Flaw strength distributions and statistical parameters for ceramic fibers: The normal distribution

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The present paper investigates large sets of ceramic fibre failure strengths (500 to 1000 data) produced using tensile tests on tows that contained either 500 or 1000 filaments. The probability density function was determined through acoustic emission monitoring which allowed detection and counting of filament fractures. The statistical distribution of filament strengths was described using the normal distribution. The Weibull equation was then fitted to this normal distribution for estimation of statistical parameters. A perfect agreement between both distributions was obtained, and a quite negligible scatter in statistical parameters was observed, as opposed to the wide variability that is reported in the literature. Thus it was concluded that flaw strengths are distributed normally and that the statistical parameters that were derived are the true ones. In a second step, the conventional method of estimation of Weibull parameters was applied to these sets of data and, then, to subsets selected randomly. The influence of other factors involved in the conventional method of determination of statistical parameters is discussed. It is demonstrated that selection of specimens, sample size, and method of construction of so-called Weibull plots are responsible for statistical parameters variability.

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I. INTRODUCTION

The fracture of many materials including ceramics is caused by microstructural flaws that act as stress concentrators. The flaws are generally distributed randomly, and they exhibit wide variability in severity, as a result of variability in shape, nature, size, and location with respect to the stress state. As a consequence, stress-induced fracture is a stochastic event, and fracture stresses measured on specimens with identical dimensions have a statistical distribution.

There are various approaches to fracture statistics for brittle materials, and a particular effort was devoted to brittle materials or ceramics [1–10]. The fundamental ones recognize the flaws as physical entities, as well as the contribution of flaw severity [4–10]. Severity of fracture-inducing flaws is measured either using flaw size or flaw strength. In particular, in the so-called elemental strength approach, flaw strength is defined using an elemental strength that is the local stress that causes extension of a flaw. The elemental strength concerns microscopic length scale. By contrast, the Weibull approach to brittle fracture considers the failure stress of component (macroscopic length scale). The distribution of elemental strengths is a key issue for failure statistics.

In the elemental strength model, brittle failure is described by the following failure probability equation [5,7–10]:

$$P = 1 - \exp\left[-\int_V dV \int_0^S g(S)dS\right], \tag{1}$$

where $g(S)$ is the flaw density function and S is the elemental strength.

The power law was found to be satisfactory for flaw strength distribution $g(S)$. It allowed sound failure predictions using Eq. (1) for ceramics under various loading conditions (see [10] and references therein). However, the estimated constants generally exhibit some variation. This issue has not been solved properly, despite much effort by many researchers [11–17].

There are two possible solutions that are proposed in the present paper: either solve the constant variability issue or identify an alternative flaw strength distribution.

The normal distribution should be a natural solution. It is considered the most prominent probability distribution in statistics. It indicates the probability of occurrence of a characteristic in a population of infinite size. Then, certain distributions can be approximated by the normal distribution when the sample size is large (for example, the binomial distribution, the Poisson distribution, the χ-squared distribution, and the student's t-distribution). It is reported that this trend is also observed with the Weibull distribution when the shape parameter $3 \leqslant m \leqslant 4$ [18].

Normal distribution is not used in fracture statistics. It has been assumed by a few researchers for the distribution of strengths in the locality of flaws [19–21]. But, no satisfactory demonstration was proposed up to now. Furthermore, the sets of failure data in [21] were not statistically relevant (nine data) so that it cannot be considered that normal distributions were obtained [22].

The objective of the present paper is to examine the validity of the normal distribution for the distribution of flaw strengths. Then, approximation of the flaw density function using the power law was evaluated with a view to estimate the true constants. For this purpose, the present paper investigates large sets of flaw strengths measured on ceramic fibres. Under uniaxial uniform tension, failure stress of a filament is the strength of the flaw that caused filament fracture (flaw strength or elemental strength). For this reason, failure tests on fibres are of great interest to determine the flaw strength distributions. Tests were performed on tows made of several hundreds of parallel ceramic filaments. As discussed in previous papers [23–25], 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: in this work about 500 or 1000 data per test. This is the only possible way to generate large databases within a reasonable amount of time and in a repeatable manner. Moreover, accurate flaw strength distributions for fibres are required for sound

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failure predictions and proper modeling of multiple cracking in fibre-reinforced ceramic matrix composites.

