On the estimation of the tensile strength of carbon fibres at short lengths

EL. M. ASLOUN, J. B. DONNET, G. GUILPAIN, M. NARDIN, J. SCHULTZ* *Centre de Recherches sur la Physico-Chimie des Surfaces So/ides CNRS, 24,* Avenue du Président Kennedy 68200 Mulhouse, France

Generally, to determine the fibre-matrix interfacial properties in fibre reinforced plastics, it is necessary to know the tensile strength of the fibre at very short lengths, for which direct measurements are not possible. Accordingly, in this study, the determination of the tensile strength of high strength carbon fibres and their gauge length dependence are analysed by means of the Weibull model. The influence of the estimator chosen and of the sample size on the calculated value of the tensile strength of the fibre are first determined. Secondly, the accuracy of the three- and the two-parameter Weibull distributions is examined. Finally, it is shown that the most appropriate extrapolation at short length is performed by means of a linear logarithmic dependence on gauge length of the tensile strength. This method seems to be valid for untreated as well as for surface-treated high strength carbon fibres.

1. Introduction

The structure and the properties of the fibre-matrix interface play a major role in the mechanical and physical properties of composite materials. In particular, the fibre-matrix interfacial shear strength is one of the most important parameters in determining the strength and toughness of a unidirectional composite, since the load working on the composite is transmitted to the fibre through the interface. In order to relate the strength of unidirectional carbon fibre reinforced plastic composites to the strength of their constituents, it is, therefore, necessary to know as precisely as possible the value of this fibre-matrix interfacial shear strength. However, according to the theories generally used in this domain [1-3], this shear strength is dependent on the tensile strength of the fibre at lengths corresponding to the fibre-resin transfer length, usually called the critical fibre length l_c [2]. For example, the value of l_c is generally equal to about 0.5 mm or less for carbon fibre reinforced epoxy resin. Consequently, it is impossible to carry out experimental measurements of individual fibre strength at these short lengths and most analyses extrapolate fibre mean strength and strength distribution data obtained at longer lengths, i.e. 3 to 100 mm. The most widely used expression for this extrapolation is the cumulative distribution function proposed by Weibull [4, 5]. This statistic is based on the "weakest link hypothesis" which means that the most important flaw in the fibre will control its strength. Nevertheless, the extrapolation of strength at critical length l_c has to be examined very carefully and accordingly, the object of the present paper is to determine the influence on the calculated values of fibre tensile strength (i) of the different Weibull distributions (two or three parameters), (ii) of the different estimators defining the cumulative failure probability and (iii) of the dependence of fibre strength on length. The fibres considered in this study are high strength carbon fibres, denoted HT carbon fibres according to Donnet *et al.* [6], based on polyacrylonitrile. As an example, the value of the fibre tensile strength at a given critical length, experimentally determined in a previous study [7], is estimated in each case. Comparisons are, therefore, made between the different results in order to choose which type of extrapolation is the most appropriate one.

2. **Theory**

The three-parameter Weibull distribution [4, 5] is given by

$$
P = 1 - \exp\left[-l\left(\frac{\sigma - \sigma_{\rm u}}{\sigma_0}\right)^m\right] \qquad (1)
$$

where P is the cumulative probability of failure of a fibre of length *l* at applied stress σ , *m* is a shape parameter known as the Weibull modulus, σ_0 a scaling parameter and $\sigma_{\rm u}$ a threshold stress below which the failure probability is zero. To establish this equation, a single flaw population (volume or surface flaws) and a time independent strength are assumed. It is also assumed that compressive stresses do not contribute to fracture.

The following four estimators are generally used to calculate the probability of failure P_i for the i^{th} strength

$$
P_i = \frac{i - 0.5}{N} \tag{2}
$$

$$
P_i = \frac{i}{N+1} \tag{3}
$$

and
$$
P_i = \frac{i - 0.3}{N + 0.4}
$$
 (4)

$$
P_i = \frac{i - 3/8}{N + 0.25} \tag{5}
$$

*Author to whom all correspondence should be addressed.

where N , the sample size, is the total number of specimens tested. As demonstrated elsewhere [8], for sample sizes less than about 50, the estimator defined by Equation 2 leads to less biased m values than the other ones and should therefore be preferred [9, 10].

