.72
Ciba Fibredux 914C	2 h/190° C	4.02	62.7	1.77
Narmco 5208	2 h/180° C	3.85	79.1	2.41
Narmco 5245	2 h/180° C	3.95	75.1	2.03

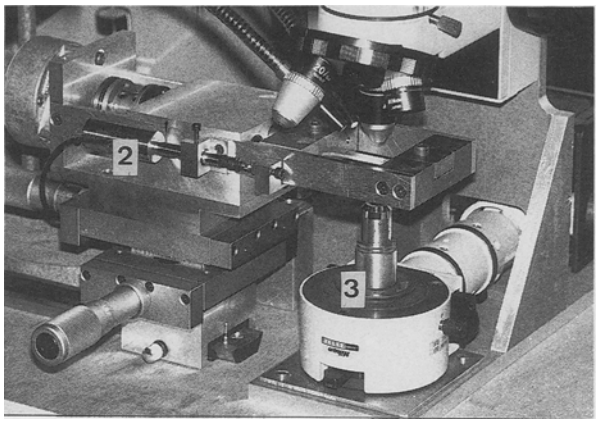

Figure 3 Detail of the fragmentation testing equipment: (1) crosshead, (2) LVDT transducer, (3) source of transmitted light.

period of "quiet" loading has been spent, which is an indication that saturation has been achieved.

The final number and dimensions of broken fibre pieces are determined either from the above AE signals, with $\bar{l} = L/(N + 1)$, where $\bar{l}$ is the mean fragment length, L the initial fibre length (30 mm) and N the number of acoustic events or, directly, by visual inspection of the held-on sample. Both values generally agree with each other within a few per cent.

Fig. 5 shows typical histograms of the length distribution of three specimens of the same batch of fibres and resin: the first one displays classes arranged upwards from the minimum, while in the second, classes are arranged on either side around the mean value. Considering the latter there is no fundamental reason to analyse the l/d data in terms of a Weibull distribution as Drzal and co-workers [2] used to do, and the arithmetical mean was taken thoughout the present work.

Finally, the critical length is derived from the mean length $\bar{l}$ using the approximation of Ohsawa *et al.* [4] that $l_c = \frac{4}{3}\bar{l}$. A cumulative number of 120 to 300 fragments, depending on the fragment production, was considered to form a sufficient set of data for each fibre–matrix system.

The fibre diameter was taken as the average of numerous separate SEM determinations for each type of fibre.

TABLE III Fragmentation data with the plain LY556 DGEBA matrix (simple specimens)

Fibre	Fibre properties		l_c (mm)	τ_m (MPa)
	m	$\sigma_{fR(l_c)}$ (GPa)		
HT UT	3.6	4.69	1.52	12.2
T300 UT	11.5	4.13	0.58	24.9
T300 ST	11.5	4.21	0.46	32.1
T300 STS	11.5	4.30	0.37	40.6
T800 ST	7.5	6.79	1.36	13.2
T800 STS	7.5	6.84	1.29	14.0
AS4 ST	4.1	8.03	0.58	50.2

3.4. Fibre strength at the critical length

The fibre strength at $l = l_c$ cannot be experimentally measured and calls for some extrapolation from tensile data at convenient lengths, using a Weibull statistics that is often beset with a lot of suspicion. The determination of Weibull parameters from the testing of fibres at only one gauge length may entail a great deal of error in the subsequent computation of the short fragment strengths, because minor changes in the shape parameter produce large variations in the estimated strength.

Taking advantage of various recent contributions [14–16], the following method was adopted in practice: the fibres are tested in tension at several (at least 4) gauge lengths from 2 up to 20 mm, and σ_{fR} is plotted as a function of the gauge length on a logarithmic scale. A simple linear regression gives σ_{fR} at $l = l_c$.

A typical plot for T800 fibres is shown in Fig. 6, each point representing the mean value of about 30 samples. The local Weibull modulus m for each of the five sets of data has been also indicated. The value $m = 7.5$ from the σ_{fR} against l straight line is, on average, consistent with the five local values of m for that fibre. Even if the method involves a tedious handling of the same number of single fibres, the risks of large errors attached to the extrapolation from only one set of data at fixed length are minimized.