II. STATISTICAL ANALYSIS OF FAILURE DATA

A. Fracture statistics

The fundamental equations of failure probability are recalled first with a view to highlight the significance of underlying flaw strength distribution. It is demonstrated that Eq. (1) reduces to the following equation, when the distribution of elemental strengths is described using a power law [7–10] with constants m and λ_0 (connection between brittle failure and distribution of extreme values):

$$P = 1 - \exp\left[-\frac{V}{V_0} K \left(\frac{\sigma_{\mathrm{ref}}}{\lambda_0}\right)^m\right], \tag{2}$$

where λ_0 is a scale factor and m is a shape parameter. K is obtained by integrating the stress state over stressed volume V. K depends on the probabilistic model that is considered [7–10]. σ_{ref} is a reference stress (peak stress) in the stressed volume V. V_0 is the reference volume ($V_0 = 1$ m^3 when International Units are used).

Using Eq. (2), it is demonstrated that the Weibull equation of failure probability is a particular solution of Eq. (1) [5,10]. Under a uniform uniaxial tension, $K = 1$, σ_{ref} is the specimen tensile strength, so that Eq. (2) reduces to

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

where σ_0 is the scale factor. In this particular case, $\sigma_0 = \lambda_0$.

Equation (1) can be used to predict failure in several loading cases including multiaxial stress-states [7–10], for various formulas of flaw density function.

The power law distribution of extreme values (often referred to as the Weibull distribution) is a widely used distribution 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 true statistical parameters is an important issue for failure prediction purposes when dealing with power law distributions. 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 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 [11–17]. The estimation of Weibull statistical parameters from experimental failure data may be skewed as a result of the following critical steps.

(i) 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.

(ii) The selection of a sample which may be undersized [26], especially for highly heterogeneous materials, i.e., containing large amounts of flaws with broad size range.

(iii) The derivation of strength data from applied load, which may not be easy for certain geometries (like small diameter fibres) or loading conditions.

B. Normal distribution-based analysis

Strain instead of stress-based fibre strength was used, which allowed elimination of source of variability associated to fibre diameter. Equations of Gaussian probability density function $f(\varepsilon)$ and normal distribution $P_N(E < \varepsilon)$ are

$$f(\varepsilon) = \frac{1}{S\sqrt{2\pi}} \exp\left[-\frac{(\varepsilon - \mu)^2}{2S^2}\right], \tag{4}$$

$$P_N(E \leqslant \varepsilon) = \int_0^{\varepsilon} f(\varepsilon) d\varepsilon, \tag{5}$$

where ε is the strain to failure, μ is the mean, and S is the standard deviation.

S and μ were obtained by fitting Eq. (4) to the histogram of fibre failure data N_i vs ε_i, where N_i is the number of failures during a deformation increment ($\Delta\varepsilon = 0.1\%$).

For a single gauge length and uniform tensile stresses, the following strain-based Weibull equation of failure probability P_W is derived from Eq. (3):

$$P_W = 1 - \exp\left[-\left(\frac{V}{V_0}\right)\left(\frac{\varepsilon}{\varepsilon_0}\right)^m\right] = 1 - \exp\left[-\left(\frac{\varepsilon}{\varepsilon_l}\right)\right], \tag{6}$$

with $\varepsilon_0 = \sigma_0/E_f$ and $\varepsilon_l = \varepsilon_0(\frac{V_0}{V})^{1/m}$.

The statistical parameters were estimated by fitting Eq. (6) to the normal distribution of strains to failure.

C. Conventional estimator-based approach

A so-called Weibull plot is constructed, using an estimator for estimation of failure probabilities associated to strain data ranked in ascending order. Various estimators have been devised in the literature [11–17]. The estimator $P_j = j/N$ can be used on large sample sizes, as in the present paper. 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 are then obtained by fitting Eq. (6) to the Weibull plot of P_j vs ε_j. When a linear regression analysis method is used, Weibull modulus m is determined as the slope of the Weibull plot of $\ln[-\ln(1 - P)]$ vs $\ln\varepsilon$:

$$\ln[-\ln(1 - P)] = m \ln\varepsilon + \ln k, \tag{7}$$

where k is a constant: $k = V/V_0\varepsilon_0^m$.

