DETERMINATION OF THE TRUE STATISTICAL FLAW STRENGTH PARAMETERS FOR CERAMIC FIBRES FROM TESTS ON TOWS

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Keywords: fibers, fracture statistics, Weibull, normal distribution

Abstract

The present paper investigates fracture statistics for brittle fibers used for composite reinforcement. Large sets of failure strengths (500 to 1000 data) were produced using tensile tests on tows that contained either 500 or 1000 filaments. The statistical distributions of filament strengths were described using the normal distribution. The Weibull distribution fitted the normal distributions. A quite negligible scatter in Weibull statistical parameters was observed. Then, the estimator-based method of estimation of Weibull parameters was applied to subsets selected from the initial large ones. Finally, the factors that dictate Weibull parameters variability are discussed.

1. Introduction

Fibers are key constituents for composites, as they carry most or all the load. They exhibit a wide variability in strength. The Weibull distribution is one of the widely used distributions in fracture statistics, and reliability engineering, owing to its simple form. It is a versatile distribution that is very sensitive to the statistical parameters. Thus, the estimation of statistical parameters is an important issue for failure prediction purposes. There has been a great deal of papers on the variability in Weibull statistical parameters. In quite all the cases, the authors looked for methods of correction of the estimates that have been obtained on limited sample sizes. For this purpose, they used more or less complex analyses and computations to define appropriate estimators of experimental failure probability or they introduced additional parameters into the Weibull equation in order to improve the fit to experimental distribution of strength data [1-7].

The estimation of Weibull statistical parameters from experimental failure data may be skewed as a result of:
- the use of the construction of a so-called Weibull plot of strength data that requires an estimator for the determination of the failure probabilities associated to experimental data.
- the sample of data that may be undersized [8], especially for highly heterogeneous materials, i.e. containing large amounts of flaws with broad size interval.
- the derivation of strength data from experimental test results.

The present paper investigates large sets of failure strengths measured on tows made of several hundreds of parallel ceramic filaments. As a consequence, a method to determine the true statistical flaw strength parameters for ceramic single filaments was proposed. The term flaw strength is used because the fibers are loaded under tension, so that the strength of a fiber represents the strength of the flaw that caused fracture. As discussed in previous papers [9-11], a tensile test on a tow provides the strengths of the single filaments it is made of. So, this technique is powerful to generate very large sample sizes. The normal distribution is the most appropriate for large sample sizes. It presents the following features: it indicates the probability of occurrence of a characteristic in a population of infinite size, and certain distributions can be approximated by the normal distribution when the sample size is large (for example, the binomial distribution, the Poisson distribution, the chi-squared distribution, the Student's-t-distribution). It is reported that this trend is also observed with the Weibull distribution when the shape parameter $3 \leq m \leq 4$ [12].

The Weibull distribution was fitted to the normal distribution of filament strengths, giving the Weibull statistical parameters. The great advantage of this approach is that an estimator of failure probability is not necessary. The influence of the above-mentioned skewing factors was examined. A certain emphasis was placed on sampling. Strain instead of stress was used as fiber strength, which allows a source of variability to be eliminated, since the diameter of each filament is not required.

2. Experimental

Test specimens that contained either 500 or 1000 SiC-based Nicalon filaments were prepared according to the protocol described in a previous paper [9]. The main filament characteristics are: nominal diameter ranged from 10-15 micrometers, Young's modulus E_f = 200 GPa [10,13,14]. The precise number of filaments was determined from the initial slope of the stress-strain curve [10,11].

The tensile tests were carried out at room temperature under monotonous loading (displacement rate = 2μm/sec) on a servo-pneumatic testing machine equipped with a 500 N load cell. Test specimen elongation was measured using a contact extensometer (with a ± 2.5 mm elongation displacement transducer) that was clamped to the specimen using two 4-mm long thermo-retractable rings. The rings were located close to the grips in order to avoid possible bending introduced by the extensometer. The inner distance between the rings defined the gauge length (115 mm). Thus, strain measurement was direct and unpolluted by load train deformations. The samples were first loaded up to 5% of the ultimate load, and then the extensometer was put and adjusted. Lubricant oil was used to avoid friction between the fibres.