4. Results

In Table III have been tabulated the results of the fragmentation tests for the plain DGEBA resin (simple specimens) with various fibres. The results for

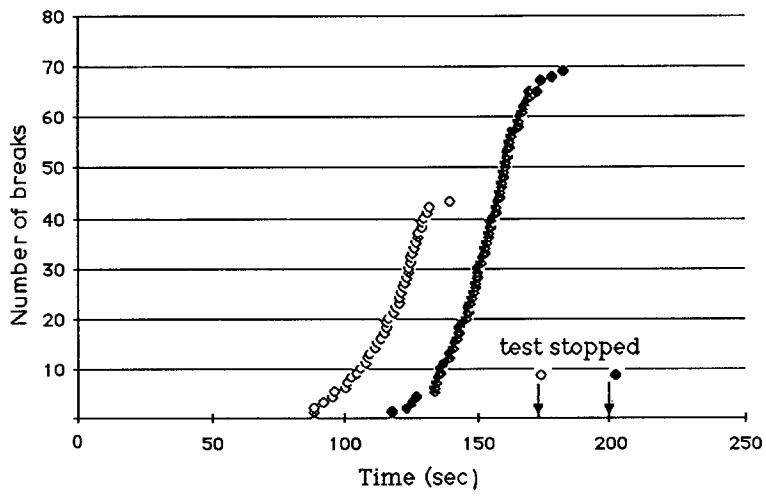

Figure 4 Cumulative number of fibre breaks against time as monitored by acoustic emission for two plain resin specimens: (◆) T300 ST, (◇) T300 UT.

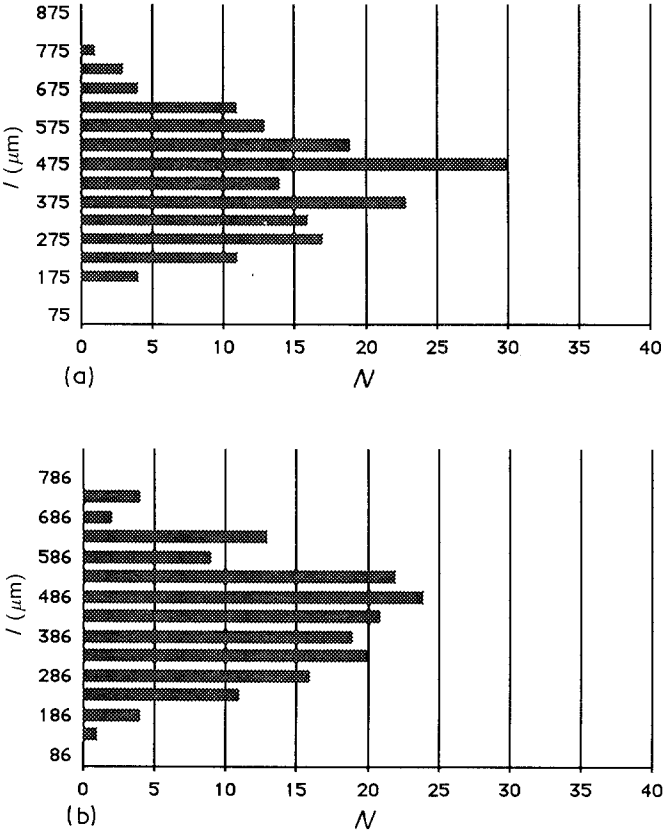

Figure 5 Typical fragment length distribution (N = 166; combination of 3 samples) with ranking (a) from the minimum and (b) from the arithmetical mean. T300 6K UT/LY556.

the other resins (coaxial specimens) are given in Table V below and Fig. 7.

4.1. DGEBA resin

As regards the influence of the fibre surface treatment, HT, T300 and AS4 fibres produce a regular range of τ_m values in the expected direction: the more "active" are the fibres, the better is the stress transfer. This makes the T800 data all the more unexpected; yet monitoring by the video camera of the fragmentation process gives a visual confirmation that, for the T800–resin system, a considerable length along the new fragments is affected by debonding, resulting in a high l_c and correspondingly low values of τ_m.