In the second step, subsets of 20 and 30 strain-to-failure data were selected randomly (five draws per sample size) from a set of 1000 failure strains (obtained on specimen 2) in order to simulate the effects of sample size and of sampling. Such sample sizes are generally used for the estimation of statistical parameters. The conventional method of estimation of Weibull parameters with $P_j = (j - 0.5)/N$ as estimator was used.

In the third step, the subset corresponded only to the failures prior to maximum load.

III. EXPERIMENT

A. Bundle test specimens

The bundle test specimens contained either 500 or 1000 SiC-based Nicalon filaments. The main filament characteristics are nominal diameter 10–15 micrometers, Young's modulus (E_f) 200 GPa [24,27,28]. The exact number of filaments was determined from the initial slope of the force-strain curve [25,27]. Test specimens were prepared according to the protocol described in a previous paper [23].

B. Tensile tests on bundles

The tensile tests were carried out at room temperature under monotonous loading (displacement rate = 2 μm/s) on a servopneumatic 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 thermoretractable 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 placed and adjusted. Lubricant oil was used to avoid friction between the fibres.

Acoustic emission monitoring was aimed at detecting and counting fibre fractures [25], in order to determine the strain-to-failure data histograms. It is worth emphasizing that, unlike acoustic emission monitoring, the load decrease curve does not permit determining the numbers of failure events during any strain steps. 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 with amplitude > 60 dB (corresponding to fibre failure) were kept. The transducers were acoustically connected to the samples by 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 minimizing interference noise from outside.

IV. RESULTS

A typical force-strain curve together with locations of acoustic emission events in the gauge length is shown in Fig. 1. The curve displays the conventional features of bundle tensile behavior, i.e., initial elastic deformations for strains <0.5%, and then nonlinear deformations as a result of individual fibre 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 fibre interactions probably did not operate.

Figure 2 shows the probability density function derived from acoustic emission monitoring. It is a bell curve described by a Gaussian function, symmetric about its mean. Quite identical parameters μ and S were estimated for the three bundles that were tested (Table I). Figure 3(a) shows the corresponding normal distribution. It also shows that the

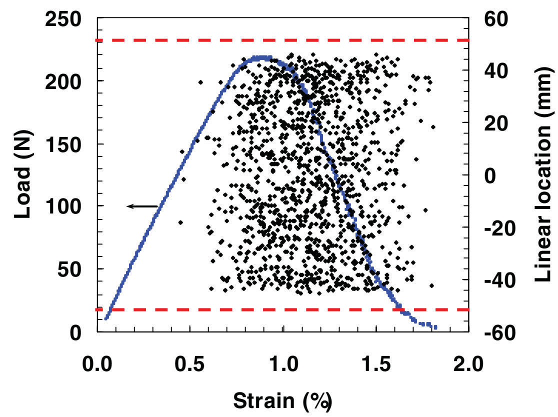

FIG. 1. (Color online) Load-strain curve and location of AE events along specimen axis for a Nicalon fibre bundle (test specimen 2). The dotted lines delineate the gauge length.

Weibull distribution function Eq. (6) fits quite well the normal distribution for the statistical parameters reported in Table I. It is worth pointing out that the scatter in the scale factors is quite negligible. The shape parameter values (5.23–5.43; Table I) show a very small variation when comparing to the data reported in the literature: 2.3–7.1 [28–30]. 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 those in the literature that indicate that this is obtained only when $m \leqslant 3.6$ ($3 \leqslant m \leqslant 4$).

A. Comparison with the estimator-based estimation approach

Figure 3(b) shows that the Weibull plot obtained for $P_j = j/N$ fits quite well the normal distribution, which confirms that $P_j = j/N$ is a satisfactory estimator with large sample sizes. Estimates quite close to the true statistical parameters were obtained (Table II). However, the m estimates are smaller and they exhibit a wider variation, whereas the scale factors agree quite well (Table II). Figure 4 compares the log-log graph of normal distribution with the linearized Weibull

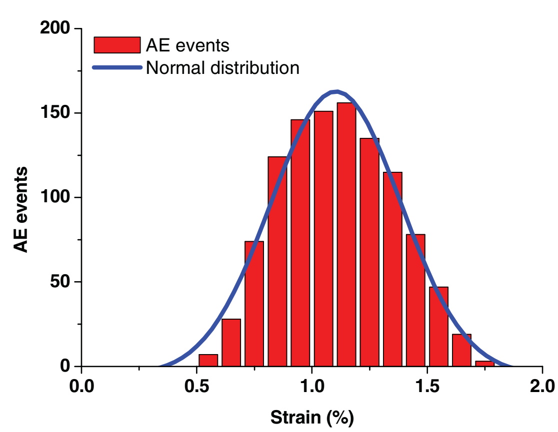

FIG. 2. (Color online) Typical histogram of strain-to-failure data obtained on test specimen 2.