It is possible to determine the parameters σ_0 , σ_u and m by rewriting Equation 1 as follows

$$
\ln (1 - P) = -l \left(\frac{\sigma - \sigma_{\rm u}}{\sigma_0} \right)^m \tag{6}
$$

where $(1 - P)$ is now the probability of survival at stress σ . Equation 6 can also be written

$$
\ln\left[\ln\left(\frac{1}{1-P}\right)\right] = m \ln(\sigma - \sigma_u)
$$

+
$$
(\ln l - m \ln \sigma_0)
$$
 (7)

Therefore, experimental data plotted in the form of ln {ln [1/(1 - P)]} against ln ($\sigma - \sigma_u$) give a straight line if the Weibull treatment is appropriate. The parameters m, the slope of this straight line, σ_0 and σ_u can be determined by a simple least squares method. It must be noted that, in this case, σ_u is an adjustable parameter and, therefore, partly defines the value of the other parameters. Sometimes, such a plot gives two or more different straight lines. This means that the fibres exhibit multiple modes of failure and it is therefore necessary to use a more complex statistics, i.e. a bi- or multi-modal Weibull distribution.

Finally, the mean strength σ_w is given by

$$
\sigma_{\rm w} = \sigma_{\rm u} + \sigma_0 \, l^{-1/m} \Gamma \left(1 + \frac{1}{m} \right) \tag{8}
$$

where Γ is the gamma function.

It follows from Equation 8 that the mean failure strength σ_1 , and σ_2 of two specimens of the same material with respective length l_1 and l_2 are related by the equation

$$
\sigma_2 = \sigma_{\mathsf{u}} + (\sigma_1 - \sigma_{\mathsf{u}}) \left(\frac{l_1}{l_2} \right)^{1/m} \tag{9}
$$

This is a useful result, since the mean failure strength for a small length l_2 can be estimated from the observed mean failure strength of a sample of large length l_1 . Nevertheless, it is implicitly assumed that m , σ_0 and σ_u are not dependent on the gauge length.

The three-parameter function (Equation 1) can be used when it is assumed that the fibre has a minimum strength. However in general, it is recommended to take $\sigma_{\rm u} = 0$ for brittle materials since as observed elsewhere [8] the Weibull distribution with $\sigma_u = 0$ leads to the least-biased results.

Consequently, for the two-parameter (*m* and σ_0) Weibull distribution, Equations 7, 8 and 9 are respectively modified as follows

$$
\ln\left[\ln\left(\frac{1}{1-P}\right)\right] = -m\ln\sigma + (\ln l - m\ln\sigma_0)
$$
\n(10)

$$
\sigma_{\mathbf{w}} = \sigma_0 l^{-1/m} \Gamma\left(1 + \frac{1}{m}\right) \tag{11}
$$

$$
\sigma_2 = \sigma_1 \left(\frac{l_1}{l_2} \right)^{1/m} \tag{12}
$$

The values of *m* and σ_0 are now determined by a least squares method without adjustable parameter.

For two- or three-parameter unimodal distributions, the Weibull modulus m is related to the coefficient of variation CV by the expression

$$
CV = \left(\frac{\Gamma(1+2/m)}{\Gamma^2(+1/m)} - 1\right)^{1/2} \tag{13}
$$

At large values of $m (m > 8)$, CV becomes equal to about *1.2/m.* The coefficient CV is concerned with the variation of the distribution since it is also equal to the ratio of the strength standard deviation to the mean strength.

As seen above, knowing the Weibull parameters, it is easy to extrapolate (Equations 9 and 12) at short lengths the value of the mean fibre strength, if this strength was previously determined at any large gauge length. However, it is possible to carry out this extrapolation in another way, by taking the logarithm of σ_w in Equation 11 as follows

$$
\ln \sigma_{\rm w} = -\frac{1}{m} \ln l + \ln \left[\sigma_0 \Gamma \left(1 + \frac{1}{m} \right) \right] \tag{14}
$$

It is seen that a graph of $\ln (\sigma_w)$ plotted against $\ln (l)$ should be linear with a slope $(-1/m)$. Thus, the tensile strength of a fibre at a given length could be easily estimated in this way by testing single fibres of a range of gauge lengths.

