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Strength variability and size effect of Nicalon fibre bundles

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Statistical strength and size effect of Nicalon fibre bundles are studied. The Weibull type of statistical theory underlying predictions of bounding Nicalon fibre bundle strength is presented and discussed. The relationship of bundle strength to single Nicalon filament strength and a model explaining the correlation are also discussed. The predicted values for Nicalon fibre bundles were in close agreement with the experimental data. Characterization of Nicalon fibres or bundles provides an insight into the ultimate mechanical performance of ceramic-matrix composites.

1. Introduction

It is well known that Nicalon fibre can be used as a reinforcement for ceramic, plastic and metal matrices to produce high-performance composite materials with optimum mechanical and electrical properties. Some articles about Nicalon reinforcement composites and fibre mechanical properties have been published by Brennan and Prewo [1], Dauchier *et al.* [2], Simon and Bunsell [3], and Wu and Netravali [4]. However, to date very limited mechanical property data for Nicalon fibres have appeared in the scientific literature. In order to better understand ceramic-matrix composites with Nicalon fibres, characterization of the mechanical properties of these fibres is mandatory.

The tensile failure of a bundle of brittle fibres in a flexible matrix is a complex process involving the failure of fibres at scattered flaw sites, the overloading of neighbouring fibres at these sites and the growth of sequences of adjacent fibre breaks to some critical size. It is known that the strength of a bundle of fibres is not accurately predicted by simple averaging over the strengths of the fibres in the bundle. In fact, in the case of equal load sharing developed by Daniel [5], it can easily be shown that averaging of fibre strengths yield an optimistic estimate of bundle performance. The analysis becomes even more complex under schemes in which the load is differentially shared among the surviving fibres. Details of the statistical modelling may be found in Phoenix and Smith [6], Pitt and Phoenix [7], and Hedgepeth and van Dyke [8]. Such is the case in tightly twisted bundles such as yarns, cables and ropes, or in continuous fibre-reinforced composites where the load of the failed fibre element is locally redistributed on to surviving neighbours.

This paper describes the strength variability and size effect for Nicalon fibre bundles. A predicted model of the fibre bundle has been presented. Experimental results for the strength of both dry and impregnated Nicalon fibre bundles are compared with those from the predicted model.

2. Experimental procedure

2.1. Single filament testing

Fibre bundles were made of ceramic grade Nicalon and supplied by Nippon Carbon Co. of Japan. Nicalon fibres were extracted from randomly selected bundles. Each bundle contained approximately 500 filaments. Sample preparation and test procedure used are given below.

In previous work, Wu and Netravali [4] performed a Weibull analysis of strength–length relationships for single Nicalon fibres. In their work, gauge lengths of 10, 50, 76.2 and 175 mm were investigated. They found that the logarithmic strength–length relationship of single Nicalon filaments follows the weakest-link rule [9] and both failure load and failure stress of the fibres fit well to a two-parameter Weibull distribution [10]. All fibres tested for a length effect underwent a Type III surface treatment as reported previously [4]: the M-sized fibre bundle was placed in a furnace for desizing using a heating rate of $204\,^{\circ}\mathrm{C\,h^{-1}}$ to $400\,^{\circ}\mathrm{C}$, kept in the furnace at $400\,^{\circ}\mathrm{C}$ for 6 h, then cooled to room temperature at a rate of $316\,^{\circ}\mathrm{C\,h^{-1}}$. Fibres were tabbed with light cardboard tabs following procedures described elsewhere [11, 12]. The adhesive used for tabbing was a quick-setting cyanoacrylate adhesive (910 Fs-Gold, Permabond International). The tension tests were performed in an Instron machine model 1122 at $21\,^{\circ}\mathrm{C}$ and 65% relative humidity. Crosshead motion was such that the ratio of crosshead speed to fibre length was $0.02\ \mathrm{min^{-1}}$.