TABLE I. Statistical parameters of normal and Weibull distributions of flaw strengths for Nicalon filaments. $V_0 = 1$ m^3.

		Normal distribution		Weibull distribution			
Test specimen	Number of filaments	$\mu(\%)$	$S(\%)$	m	$\varepsilon_l(\%)$	$\varepsilon_0(\%)$	σ_0(MPa)
1	487	1.16	0.25	5.30	1.25	0.01	23.4
2	986	1.11	0.24	5.23	1.20	0.01	21.1
3	924	1.15	0.24	5.43	1.23	0.01	25.7

plot. A discrepancy can be observed on Fig. 4 at the low extreme for probabilities <4%. It can be attributed to a slight underestimation of the number of first failures, as it can be noted on Fig. 2. It is related to the detection of low energy events near the filtering threshold of 60 dB (Fig. 2). The log-log scale magnifies this discrepancy. It may not be considered that the apparent presence of two domains reflects a bimodal population of fracture inducing flaws, since the probability density function has shown the presence of a single population (Fig. 2). Furthermore, previous papers on Nicalon SiC fibres did not clearly identify the presence of a bimodal population of flaws [28,30], although certain linearized Weibull plots revealed a discrepancy at the low strength extreme. Fractures from pores located in the surface or in the interior of filaments were essentially evidenced by fractography. This effect is interesting to point out an error that may result from the shape of the linearized Weibull plot at the low strength extreme.

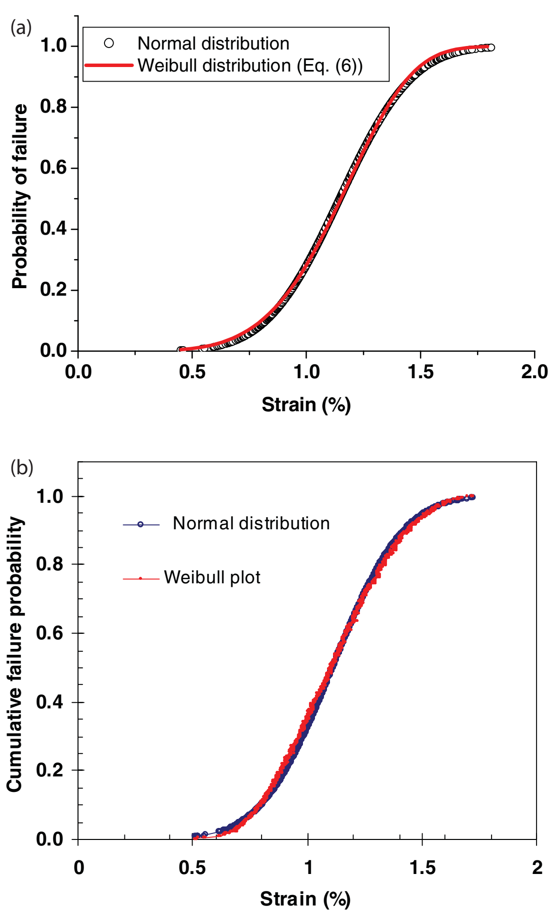

FIG. 3. (Color online) Cumulative distribution functions of failure strains for Nicalon filaments obtained for test specimen 2: (a) normal distribution vs Weibull distribution Eq. (6); (b) normal distribution vs Weibull plot ($P_j = j/N$).

B. Influences of selection and number of data

The well-known effect of increasing sample size on variability is observed here (Table III): the scatter in Weibull modulus estimates was found to decrease with increasing sample size. The m values obtained on small sample sizes show a wide variation (Table III). Note that the scale factor was also affected.

This effect is well documented in the literature. Several authors have investigated the influence of the number of data on Weibull modulus estimates. For this purpose, data sets were generated using a Monte Carlo method for given m values: in [31], $1 < m < 50$ and $10 < N < 50$, and the procedure was repeated 10^7 times, giving a large amount of data sets. Correction factors have been proposed and the diagrams that have been produced are expected to allow the number of specimens for the estimation of Weibull modulus to be refined with respect to desired accuracy.