Consequently, three methods can allow us to determine the mean tensile strength of a fibre at any length: for the first two (three- or two-parameter Weibull distributions) it is necessary to know the Weibull parameters and the mean fibre strength at a given length; for the third (linear extrapolation using Equation 14) it is only necessary to know the mean fibre strength at different gauge lengths. The aim of this paper is to determine which method is the most appropriate one in the case of HT carbon fibres.

3. Experimental details

Two types (T1 and T2) of high strength PAN-based carbon fibres were used in this study. These fibres were untreated and unsized. In a separate set of experiments, oxidized T1 fibres were also employed. They has received an electrolytic surface treatment in order to increase the fibre-matrix adhesion in composites. The mean diameter of all these fibres was almost constant and equal to 7 \times 10⁻⁶ m. Their mechanical properties were determined on monofilaments, carefully extracted from a $6K$ bundle, at different gauge lengths $l_{\rm g}$ varying from about 3 to 100 mm. Each monofilament was glued with. epoxy resin on a cardboard frame cut at a span length equal to the given gauge length and maintained in its initial shape by means of two clips. Then, the frame, holding the carbon fibre, was carefully put into the clamps of an Instron 1195 H tensile testing machine. Prior to testing, the true gauge length was precisely measured in each case with a travelling microscope. For each set of experiments, the scatter on the gauge length did not exceed about 0.3mm. Tensile strengths were determined at a constant cross-head speed of 0.5 mm min⁻¹. All the samples for which failure occurred nearby the clamps were rejected. About 20 monofilaments were tested for each gauge length, excepted for $l_g =$ 27.55 mm where $N = 85$. Whatever the experimental conditions, the modulus of elasticity and the strain at failure of these HT carbon fibres were found equal to 240 \pm 15 GPa and 1.5 \pm 0.2% respectively.

At each gauge length, three- and two-parameter Weibull statistics were applied according to Equations 1 to 13. Linear fittings were always obtained by a classical least-squares method with a coefficient of correlation r^2 . The value of the adjustable parameter σ_u was determined by the highest value of r^2 .

Finally, the tensile strength $\sigma_f(l_c)$ of the fibres at their critical length l_c in epoxy resin, determined in a previous study [7] by a fragmentation test on single fibre-epoxy composites, was estimated in all cases by means of Weibull equations as well as by linear extrapolation. The experimental value of l_c was always taken equal to 0.43 mm. Obviously, this length was too small to perform a direct experimental determination of tensile strength.

4. Results and discussion

In order to check if the Weibull treatment is appropriate for our TI carbon fibres, Fig. 1 gives, for example, the variation of $\ln \{\ln [1/(1 - P)]\}$ with $\ln \sigma$ at different gauge lengths $l_{\rm g}$ varying from about 3 to 100mm. In this case, the two-parameter Weibull distribution and the estimator defined by Equation 2 are used. In agreement with Equation 10, straight lines having slope equal to the Weibull modulus *m* are obtained. Fig. 1 also shows that it is difficult to evidence a bi- or multi-modal distribution of fibre strength as observed in other studies [11-14] for different fibres. Nevertheless, in a few cases ($l_g = 2.94$) and 27.55mm) it could be possible to draw two

Figure 1 ln $[-\ln(1 - P)]$ plotted against ln (σ) (two-parameter Weibull analysis) for untreated T1 carbon fibres at different gauge lengths: (a) 2.94 mm, (b) 4.92 mm, (c) 9.96 mm, (d) 27.55 mm, (e) 45.96 mm, (f) 100.43 mm (estimator = $(i - 0.5)/N$).

