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Weibull analysis of strength–length relationships in single Nicalon SiC fibres

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Nicalon SiC fibre is naturally brittle and offers high-temperature application in fibrous composites. Due to the randomly distributed flaws along the fibre, the statistical variability in single-fibre strength is obvious. In this paper, the effect of heat-cleaning procedures on Nicalon fibres has been investigated, and the statistical strength and variability of single Nicalon fibres have been characterized in tension and compared. Experimental results show that the strengths of single Nicalon fibres among the three types of heat-cleaning procedures are less than that of as-received unsized fibres by 22–30%. In addition, both the failure load and the failure stress of the fibres, for a given gauge length (50 mm) and yarn cross-section, can be well fitted to a two-parameter Weibull distribution. The effect of gauge length over the range from 10–175 mm, holding the strain rate constant, was also studied. The logarithmic strength–length plots show that the strength of single Nicalon fibres follows the weakest-link rule.

1. Introduction

Nicalon ceramic fibre is a silicon carbide ceramic fibre manufactured through a polymer pyrolysis process by Nippon Carbon Co. Ltd, Tokyo, Japan. The fibre can be used as a reinforcement for plastic, ceramic and metal matrices to produce high-performance composite materials with optimum mechanical and electrical properties. It is also good for high-temperature applications. Yajima and colleagues [1–3] initially demonstrated the properties of Nicalon fibre in 1976. In 1980, it was commercialized by Nippon.

Several articles about Nicalon reinforcement composites and fibre mechanical properties have been published by Brennan and Prewo [4], Dauchier *et al.* [5], and Simon and Bunsell [6]. However, to date very little mechanical property data for Nicalon fibres have appeared in the scientific literature. Also, the experimental results from several sources appear to be inconsistent. In order to fully understand ceramic matrix composites with Nicalon fibres, characterization of the mechanical properties of these fibres is required.

The tensile failure of fibrous composites is a complex statistical process involving scattered failure of fibres at flaw sites; overloading of neighbouring fibres at these sites by way of stress transfer through the matrix; and the growth of sequences of adjacent fibre breaks to some critical size. The "weakest link" maxim was first applied quantitatively to fibrous structures by Peirce [7] who used weakest-link statistics (assuming statistical independence of links) to predict the mean and variance of the failure strength of long yarns when the statistics of shorter lengths were known. Peirce considered a long yarn to be made up of a series of shorter-length yarns and, under the assumption that the strengths of the short yarns followed a Gaussian distribution, predicted the strength of the long yarns. His experimental results fit reasonably well with his theoretical predictions, given the assumptions with which he was working.

It has long been known that the strength of a bundle of fibres is not predicted accurately by simple averaging rules over the strengths of the fibres in the bundle. In fact, in the case of equal load sharing, developed by Daniel [8], it can easily be shown that averaging of fibre strengths yields 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 [9–11]. Such is the case in tightly twisted bundles (yarns, cables, and ropes) or in continuous fibre-reinforced composites where the load of a failed fibre element is locally redistributed onto surviving neighbours.

This paper describes the statistical characterization of the strength distribution resulting from three types of heat-cleaning procedures and the effect of changing the gauge length for single Nicalon fibres. The results show that the strength of Nicalon fibres can be well fitted to a two-parameter Weibull distribution [12]. Also, like most polymeric fibres, Nicalon fibre seems to exhibit gauge-length effects over the range from 10–175 mm, holding the strain rate constant. The results of dry and impregnated Nicalon fibre bundles and eight-harness satin Nicalon cloths will be reported in the future.

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2. Theoretical model

2.1. Failure in brittle materials

Brittle materials like glass or carbon fibres tend to fail catastrophically when the stress level reaches the strength of the weakest flaw. The most common assumption about the distribution of weakest flaws is that they are not only randomly scattered throughout the material, but are also of random severity. This assumption is reasonable because brittle materials often have large variations in strength, as well as significant strength reductions, as size or volume is increased. Size effects are encountered because as the volume of material is increased, the probability of encountering a severe flaw increases.