However, it is worth pointing out that, in the present paper, only 5 samples of 20 or 30 data were selected among 1000 experimental data. Therefore, it can be anticipated that the bounds of the m intervals are not correct. The reason for this is that these sample sizes are too small when compared to the amount of data sets that can be extracted from the original one. The number of possible subsets is given by the binomial coefficient:

$$C_n^N = \frac{N!}{(N-n)!n!}. \quad (8)$$

For $n = 20$ and $n = 30$ it is quite huge: $C_{20}^{1000} = 3.4 \times 10^{41}$ and $C_{30}^{1000} = 3.1 \times 10^{27}$.

The huge number of possible subsets given by Eq. (8) indicates how high the probability is that the samples of Nicalon filaments that have been used by authors for the estimation of Weibull parameters were different (see [29] and references therein). It also indicates that sets of 10^7 data may

TABLE II. Statistical parameters estimated using the estimator-based conventional method (Weibull plot for $P_j = j/N$).

	Weibull plot	
Test specimen	m	$\varepsilon_l(\%)$
1	5.23	1.26
2	4.86	1.20
3	5.12	1.23

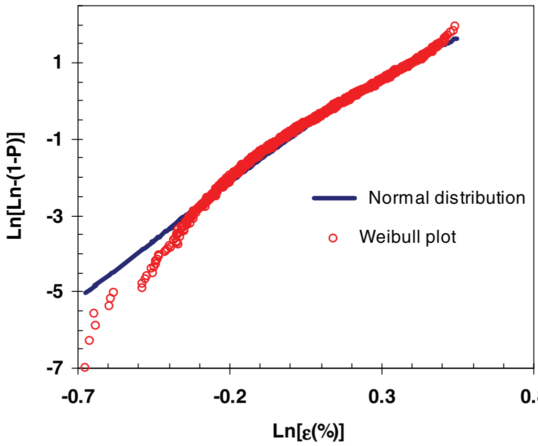

FIG. 4. (Color online) Comparison between Weibull plot (empirical distribution function "j/N") and normal distribution of strain to failure for Nicalon filaments (test specimen 2) (log-log plots).

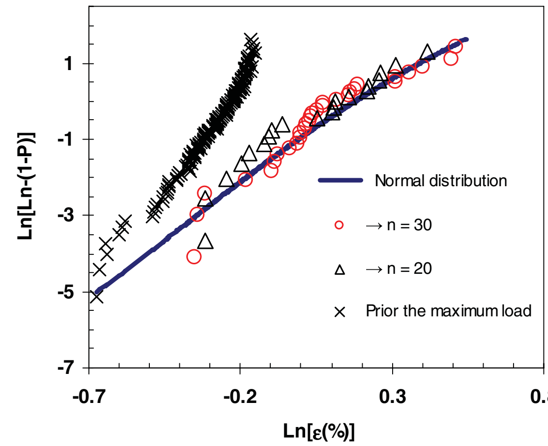

FIG. 5. (Color online) Weibull plots of strain-to-failure data obtained on subsets (20, 30, and data prior to maximum load) derived from the set of data for test specimen 2.

be too small, which may lead to underestimation of the scatter in m.

The exact values of m range can be determined. But, this requires a computerized method of selecting a very huge amount of data sets among the 1000 experimental failure data. Development of an efficient method is out of the scope of the present paper, although the approach would be more accurate than that using Monte Carlo generated data sets, since the data sets would be selected among an experimental one.

The above comments about m range are confirmed by the analysis of the data set for failures prior to maximum load, from which m as large as 11 was estimated (Table III). This subset is an extreme one for the size of 168 data since it comprises all the lowest strengths in the total distribution. It shows that $m \geqslant 11.2$ is the upper bound for this sample size. This value is much larger than the ones obtained on smaller sample sizes (Table III), and than those predicted by Davies in [31] for ceramics with comparable $m = 5$. These results demonstrate that the selection of samples is responsible for a wide scatter in statistical parameters.