Estimator m σ_0 σ_u σ_w r^2 CV $\sigma_f(l_c)$ (GPa) (GPa) (GPa) $(\forall o)$ (GPa) *i - 0.5IN* 2.67 31.325 1.877 3.127 0.988 40.3 7.822 *i/N* + I 2.92 26,202 1.750 3.127 0.993 37.2 7,463 *i - 0.3/N +* 0.4 2.79 28.513 1.820 3.127 0.990 38.8 7.623 *i - 3/8/N +* 0.25 2.75 29.378 1.840 3.127 0.990 39.3 7.684

TABLE I Influence of the estimator on the Weibull parameters and mean TI fibre strengths (three-parameter analysis; gauge length = 27.55 mm; sample size = 85).

straight lines of different slopes, thus defining two different modes of fibre failure. However, this presumed bi-modal strength distribution is not systematically found in the other graphs. It is particularly clear for $I_g = 27.55$ which is a unique case of supposed bi-modality in this range or gauge lengths. Therefore this assumption can be rejected. It is more hazardous to suppose that a unimodal strength distribution can describe the results at $l_g = 2.94$ mm. It is possible that a new mode of fibre failure appears at these short gauge length. Experiments at shorter lengths should be carried out in order" to confirm or disprove this fact, but the difficulty of making tensile strength measurements at gauge lengths inferior to about 2.5 to 3 mm is considerable.

Finally, it is concluded that the uni-modal Weibull model can be used to describe the strength behaviour of HT carbon fibres.

Tables I and II show the influence of the estimator chosen (Equations 2 to 4) on the Weibull parameters m, σ_0 and σ_u for three- and two-parameter distributions respectively, at a given gauge length I_g = 27.55 mm. It appears that the results are not or only slightly modified by the mathematical form of the estimator. In particular, the values of the modulus m are only affected by a few per cent. For this gauge length the arithmetical mean of the tensile strength $\sigma_{\rm arith}$ was found to be 3.124 GPa. The mean strengths σ_w determined by the three- or two-parameter Weibull distributions are very close to this value in all cases as shown in Tables I and II. As expected, it also appears that the coefficient of correlation r^2 is higher in the case of three-parameter function, since an adjustable parameter $\sigma_{\rm u}$ is used, than in the case of two-parameter distribution. It must be noted, however, that $\sigma_{\rm u}$ takes very high values, equal to about half those of σ_w . Moreover, the values of CV indicate that the variation of the distribution is more important for a threeparameter determination than for a two-parameter one.

Finally, in both cases, estimated values (Equations 9 and 12) of the tensile strength $\sigma_f(l_c)$ of the fibre at the critical length $l_c = 0.43$ mm are also given in Tables I and II respectively. It is thus shown that the choice of an estimator does not affect the value of $\sigma_f(l_c)$, whereas the method used (three- or two-parameter) does considerably change this value.

In agreement with other studies [8-10], since the mathematical form of the estimator is not an important parameter in determining the strength of HT carbon fibres, the estimator defined in Equation 2 will be used by now to calculate the cumulative failure probability P.

Table III gathers the results concerning the influence on the Weibull parameters of the sample size N. These results are given as examples and correspond only to a two-parameter Weibull function ($\sigma_u = 0$) and at a gauge length $l_p = 27.55$ mm. Experimentally, 15 measurements of tensile strength are first obtained and then the sample size is progressively increased by steps of 10 specimens until $N = 85$. It is seen that the coefficient of correlation r^2 first increased to reach a constant value for N greater than or equal to 35. At the same time, the modulus m and the coefficient of variation CV are very slightly affected for all the values of N except for the smallest one. Whatever the sample size, the values of σ_w and σ_{arith} are identical. It is also shown that $\sigma_f(l_c)$, calculated by Equation 12, is kept almost constant in the range of sample sizes studied, the relative scatter on the mean value of $\sigma_r(l_c)$ being less than about $\pm 3\%$. Similar results are obtained for a three-parameter Weibull analysis. Consequently, it could be concluded that a sample size equal to about 20 is sufficient to obtain results which are statistically valid and, in particular, to lead to a good estimation of the tensile strength of the fibre at short lengths. Hence, for the continuation of this paper, the sample sizes will be always equal to about 20.