The present status of the single-fibre methodology makes it difficult to compare data from various sources, because of the scarcity of the results and the diversity of testing conditions. Fortunately, for the AS4 fibres, there are four sets of valuable independent data in the recent literature, with resin systems (Epon 828 + mPDA or DDM) very similar to the Araldite used in the present work. The results are listed in Table IV: except for the first two values (both originating from Drzal and co-workers, which implies a reconsideration of their earliest figures, possibly due to a variation in fibre properties), there is a very good agreement around $\bar{l}/d = 60$, $\bar{l}$ referring here to the average fragment length without the correction of 4/3 we introduced previously for l_c.

Things are also good when values of τ_m are compared. As the tensile strength of the AS4 at $\bar{l}$ is now required the data of Drzal and co-workers, relying on a relatively low value for $\sigma_{fR(\bar{l})}$, finally fall in with other results. As pointed out before, Bascom and co-workers [9, 10] did not measure $\sigma_{fR(\bar{l})}$ and thus gave no τ_m in their papers. Finally, there is apparently some mistake in the paper of DiBenedetto *et al.* [16], as τ_m (42.2 MPa) does not appear to be evaluated by using the $\sigma_{fR(\bar{l})}$ that should be derived from the Weibull parameters of the distribution reported (m = 4.75 should give an average fibre strength of 9500 MPa and 79.2 MPa for τ_m instead of the 42.2 reported).

As pointed out before, the determination of $\sigma_{fR(l_c)}$ is of primary importance to get a representative value of τ_m. With the procedure of tensile-testing fibres at various lengths, a Weibull modulus of 4.1 has been found here. That can be favourably compared with 4.75 [16], 3.1 [2] and are arduously discussed m = 5

TABLE IV Summary of data from the literature for AS4 fibres

Resin system	$\bar{l}/d$	$\sigma_{fR(\bar{l})}$ (GPa)	$\tau_{m(\bar{l})}$ (MPa)	Ref.
Drzal and co-workers	(42)	5.92	69.3	[3]
	(42)	5.32	62.3	[12]
	57	(9.80)	86	[17]
Bascom and co-workers	55–65	–	–	[9, 10]
DiBenedetto and co-workers	60	(9.51)	(79.2)	see text
			42.2	[16]
Favre and Jacques	60	8.68	71.5	This paper

( ) Brackets stand for recalculated values from author's data.

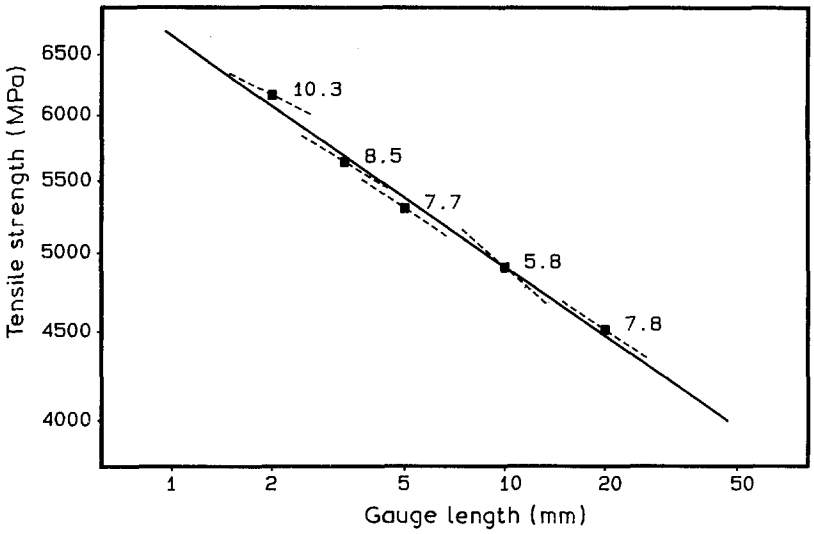

Figure 6 Linear regression of the rupture strength of T800 single carbon fibres against gauge length. Also indicated is the local Weibull modulus computed for each set of fibres (about 30 tests for each length) of constant length. Weibull modulus from line slope = 7.5.

from a paper by Manders and Chou [15]. Yet the ability of the Weibull statistics to correctly describe the fibre strength distribution at very short lengths is still a matter of dispute.