2.2. Statistics of single fibre strength

Single Nicalon fibre segments removed from the cross-section of a yarn or tow and tension tested at a gauge length, l_0 (say 50 mm), typically have a strength distribution which follows a classical Weibull distribution of the form

$$F(\sigma) = 1 - \exp[-(\sigma/\sigma_{l_0})^{\zeta}] \quad \sigma \geqslant 0 \qquad (1)$$

with shape parameter, ζ, between 3 and 4; and scale parameter, σ_{l_0}, between 2000 and 4000 MPa. To predict the strength at a different gauge length, l, the classical, weakest-link rule with statistical independence of fibre segments is usually invoked to yield the same Weibull distribution except that the scale parameter, σ_{l_0}, is modified to

$$\sigma_l = \sigma_{l_0}(l/l_0)^{-1/\zeta} \qquad (2)$$

This result predicts a rather severe size (length) effect for the fibre in that a tenfold increase in length yields roughly a 30–50% decrease in fibre strength. The underlying assumptions to the Weibull model above are that flaws occur along the fibre as a compound Poisson process in position.

For some fibre types, this scaling strength with length is somewhat severe; that is filaments with length l longer than l_0 are stronger than predicted by Equation 2, whereas those with shorter length are weaker.

Independently, Watson and Smith [13] and Gutans and Tamuzs [14] have suggested that the exponent $1/\zeta$ in Equation 2 be modified to α/ζ, that is

$$\sigma_l = \sigma_{l_0}(l/l_0)^{-\alpha/\zeta} \qquad (3)$$

where α is a parameter between 0 and 1. Thus the underlying Weibull distribution for length l becomes

$$F(\sigma) = 1 - \exp[-(l/l_0)^{\alpha}(\sigma/\sigma_{l_0})^{\zeta}] \quad \sigma \geqslant 0 \qquad (4)$$

Recently, Phoenix *et al.* [15] suggested that $\alpha = 0.60$ for Kevlar 49 fibres, and Watson and Smith [13] obtained $\alpha = 0.90$ for carbon fibres. In contrast, recent data for ultra high-strength polyethylene fibres yield α near zero [16].

Watson and Smith [13] offer an explanation for the above effect, which is based on random variation in the diameter of the fibres within a yarn cross-section. This yield increased variability in the strength of sampled fibres that is not revealed in changing the length. Of course the diameter itself is often found to vary along the fibre so that realistically we must view Equation 4 as a useful approximation over a limited length range about l_0.

3. Experimental procedure

3.1. Fibres and heat-cleaning procedures

The fibre used in this study was Nicalon ceramic grade silicon carbide fibre supplied by Nippon Carbon Co. Ltd. of Japan in tow form on a spool. Two types of Nicalon spool were used. One spool, from Lot No. 133, had M-sizing, and the other spool, from Lot No. 132, was unsized (X9-6102). The yarn or tow contained 500 individual filaments in these two spools.

Fibres heated with three different procedures were compared with unsized fibres for strength. The three heat-cleaning procedures were as follows. Type I: the M-sized tow was placed in the furnace for de-sizing using a heating rate of 5 °C min^{-1} to 600 °C, then kept in the furnance at 600 °C for 1 h and cooled down to room temperature. Type II: the M-sized tow was placed in the furnace for de-sizing using a heating rate of 5 °C min^{-1} to 400 °C, then kept in the furnace for 1.75 h at 400 °C and cooled down to room temperature. Type III: the M-sized tow was placed in the furnace for de-sizing using a heating rate of 204 °C h^{-1} to 400 °C, kept it in the furnace at 400 °C for 6 h, then using a cooling rate of 316 °C h^{-1}, was cooled down to room temperature. The unsized, as-received spool was designated as type IV. The heat-cleaning procedures of types I and II were suggested by the fibre supplier, Dow Corning Corporation, Midland, MI. A large number of specimens about 250 mm long were extracted from various places in the yarn to obtain a representative sample. To determine the linear density, and thus the cross-sectional area of each specimen, we removed about 60 mm from one end and placed it in an electromechanical driven vibroscope [17]. The vibroscope used the vibrating string principle to measure indirectly the mass per unit length of the fibre. The cross-sectional area was calculated assuming a mass density of 2.55 g cm^{-3} [18]. A key assumption was that the variation in mass per unit length along a fibre was small compared to such variations in filaments from across a yarn. This proved to be true as the former coefficient of variation was at most 4%, as compared to about 7% in the latter case; the equipment error of the vibroscope was less than 1%. These cross-sectional areas were used in the calculation of all filament stresses.