Acoustic emission monitoring allowed detection and counting of fiber fractures [11], in order to determine the strain-to-failure data histograms. Two resonant PZT transducers (Acoustic Emission type μ 80) were placed at specimen ends, in order to locate fracture origins. Only those events located in the gauge length, and those signals with amplitude > 60 dB were kept. The transducers were acoustically connected to the samples with vacuum grease. A two channel Mistras 2001 data acquisition system of Physical Acoustics Corporation (PAC) was used for the recording of AE data. A fixed threshold of 32 dB was selected for minimising interference noise from outside.

3. Statistical analysis of fracture data

The cumulative distribution function of fiber failure data was described using the normal distribution. It was obtained probability density function:

$$P_N(\mathrm{E} \leq \varepsilon) = \int_0^{\varepsilon} f(\varepsilon)\, d\varepsilon \tag{1}$$

$$f(\varepsilon) = \frac{1}{S\sqrt{2\pi}} \exp\left[-\frac{(\varepsilon-\mu)^2}{2S^2}\right] \tag{2}$$

where ε is the strain to failure, μ is the mean and S is the standard deviation.
S and μ were obtained by fitting equation (2) to the histogram of fiber failure data (N_i, ε_i), where N_i is the number of acoustic emission events (number of fiber failures) during a 0.1% increment at deformation ε_i.

For a uniform tensile stress (σ), the Weibull equation of failure probability P_W reduces to:

$$P_W = 1 - \exp\left[-\frac{V}{V_0}\left(\frac{\sigma}{\sigma_0}\right)^m\right] \tag{3}$$

$$P_W = 1 - \exp\left[-\frac{V}{V_0}\left(\frac{\varepsilon}{\varepsilon_0}\right)^m\right] \tag{4}$$

with V the stressed volume, V_0 a reference volume (for instance $1m^3$), the scale parameters: $\varepsilon_0 = E_f\, \sigma_0$.

The Weibull statistical parameters were estimated by fitting equation (4) to the normal distribution of strains-to-failure.
In a second step, they were estimated by fitting equation (4) to the Weibull plot of strain failure data. The so-called Weibull plot was constructed, using an estimator for estimation of failure probabilities associated to strain data ranked in ascending order. The estimator $P_j = (j-0.5)/N$ is recommended for limited sample sizes (*j* is the rank of filament strain-to-failure). Statistical parameters were then obtained by fitting equation (4) to the Weibull plot of P_j vs. ε_j.
In a third step, subsets of 20 and 30 strain-to-failure data were used in order to illustrate the effects of sample size, and of sampling (five draws per sample size). The subsets were selected from a large distribution of filament strengths using a random process (five draws per sample size). Such sample sizes are generally used for the estimation of statistical parameters. Then, an additional subset comprised only the failure data prior to maximum load.

4. Results

A typical force-strain curve together with locations of acoustic emission events in the gauge length is shown in figure 1. The curve displays the conventional features of bundle tensile behavior, i.e. initial elastic deformations for strains < 0.5%, and then non-linear deformations as a result of individual fiber breaks as indicated by acoustic emission events. Note that the load decreases progressively and regularly to 0, and that the density of AE event sources is homogeneous which suggests that fiber interactions probably did not operate.

Figure 2 shows that the histogram of strain-to-failure data has a symmetrical bell shape about its mean, and that it is fitted by the normal distribution function. Quite identical statistical parameters were estimated from three bundle tests (table 1). Figure 3 shows that the corresponding normal cumulative distribution function fits quite well the Weibull distribution function for the statistical parameters reported in table 1. It is worth pointing out that the scatter in scale factors is quite negligible. Shape parameter values (table 1) show a very small variation (5.23-5.43) when comparing to the data reported in the literature (2.3-7.1 [14-16]). Referring to the limited scatter in Weibull parameters that was obtained, and to the large size of the data samples that were analyzed, it can be considered that these are the true statistical parameters. It is worth pointing out that the Weibull distribution fits a normal distribution when the sample size is large. This result is at variance with the literature that indicates that this is obtained only when $m \leq 3.6$.