The accordance with available literature data on AS4 fibres gives confidence in the results of Table III, particularly for T800 fibres with the smallest τ_m (except for the "old" untreated HT fibre).

4.2. Brittle resins

While Table III referred to the fragmentation tests with a plain DGEBA-type resin matrix, Table V gives data for fibres first coated with TGDDM or cyanate resins and then encapsulated in the standard DGEBA matrix.

In view of the higher Young's modulus of the TGDDM resins, a decrease of l_c/d (or an increase of τ_m) is involved in the Cox equations for the elastic stress transfer at the fibre–matrix interface. In accordance with the approximations made by Galiotis *et al.* [7] and Nardin *et al.* [18], l_c/d is roughly proportional to $(E_f/E_m)^{1/2}$ when fibre–matrix bonding is good. Thus the correction

$$l_c = l_c \times (E_{LY556}/E_{matrix})^{1/2}$$

was introduced by Jacques [19] to describe more accurately the existence of a bi-modulus material around the fibre in the coaxial specimens, the fibre pieces being reloaded by the "high-modulus" brittle matrix that is in turn loaded by the supporting "low-modulus" LY556 resin. Corrected data are shown in Table V as well as data based upon Kelly's general expression. The latter are also shown in Fig. 7 together with the LY556 data. A general improvement in τ_m values is observed for all systems whatever the initial surface conditions. Again, the smallest τ_m values are obtained for T800 fibres.

Though the actual stress conditions in the "coaxial" geometry broadly elude analysis and the TGDDM sheath around the fibres looks very much like a sizing rather than a plain matrix, the enhancement of τ_m calls for the following comments:

(i) Clearly enough, neither the presence of some droplets nor the thickness of the TGDDM resin coating, which may vary within a broad range owing to the coating process we used, do affect the general trend towards higher τ_m values.

(ii) On the rough basis of the ratio of moduli, the data, whether corrected or not, are consistent with the theory that just predicts a better stress transfer with TGDDM resins. As explained elsewhere [19], this better transfer is fully achieved owing to the excellent adhesion of the resins to the fibres (the pull-out testing of T300 fibres gives 115 to 130 MPa for 5208 or 914 resins compared to 60 MPa for LY556).

(iii) In fact, comparable situations have been reported before when the effect of various coatings was evaluated. In some cases, Drzal *et al.* [20] and Ying [21] took notice of a favourable effect on τ_m. The explanation put forward by Drzal *et al.*, who simply coated the fibres with pure DGEBA resin, is that an interphase of relatively high modulus is formed,

TABLE V Fragmentation data from brittle resin systems (coaxial specimens)

Fibre	Matrix	l_c (mm)	τ_m (MPa)	Corrected values*	
				l_c (mm)	τ_m (MPa)
T300 UT	5208	0.429	38.9	0.359	47.6
T300 ST	5208	0.358	47.7	0.300	58.3
T300 STS	5208	0.377	45.0	0.316	55.0
T300 UT	914	0.361	47.3	0.302	57.9
T300 ST	914	0.380	49.0	0.293	59.9
T300 STS	914	0.325	53.3	0.272	65.2
T800 ST	5245	0.675	29.3	0.558	36.3
T800 STS	5245	0.567	35.7	0.469	44.3

*Corrected $l_c = l_c \times (E_{LY556}/E_{matrix})^{1/2}$.