Figure 5 compares the corresponding linearized Weibull plots to the normal distribution. It visualizes the above-mentioned discrepancy in m. It indicates that the selection of the small sets of 20 or 30 data was not too bad. But, this selection was obtained by chance. And, in the absence of reference distribution there was no means to evaluate the validity of this selection. In the present paper, the reference distribution was established, which highlights the importance of getting the normal distribution or the true statistical parameters. In [22] it is demonstrated that sets of at least 30 data are required to obtain normal distributions.

TABLE III. Weibull parameters estimated using subsets (20, 30, and failure data prior to maximum load) selected from failure data obtained on test specimen 2. Asterisk indicates number of events prior to maximum load.

	Weibull plot	
Population size	M	$\varepsilon_l(\%)$
20	4.75–7.18	1.20–1.31
30	4.55–6.05	1.23–1.24
168*	11.20	0.79

V. DISCUSSION

There are several objective reasons why it can be considered that the true flaw strengths parameters have been determined: $m = 5.2$ and $\varepsilon_l = 1.20\%$, $\varepsilon_0 = 0.01\%$, and $\sigma_0 = 22$ MPa.

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, and use of an empirical estimator.

However, the question may arise on the pertinence of the failure data that have been generated experimentally. Fibre interactions can influence the results, since they can cause either overestimation of the force on fibres (owing to the effect of a frictional force $F_{\mathrm{tot}} = F_{\mathrm{true}} + F_{\mathrm{fric}}$) or fracture of several fibres (leading to a steep force decrease beyond maximum). The effect of fibre interactions during the tests was investigated by comparing the experimental force-strain curve with that predicted using the bundle model considering that fibres are parallel and independent. The force-strain relation during a tensile test is given by [32]

$$F(\varepsilon) = N S_f E_f \varepsilon [1 - P(\varepsilon)], \qquad (9)$$

where $P(\varepsilon)$ is the probability of failure at strain ε; S_f is the average filament cross sectional area. Figure 6 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 [24]. All these results converge on the conclusion that there was not significant pollution by fiber friction.

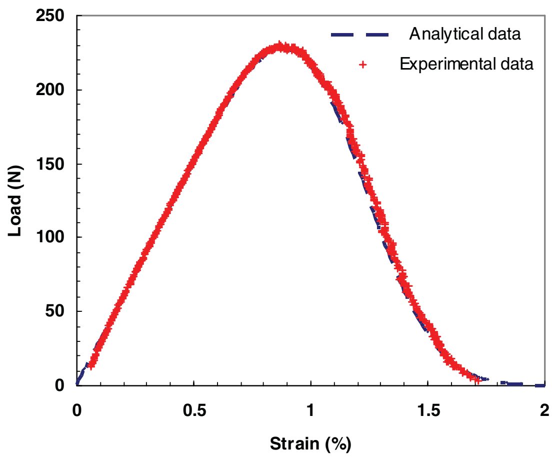

FIG. 6. (Color online) Comparison of experimental and predicted load-strain curves for a Nicalon fibre bundle (test specimen 2).

VI. CONCLUSIONS

One of the major results of the present paper is that flaw strengths in brittle fibres follow normal distribution. More generally, this result applies to brittle materials. Using the appropriate equation for flaw density function is of great interest for sound failure predictions using the elemental strength approach based equation of probability. Unlike with conventional Weibull analysis of data, with normal distribution there is no need for an estimator for the estimation of statistical parameters, which eliminates a source of variability.

Then, it was shown that power law (Weibull type) is a satisfactory approximation of normal distribution, which assesses the validity of power law for the flaw strength distribution. This leads to simpler equations of failure probability.

The values of statistical parameters that were derived from the comparison of Normal and Weibull distributions of 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, and the scale factors are $\varepsilon_0 = 0.01\%$ and $\sigma_0 = 22$ MPa. It should be noted that $m = 5.5$ and $\sigma_0 = 19$ MPa had been estimated a few years ago for this fibre in a previous work on single filaments [28]. Then, it was shown that variability in statistical parameters results from the construction of Weibull plots using an estimator, from sample size and from selection of test specimens (sampling). Sampling exerts a major influence when small data sets are considered. Variability cannot be corrected using various estimators or modified Weibull equations. Instead, statistically relevant database and normal distribution should be used for failure analysis.

It is important to note that the analysis used large sets of failure data (500 to 1000) determined experimentally. The data produced in the present paper can be regarded as reference data for further analysis on the commercial fibres 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.

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