Tables IV and V present the results concerning the influence of the gauge length $l_{\rm g}$ on the parameters m, σ_0 , σ_u and the mean fibre strengths for three- or twoparameter Weibull distributions respectively. It immediately appears that, in both cases, m , σ_0 and σ_u are not constant and accordingly the values of $\sigma_f(l_c)$ (Equations 9 and 12 respectively) are greatly affected.

Whatever the gauge length, the values of $\sigma_f(l_c)$ and the dispersion on these values are greater in the case of

TABLE II Influence of the estimator on the Weibull parameters and mean TI fibre strengths (two-parameter analysis; gauge length = 27.55 mm; sample size = 85).

Estimator	\boldsymbol{m}	$\sigma_{\rm n}$ (GPa)	$\sigma_{\rm w}$ (GPa)		CV (0/0)	$\sigma_{\rm f}(l_{\rm c})$ (GPa)
$i - 0.5/N$	7.77	9.632	3.124	0.958	15.2	5.334
$i/N + 1$	7.43	10.128	3.120	0.973	15.9	5.461
$i - 0.3/N + 0.4$	7.62	9.844	3.122	0.966	15.5	5.388
$i - 3/8/N + 0.25$	7.68	9.768	3.123	0.963	15.4	5.369

TABLE III Influence of the sample size on the Weibull parameters and mean T1 fibre strengths (two-parameter analysis; gauge length = 27.55 mm; estimator = $(i - 0.5)/N$).

N	m	σ_0 (GPa)		$\sigma_{\rm w}$ (GPa)	CV $(\%)$	$\sigma_{\rm{arith}}$ (GPa)	$\sigma_{\rm f}(l_{\rm c})$ (GPa)
15	9.62	8.047	0.905	3.233	12.5	3.235	4.982
25	9.03	8.311	0.914	3.148	13.2	3.149	4.989
35	7.98	9.437	0.966	3.149	14.9	3.150	5.304
45	7.98	9.333	0.968	3.115	14.9	3.115	5.246
55	7.84	9.404	0.956	3.076	15.1	3.076	5.230
65	7.88	9.395	0.956	3.091	15.1	3.091	5.242
75	8.23	8.964	0.960	3.094	14.4	3.094	5.127
85	7.77	9.632	0.958	3.124	15.2	3.124	5.334

TABLE IV Influence of the gauge length on the Weibull parameters and mean Tl fibre strengths (three-parameter analysis; estimator $=$ $(i - 0.5)/N$).

the three-parameter function than those of the twoparameter function. On the contrary, the coefficient of variation CV is lower for the two-parameter method than for the three-parameter one. As discussed in the theoretical section, it is assumed that the Weibull parameters are independent of the gauge length in order to estimate the tensile strength of the fibres at any lengths. Experimentally, it is now clearly proved that this assumption is not valid and the measurements at each gauge length lead to a value of the tensile strength different from the other one. Nevertheless, as already shown, whatever the gauge length, the arithmetical σ_{arith} and the Weibull σ_{w} mean strengths are almost identical in both cases. Finally, we can conclude that the three- or two-parameter Weibull distributions, although they are well adapted to describe the strength behaviour of high strength carbon fibres at a given gauge length, are not appropriate to estimate their tensile strength at short lengths.

It is, therefore, necessary to verify if the last method presented above and corresponding to Equation 14, i.e. a logarithmic dependence of mean strength on gauge length, can be used for this kind of estimation. It has been well established that in all cases, whatever the factor examined in this study (estimator, sample size and gauge length) the mean fibre strengths σ_{arith} and σ_w are equal. Therefore, it is easier to use directly the values of σ_{arith} than the other ones in Equation 14.

Fig. 2 shows, that for the 11 gauge lengths listed in Tables IV or V, such a linear relationship does indeed exist. The following parameters can then be calculated according to Equation 14.