Fibre diameter was measured with the use of an electromechanically driven vibroscope. Samples for diameter measurements and for strength measure-

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ments came from the same population of fibres. The fibre length used in the vibroscope was 60 mm. The mass density used to calculate the fibre diameter was 2.55 g cm^{-3} [13]. Additional details of the vibroscope and the measurement techniques are available in the literature [11, 12].

2.2. Fibre bundle testing

Fibre bundles tested fell into two length groups: 76.2 and 175 mm. Each group was divided into two categories: as received (i.e. ceramic grade with M sizing), and desized according to the Type III surface treatment. Half of the bundles were tested dry. The other half were impregnated with epoxy, cured, and then tested. M sizing on the bundles was removed following the Type III heat-cleaning procedure. After size removal (also referred to as "desizing" in this paper), about half of the bundles were impregnated with epoxy. The epoxy/hardener system used was Epon 828/mPDA (Shell Chemical Co.). The impregnation procedure consisted of drawing the bundle (under a tension of about 150 g) through the epoxy/hardener bath and then through a 0.020 in. (0.51 mm) diameter dome die (DD-112H) made by Waldron Die Co. The pulling rate was 5 mm s^{-1}. The impregnated bundles underwent curing for 2 h at 80 °C followed by 2 h at 150 °C.

Fibre bundle samples for tension tests were prepared following the steps outlined below. Three cardboard pieces were cut for each sample. Each piece was 20 mm wide and approximately 1.5 mm thick. One piece was long (with a length equal to the length of the sample); the other two were short (32 mm in length). The individual bundles (dry or impregnated) were placed on the long piece of cardboard and each end of the bundle was sandwiched between the long and a short piece. Omegabond 101 resin/catalyst made by Omega Engineering Inc. was used to bond each end of the bundles with the sandwiching cardboard pieces. The adhesive was cured overnight under pressure at room temperature. After the bundle and cardboard assembly was mounted in an Instron model TM testing machine, the long piece of card was cut and the sample was tested at 21 °C and 65% relative humidity. Crosshead motion was such that the ratio of the crosshead speed to the bundle length was 0.02 min^{-1}. "Bundle length" is defined as the free length of the underformed bundle between the two short end pieces.

3. Results and discussion

3.1. Size effect and single fibre strength

Based on weakest-link theory [9], it is expected that Nicalon fibre and bundle strengths will decrease as their gauge length increases. Results of the gauge length effect on fibre strength have been presented by Wu and Netravali [4] and are summarized in Table I. Data are plotted in Figs 1 and 2. The strength of single Nicalon filaments fits well to a two-parameter Weibull distribution with a size effect in fibre strength. The weakest-link theory along with upgraded versions are

TABLE I Effect of gauge length on strength and Weibull parameters for single Nicalon fibres at strain rate 0.02 min^{-1} (see Equation 1)

Gauge length (mm)	Stength (MPa) (c.v. %)	m	σ_0 (MPa)	No. of specimens
10	3184 (32.52)	3.36	3548	50
50	2145 (38.03)	2.87	2411	56
76.2	2182 (33.19)	3.20	2432	50
175	1535 (34.22)	3.19	1717	50