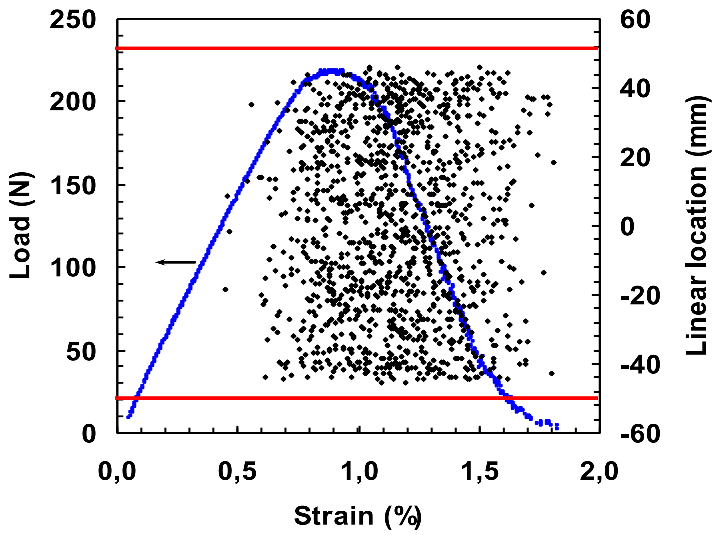


Figure 1. Load-strain curve and location of AE events along specimen axis for a Nicalon fibre bundle (test specimen 2). The horizontal lines delineate the gauge length.

test specimen	Number of filaments	Normal distribution $\mu(\%)$	$S(\%)$	Weibull distribution m	$\varepsilon_{\ell}(\%)$
1	487	1.16	0.25	5.30	1.25
2	986	1.11	0.24	5.23	1.20
3	924	1.15	0.24	5.43	1.23

Table 1. Statistical parameters of Normal and Weibull distributions of flaw strengths for Nicalon filaments $\left(\varepsilon_l = \varepsilon_0 \left(V_o / V\right)^{\frac{1}{m}}\right)$

The well-known effect of sample size on variability is obviously observed (table 2). The m values obtained for a given small sample size show a wide variation (table 2). Note that the scale parameter was also affected. The parameter variation at constant sample size results from sampling. The number of possible subsets is given by the binomial coefficient:

$$C_n^N = \frac{N!}{(N-n)!n!} \tag{5}$$

For n=20 and n=30 it is quite huge: $C_{20}^{1000} = 3.410^{41}$, $C_{30}^{1000} = 3.110^{27}$. This suggests how high is the probability that were different the samples of Nicalon filaments that have been used by authors for the determination of Weibull parameters [15 and references therein].

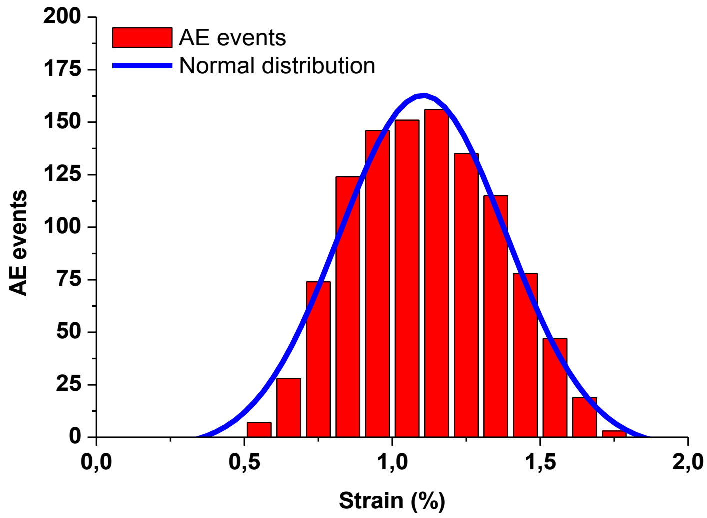


Figure 2. Typical histogram of strain-to-failure data obtained on test specimen 2.