> $m = 6.73$ $\sigma_0 = 5.435 \text{ GPa}$ $CV = 17.43$

 $= 0.906$

TABLE V Influence of the gauge length on the Weibull parameters and mean T1 fibre strengths (two-parameter analysis; estimator = $(i - 0.5)/N$).

and

Figure 2 Mean tensile fibre strength plotted against gauge length in logarithmic scale for untreated T1 carbon fibres.

As previously done, the tensile strength $\sigma_f(l_c)$ of the fibre at the critical length $l_c = 0.43$ mm can be easily estimated and the value of 5.748 GPa is obtained. This value is rather different from the other ones listed in Tables I to V. It appears that this method is the most accurate and the simplest one in order to be able to extrapolate at very short length the tensile strength of carbon fibres. This confirms other published results [12, 15-18]. It is worth noting that this method does not need any estimator, since we use directly σ_{arith} , or any other parameters previously determined at long lengths. Moreover, unique values of m, σ_0 and $\sigma_f(l_c)$ are obtained. Nevertheless, it is necessary to carry out experiments at, at least, six or seven different gauge lengths. This can be easily made, without any risk, by taking sample sizes equal to about 20 for each gauge length.

As shown in Fig. 3, this method also describes well the length dependence of strength of the T2 carbon fibres. The measurement are made at seven different gauge lengths and for sample sizes equal to about 50 in each case (total number of specimens tested $=$ 390) [19]. The calculated parameters of the straight line are the following

$$
m = 11.55
$$

\n
$$
\sigma_0 = 4.120 \text{ GPa}
$$

\n
$$
\text{CV} = 8.65\%
$$

and

$$
r^2 = 0.993
$$

Thus, it appears that the tensile strength of T2 fibres are less dependent on length than for T1 fibres. This can be explained by a flaw density of the T2 fibres greater than that of T1 fibres, since its Weibull modu-

Figure 3 Mean tensile fibre strength plotted against gauge length in logarithmic scale for untreated T2 carbon fibres.

Figure 4 Mean tensile fibre strength plotted against gauge length in logarithmic scale for oxidized T1 carbon fibres.

lus m is greater. This is confirmed by the calculation of their tensile strength at l_c . We find $\sigma_f(l_c) = 4.240 \text{ GPa}$ which is about 25% lower than the $\sigma_f(l_c)$ value (5.748 GPa) for T1 carbon fibres.

It is well known that appropriate surface treatments can modify not only the chemical surface properties of a fibre but also its mechanical behaviour and, in particular, its tensile strength by creating a great number of new flaws. Accordingly, the length dependence of the tensile strength of oxidized T1 carbon fibres was studied. Fig. 4 shows again that a linear relationship betwen σ_{arith} and l_{g} in logarithmic scales can be established for six different gauge lengths and for sample sizes of about 20 (125 specimens). The following values of the parameters are obtained

$$
m = 6.06
$$

\n
$$
\sigma_0 = 5.716 \text{ GPa}
$$

\n
$$
\text{CV} = 19.2
$$

and

$$
r^2 = 0.956
$$

Surprisingly, the value of the modulus m is lower than the one corresponding to untreated T1 fibres $(m = 6.73)$. It is however conceivable that the oxidation treatment can reduce the surface flaw density (or the stress concentration at the tip of the flaws) by a simple phenomenon of etching leading to a smoother surface. This result points out to the fact that the fibre surface flaws seem to be the most important flaws in controlling the strength of HT carbon fibres.

5. Conclusion

In this study, the determination of the tensile strength of high strength carbon fibres and its gauge length dependence was analysed by means of the Weibull model. It was first shown that the mathematical form of the estimator chosen and the sample size, when higher than about 20, do not influence the results. Secondly, it appeared that the three- as well as the two-parameter Weibull distribution are not well appropriate to describe the length dependence of the fibre strength since the experimentally determined parameters are not independent on gauge length as stated by the theory. Finally, it was shown that a linear logarithmic dependence of strength on gauge length is the most accurate and the most simple method to extrapolate the fibre tensile strength at short lengths. This method seems to be of wide applicability, at least for high strength carbon fibres, since it also works well on the surface treated fibres.

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