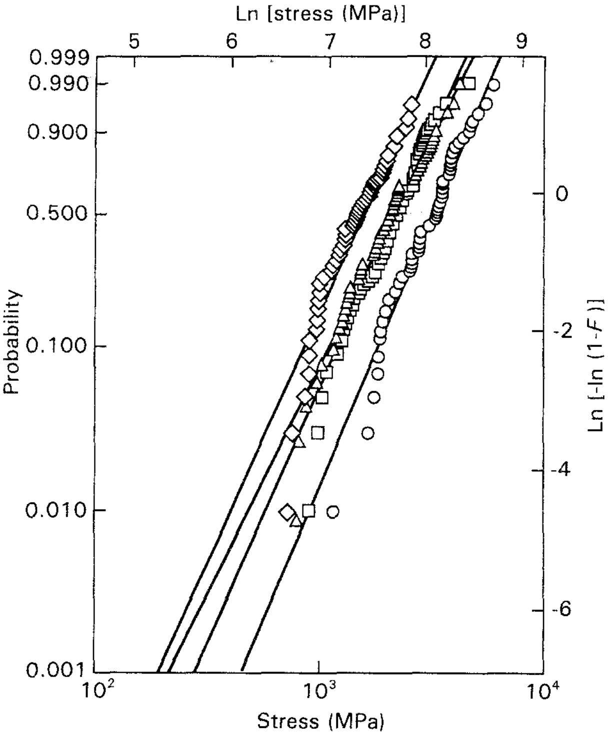

Figure 1 Type III heat-cleaned single Nicalon fibre strength at four gauge lengths, plotted in Weibull probability coordinates. (○) l = 10 mm, m = 3.36, σ_0 = 3548 MPa; (△) l = 50 mm, m = 2.87, σ_0 = 2411 MPa; (□) l = 76.2 mm, m = 3.20, σ_0 = 2432 MPa; (◇) l = 175 mm, m = 3.19, σ_0 = 1717 MPa; (——) MLE fits.

available in the literature [6–8, 14, 15]. It suffices to state that with the available single-fibre strength data a failure probability distribution of the form

$$F(\sigma) = 1 - \exp\left[-\left(\frac{l}{l_0}\right)\left(\frac{\sigma}{\sigma_0}\right)^m\right] \qquad (1)$$

is adequate to calculate fibre failure. Here $F(\sigma)$ = fibre failure probability under the stress σ, l = length of fibres under investigation and l_0 = length of the reference fibres which were tested to calculate the Weibull parameters m and σ_0.

Fibre strengths were plotted on Weibull probability paper. Using the method of maximum likelihood (MLE) [16, 17], the Weibull shape and scale parameters were estimated. A modified Newton–Raphson method was used to solve the MLE equations. In Equation 1 the size effect (l/l_0) is linearly proportional to fibre length l. It is expected [18–21] that due to a possible variation of fibre diameter, surface flaws, and

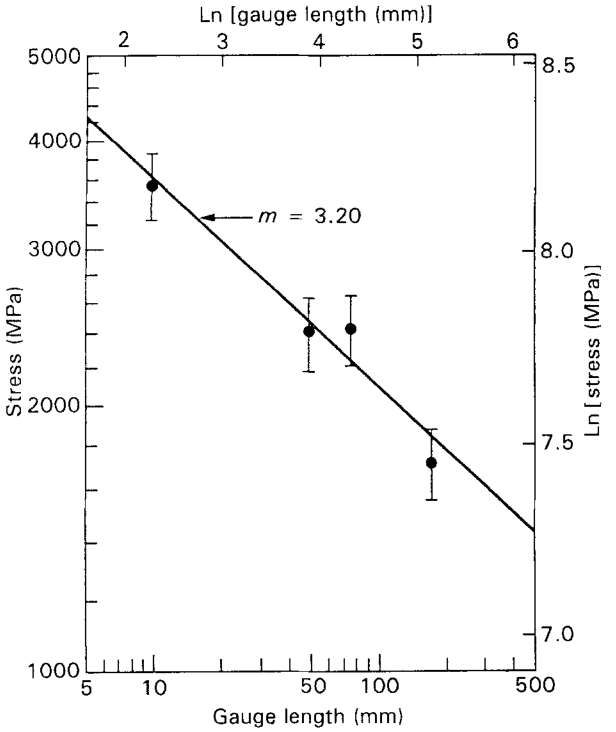

Figure 2 Weibull scale parameter for Type III heat-cleaned single Nicalon fibre strength against gauge length on log–log scale.