Population size	Weibull plot	
	m	ε_ℓ (%)
20	4.75- 7.18	1.20-1.31
30	4.55-6.05	1.23-1.24
168*	11.20	0.79

** Number of events prior to maximum load*

Table 2. Weibull parameters estimated using subsets (20, 30 and failure data prior to maximum load) selected from failure data obtained on specimen 2 ($P_j = (j-0.5)/N$).

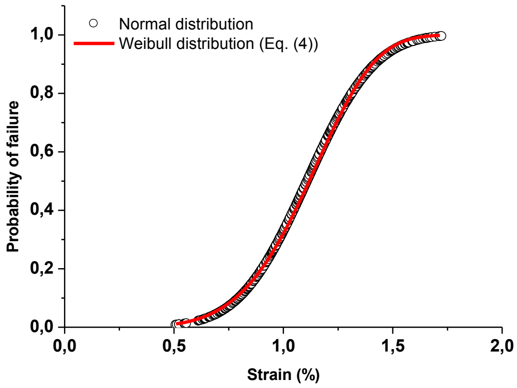
Figure 4 compares the corresponding linearized Weibull plots to the normal distribution (log-log coordinates). A significant discrepancy is observed, particularly for the subset of failure data prior to maximum load. m values as large as 11 were estimated (table 2). The above particular subset is an extreme one for the size of 168 data since it comprises all the lowest strengths: m= 11.2 is thus an upper bound for this sample size (table 2).

5. Discussion

There are several objective reasons why it can be considered that the true Weibull parameters have been determined: m = 5.2 and ε_l = 1.20% $\left(\varepsilon_l = \varepsilon_0 (V_0 / V)^{\frac{1}{m}}\right)$.

First, statistically significant sample sizes were used, and the shape parameter estimates showed quite negligible variation.

Second, those parameters that affect the analysis have been eliminated: sampling, sample size, use of an empirical estimator.



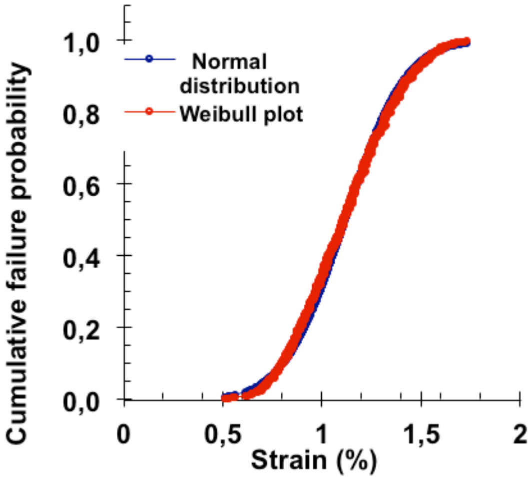


Figure 3. Cumulative distribution functions of failure strains for Nicalon filaments obtained for test specimen 2: (a) Normal distribution vs. Weibull distribution (equation (4)); (b) Normal distribution vs. Weibull plot ($P_j = j / N$).