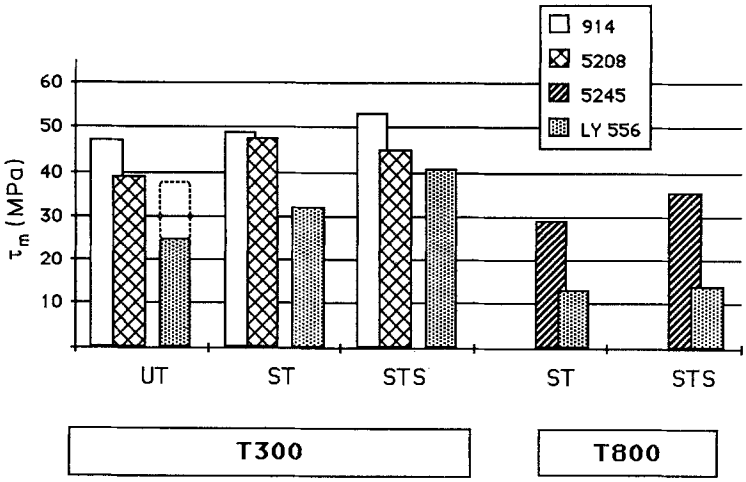

Figure 7 Uncorrected τ_m data for brittle resins (coaxial specimens) with T300 or T800 fibres compared with the plain LY556. Fibre references as in Table I. The dotted box stands for the results with the LY556–LY556 coaxial specimens.

causing the stress transfer to be improved. The present results are not strictly amenable to the same mechanisms, as the fibre coating is now completely cured before encapsulation in the low-modulus DGEBA resin.

5. Discussion and conclusions

Complementary experiments gave results that might shed some light on the difficulties in assessing what τ_m does really consist of: series of coaxial samples were prepared several times with T300 UT fibres and LY556 both as the coating and the supporting resin, in order to verify that there was no drawback in using a non-conventional sample design. The curing conditions for the LY556-coated fibres varied from no cure to standard cure. The samples prepared with mild or complete curing of the coated fibre exhibited small l_c/d ratios leading to a 44% improvement of τ_m (27.6 to 38.8 MPa, see Fig. 7). This was a baffling result, since a decrease of τ_m was expected for the reason that the stress transfer at the fibre–matrix interface takes place over a longer distance in the coaxial specimens due to the additional resin–resin interface [13].

Among the mechanisms that may be invoked to explain the somewhat confusing τ_m increase, one comes out from the observation of the "penny-shaped cracks" originating from the fibre fracture: the cracks are supposed not to propagate to the same length, depending on the material and the interfaces they have to go through. With the tough LY556 plain resin, the crack extension is limited only by the toughness of the material; but in coaxial specimens, the crack first propagates in a very brittle material, and then is deflected and slowed down at the resin–resin interface and finally stops in the LY556 supporting resin.

The influence of the cracks is likely to be negligible for high l/d, that is to say for the most part of the rupture process, when the fibre pieces are very long, and also near to saturation for systems prone to a load transfer extending over a long distance. However, for cracks close to each other (systems with a small l/d ratio and approaching the saturation state), an underestimated τ_m is calculated.

Noticeable effects are also expected to originate from the two-step processing conditions, the particular state of thermal stresses in duplex-matrix specimens and the plastic yield of the confined sheath of the matrix. The conclusions to be drawn from the present experimental work are of dual character:

(i) As regards l_c/d or τ_m, the comparison of the AS4–epoxy values from independent sources gives some confidence in the reliability of the fragmentation test as a good means of assessing the interface properties. The positive effect of fibre surface treatments is shown clearly again, as well as the influence of the matrix properties. The unpredictable low τ_m of the T800 fibres is also deserving of consideration, and could be related to the outstandingly good damage tolerance of the T800–epoxy laminates.

(ii) On the other hand, there is no doubt that the observed l_c/d and the subsequently calculated τ_m are the result of a combination of several processes which can be readily ascertained when fragmentation is dynamically monitored but cannot be quantified at the moment. The special conditions for getting τ_m by testing coaxial specimens also introduce uncontrolled extra parameters.

A comprehensive view of the fragmentation mechanisms remains to be established. Correspondingly, there are still a lot of unanswered questions about the mechanical functions of coatings or interphases in relation to the mechancial properties of the composite materials.

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