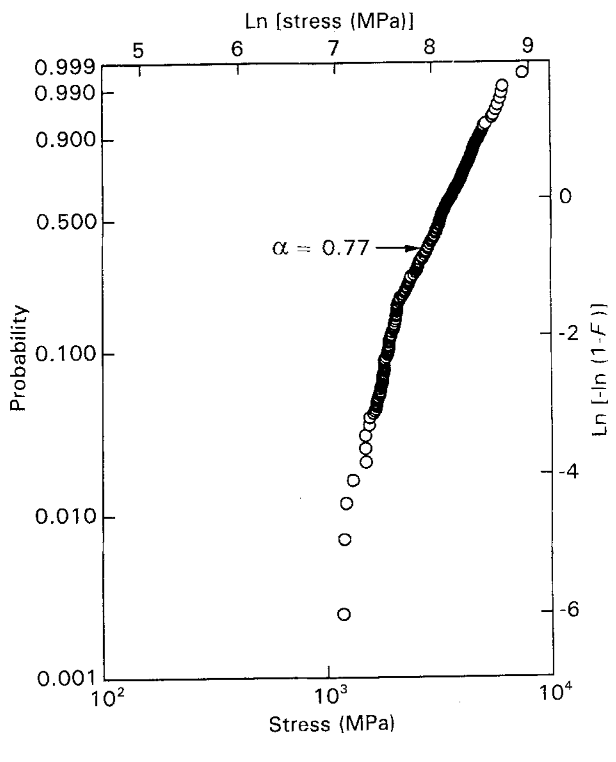

Figure 3 "Master" distribution of all the Type III heat-cleaned fibre strength distributions, scaled to a 10 mm gauge length.

volume flaws along the fibre length the size effect may not remain proportional to fibre length. Until sufficient data on the variation of fibre characteristics along their length are available, an update of Equation 1 to accommodate the non-linearity of the size effect with respect to l is not warranted. The applicability of Equation 1 with $l_0 = 10$ mm is verified in Fig. 3. Fig. 3 shows that the prediction and the measured strength values are within the scatter of the experimental data. Hence, use of Equation 1 is acceptable for the present set of experimental results.

3.2. Size effect and bundle strength

As expected, the bundle strength decreases as the gauge length increases. Results are listed in Table II and data are plotted in Figs 4 to 7. These figures contain test results as well as maximum likelihood estimates of the strength values. Like single fibres, the strength change can be predicted using Equation 1. With $l_0 = 76.2$ mm and the corresponding m and σ_0 from Table II, the predicted mean strengths of different bundles are as given below:

As-received and impregnated: 148 N

Desized and impregnated: 112 N

Desized and dry: 74 N

The corresponding measured values and the coefficients of variation (see Table II) show that the difference between the measurements and prediction is no more than one standard deviation. Thus the use of Equation 1 is acceptable.

3.3. Relationship of single-fibre strength and bundle strength

From Tables I and II one finds that the bundle strength is significantly different from the product of fibre strength and the number of the fibres in a bundle [5]. The difference is caused mainly because (a) in general, load–extension curves for fibres in a bundle vary over a wide range, and (b) the twist in a bundle affects load sharing by individual fibres [5, 6, 22–24]. These and other factors make prediction of bundle strength from single-fibre strength a difficult exercise. However, prediction of bundle strength bounds offers less difficulty.

Based on work done by Phani [22] and Chi *et al.* [24] a first-order model can be formulated that bounds the bundle strength once the Weibull parameters, m and σ_0, for the filaments are known (see Equation 1). The model assumes that (a) each fibre in a bundle has the same length l and cross-sectional area A which remains constant along the fibre length, and (b) at any stage of loading the stress σ in each filament is the same. Both assumptions can be relaxed. For example, distributions for the fibre cross-sectional area A and for σ can be introduced. These model updates have been ignored, primarily because (a) Table I suggests that a constant fibre area is an acceptable assumption for the present samples, and (b) no experimental data on the Nicalon bundles are available that support one σ distribution over others.