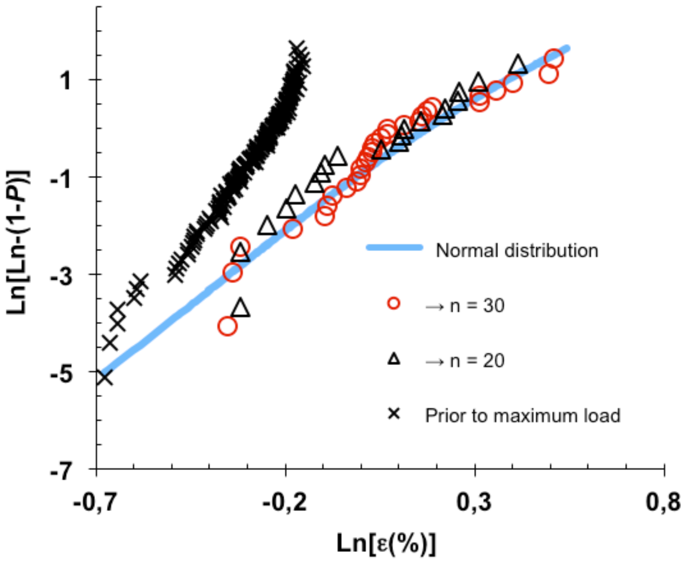


Figure 4. Weibull plots of strain-to-failure data obtained on subsets (20, 30 and data prior to maximum load) derived from the set of data for test specimen 2.

However, the question may arise on the pertinence of the failure data that have been generated experimentally. Fiber interactions can influence the results, since they can cause either overestimation of the force on fibers (owing to the effect of a frictional force $F_{tot} = F_{true} + F_{fric}$) or fracture of several fibers (leading to a steep force decrease beyond maximum). The effect of fiber interactions during the tests was investigated by comparing the experimental force-strain curve with that one predicted using the bundle model for parallel and independent fibers. The force-strain relation during a tensile test is given by [17]:

$$F(\varepsilon) = N\left[1 - P(\varepsilon)\right] S_f E_f \varepsilon \tag{7}$$

where $P(\varepsilon)$ is the probability of failure at strain ε, S_f is the average filament cross sectional area. Figure 5 shows that there is an excellent agreement between experiment and theory. It can be noticed that the force decrease beyond maximum compares fairly well with that obtained experimentally. It cannot be concluded that it is steeper, as it is obtained when groups of filaments fail [10]. All these results converge on the conclusion that there was not significant pollution by fiber friction.

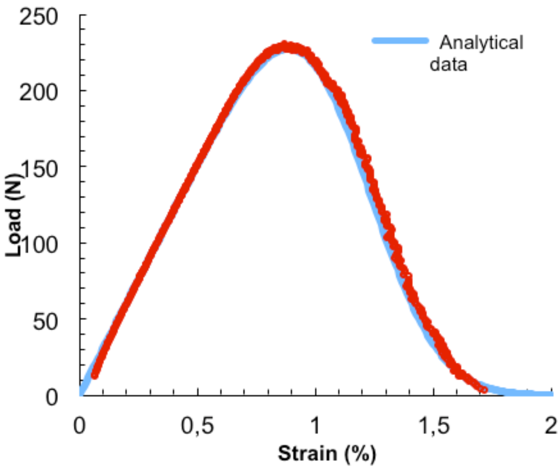


Figure 6. Comparison of experimental and predicted load-strain curves for a Nicalon fibre bundle (test specimen 2).

6. Conclusions

One of major results of the present paper is that fiber flaw strengths follow a normal distribution. Then the validity of the Weibull distribution was assessed. It is important to note that the analysis used large sets of failure data (500 to 1000) determined experimentally, as opposed to most approaches that use data generated using computations with the Weibull equation for given statistical parameters.

The values of statistical parameters that were derived from the comparison of Normal and Weibull distributions of filaments failure data can be considered as the true ones for the tested SiC fiber. In particular, the true value of m is about 5.2. The sources of variability (fiber diameter, sample size, sampling, empirical estimators) have been minimized. It has been shown that variability in statistical parameters is essentially a matter of sample size and sampling (selection of test-specimens). Using various estimators or modified Weibull equations will not permit to correct the errors associated to samples of data.

These data can be regarded as reference data for failure predictions for the commercial fibers examined in this work. Performing new analysis of a limited sample size would provide results that would not be correct, as demonstrated in this paper.

7. References

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