According to the above-mentioned assumptions and Equation 1, one can relate [22] the load supported by a bundle to the stress σ in the individual fibres by the equation

$$P = N_0 A \sigma \exp\left[-\left(\frac{l}{l_0}\right)\left(\frac{\sigma}{\sigma_0}\right)^m\right] \quad (2)$$

TABLE II Effect of gauge length on strength and Weibull parameters for dry and impregnated Nicalon fibre bundles (at strain rate 0.02 min^{-1} (see Equation 1)

Surface treatment	Dry or Impregnated	Bundle length (mm)	No. of specimens	Mean ultimate load (N) (c.v. %)	m	σ_0(N)
As-received[a]	Dry	76.2	8	78 (17.3)	7.0	84
	Impregnated	76.2	24	157 (8.4)	14.7	163
		175	19	142 (9.4)	12.2	148
Desized[b]	Dry	76.2	28	83 (13.4)	7.4	88
		175	34	65 (11.9)	9.4	69
	Impregnated	76.2	20	125 (14.7)	7.3	133
		175	22	121 (16.3)	7.3	129

[a] Ceramic grade Nicalon fibres with M-sizing.
[b] Via heat-cleaning Type III (for detailed description see section 2.1).

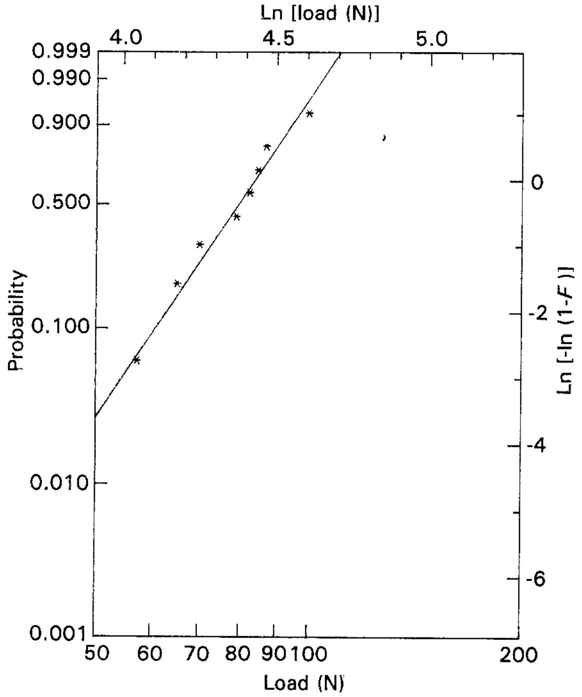

Figure 4 Weibull probability plot for failure load of as-received M-sized dry Nicalon fibre bundles at 76.2 mm gauge length.

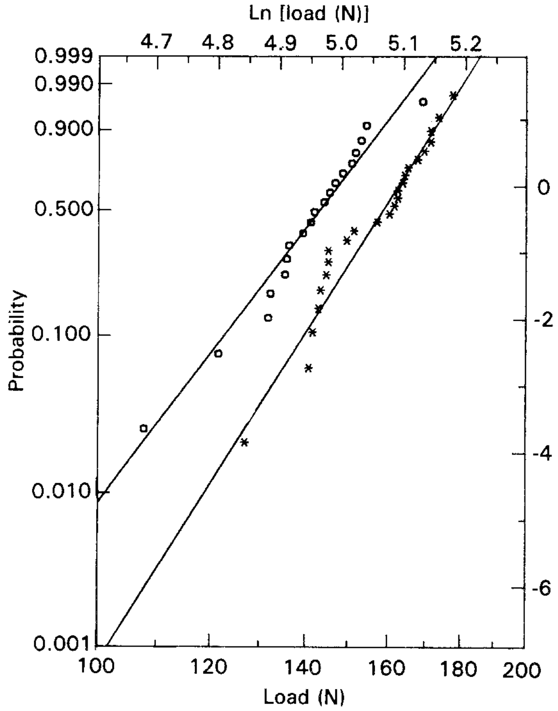

Figure 5 Weibull probability plots for failure load of as-received M-sized impregnated Nicalon fibre bundles at (∗) 76.2 and (○) 175 mm gauge length.

where N_0 = number of fibres in the bundle at the beginning of the test. Differentiating P with respect to σ one finds that the maximum load a bundle can carry is given by

$$P_{max} = N_0 A \sigma_0 \exp(-1/m) \quad (3)$$

Here

$$\sigma_m = \sigma_0 \left[m \left(\frac{l}{l_0} \right) \right]^{-1/m} \quad (4)$$

From the product data sheet [13], $N_0 = 500$. From Table I the average fibre diameter is 15 µm. Using these values together with the values of l_0 and m from Table II, Equations 3 and 4 predict the maximum strength of 76.2 and 175 mm bundles to be 109 and 77 N, respectively. The corresponding measured values are 114 and 81 N, respectively (see Table II). Thus, an acceptable agreement between prediction and measurement of the maximum bundle strength can be reached once m and σ_0 for the constituent fibres are known.

The above calculation assumes that at any stage of loading, the load is equally divided among all surviving fibres, i.e. stress σ in each fibre is the same. In reality, because of the twist in a bundle, it is expected that when one fibre fails, the load it was carrying is distributed among the neighbouring fibres [6, 23]. Thus, stress in neighbouring fibres becomes higher than stresses in fibres away from the fibre that failed. As a result, the neighbouring fibres have a higher probability of failure. Continuation of this process results in a bundle strength less than the P_{max} in Equation 3. Necessary information for the bundle strength calculation includes filament geometry, flaw distribution [25], pressure on the filament surface caused by the twist, and friction between filaments. The theory behind the calculation is available in the literature [26, 27].

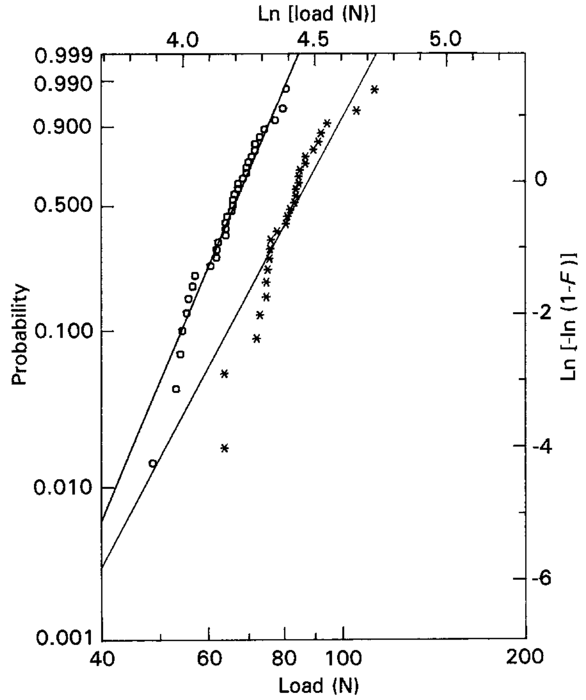

Figure 6 Weibull probability plots for failure load of desized dry Nicalon fibre bundles at (*) 76.2 and (○) 175 mm gauge length.

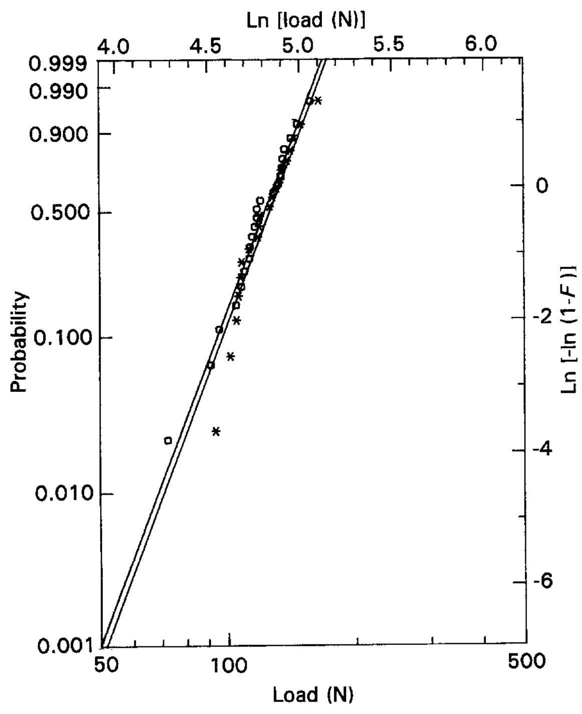

Figure 7 Weibull probability plots for failure load of desized impregnated Nicalon fibre bundles at (*) 76.2 and (○) 175 mm gauge length.

In the absence of information necessary to calculate the bundle strength, the work of Rolf and co-workers [26, 27] can be used to estimate a possible lower bound for the bundle strength. Steps necessary for the estimate are outlined below. Equation 1 can be rewritten as

$$F(\sigma) = 1 - \exp\left[-\left(\frac{S}{S_0}\right)\left(\frac{\sigma}{\sigma_0}\right)^m\right] \quad (5)$$

Here S and S_0 are the surface areas of individual fibres and the reference fibre, respectively, equal to πdl and πdl_0 here d is the fibre diameter. Thus, for a single filament the probability of failure is related to the fibre surface. When N_0 fibres in a bundle interact with each other, the failure probability of one depends on the number of fibres in the bundle and other factors mentioned in the previous paragraph. Therefore, when two identical fibres carry the same stress σ, but one of them is by itself and the other one is in a bundle, their failure probabilities will be different. The difference can be incorporated by changing the value of S from the single-filament surface area πdl to an effective surface area for a bundle. The effective surface will depend on N_0 and other factors mentioned in the previous paragraph.

According to weakest-link theories [9], the fibre interaction that produces higher probability of failure will be characterized by a larger effective surface area. A possible upper bound for the effective surface area for a bundle with N_0 fibres is obtained by assuming that the fibres are so closely interrelated that the bundle behaves like a cylindrical rod whose cross-sectional area is equal to the area of N_0 filaments. Then S, the surface area of the rod, is given by

$$S = \pi d l N_0^{1/2} \quad (6)$$

When S from Equation 6 is used in Equation 5, the P versus σ relationship (see Equation 2) will be different. Using Equations 6, 5 and 2 one can show that the new P-σ relationship is given by

$$\hat{P} = N_0 A \sigma \exp\left[-N_0^{1/2}\left(\frac{l}{l_0}\right)\left(\frac{\sigma}{\sigma_0}\right)^m\right] \quad (7)$$

Based on Equation 7, a possible lower bound of a bundle's load-carrying capacity is given by

$$P_{\text{low}} = N_0 A \hat{\sigma} \exp(-1/m) \quad (8)$$

where

$$\hat{\sigma} = \sigma_0\left(\frac{m l N_0^{1/2}}{l_0}\right)^{-1/m} \quad (9)$$

When $N_0 = 500$, fibre diameter = 15 μm, and l_0 and m are as given in Table I, one finds $P_{\text{low}} = 57$ and 40 N when the bundle lengths are 76.2 and 175 mm, respectively. The corresponding measured values are 64 and 49 N, respectively (see Table II). It should be emphasized that Equation 8 provides only a possible lower bound compatible with the assumptions for the bundle characteristics mentioned earlier.

4. Conclusions

The following can be concluded from the work presented in this paper:

1. The strength of single Nicalon fibres and bundles fits well to a two-parameter Weibull distribution.
2. Nicalon fibre bundles exhibit a size effect with a large strength variability (m is in between 3 and 15).
3. A theory behind bounding bundle strength predictions has been presented. Agreement with the experimental data is acceptable. The estimating process

requires fibre strength data as input and uses previous work cited in the literature [5, 21–26].

4. The model for predicting fibre strength can be updated. The updating parameters include the twist in bundle, a length of fibres in a bundle, friction between fibres, pressure exerted by one fibre on others, and fibre bending in a bundle.

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