3.2. Tension testing of single filaments

To determine the statistical distributions of fibre strength for the model, at least 50 single fibres having a gauge length of 50 mm were taken from each of the three types of heat-cleaned fibre, and at least 50 unsized fibres having a gauge length of 50 mm were tested in tension. The effect of gauge length on the type III heat-cleaned Nicalon fibres was determined by tension-tests using gauge lengths of 10, 50, 76.2 and 175 mm, in sample sizes of about 50, to obtain the

TABLE I Statistics for filament diameter, failure load and failure stress of single Nicalon fibres (gauge length = 50 mm)

Type of heat cleaning	No. of specimen	Diameter (μm) (c.v., %)	Failure load (g) (c.v., %)	Failure stress (MPa) (c.v., %)
I	64	15.18	34.96	1924
		(9.61)	(36.20)	(37.34)
II	83	15.34	36.78	1981
		(11.24)	(38.21)	(39.75)
III	56	15.42	40.22	2145
		(10.43)	(35.91)	(38.03)
IV	106	14.20	44.07	2753
		(9.02)	(23.05)	(23.85)

statistical strength distribution. All the specimens were tabbed on light cardboard tabs according to accepted procedures [17] using a quick-setting cyanoacrylate adhesive (910 Fs-Gold, Permabond International). Tension tests were performed in an Instron model 1122 machine under standard textile conditions of 21 °C and 65% relative humidity, and at a strain rate (assumed ratio of cross-head speed to gauge length) of 0.02 min^{-1}. These conditions applied to all experiments.

4. Results and discussion

4.1. Statistics for filament strength

The statistics for single Nicalon filament diameter, failure load and failure stress of these three types of heat-cleaned fibres and the as-received unsized fibres are listed in Table I. The mean values for failure load were determined directly from the tensile test, and using the vibroscope measurement of linear density, the failure stress for each filament was calculated. The failure stresses of types I, II and III were found to be less than that of type IV (unsized fibres) by ~ 22–30%. The average filament diameter was 14–15 μm.

4.2. Weibull analysis of the strength of single Nicalon filaments

The failure loads and failure stresses were plotted on Weibull probability papers. Using the method of maximum likelihood (MLE) [19, 20], the Weibull shape and scale parameters for both failure loads and failure stresses were estimated. A modified Newton-Raphson method was used to solve the MLE equations. All the data for failure loads and failure stresses were best fitted to a two-parameter Weibull distribution. The shape and scale parameters are presented in Table II, and the MLE lines are shown in Figs 1–8. The shape parameters of fibre stress for the three heat-cleaned fibres were ~ 2.7–3.0, while for the unsized fibre it was ~ 4.3. This indicates that the strength of the unsized fibres is less variable than that of the three heat-cleaned fibres. Comparing the strength among these four types of fibre shows that the unsized fibres (type IV) are the strongest. This suggests that extra handling while removing sizing from original sized Nicalon fibre bundles may possibly change the fibre surface properties and also induce more damage.

4.3. Effect of gauge length of filament strength

Table III presents MLEs of the Weibull shape and

TABLE II MLE of the Weibull shape $\hat{\zeta}$ and scale $\hat{\sigma}_{l_0}$ parameters for filament failure load and failure stress of single Nicalon fibres (see Equation 1)

Type of heat cleaning	Failure load		Failure stress	
	$\hat{\zeta}$	$\hat{\sigma}_{l_0}$(g)	$\hat{\zeta}$	$\hat{\sigma}_{l_0}$(MPa)
I	3.03	39.19	2.96	2159
II	2.81	41.35	2.69	2231
III	3.12	45.07	2.87	2411
IV	4.52	48.09	4.30	3011

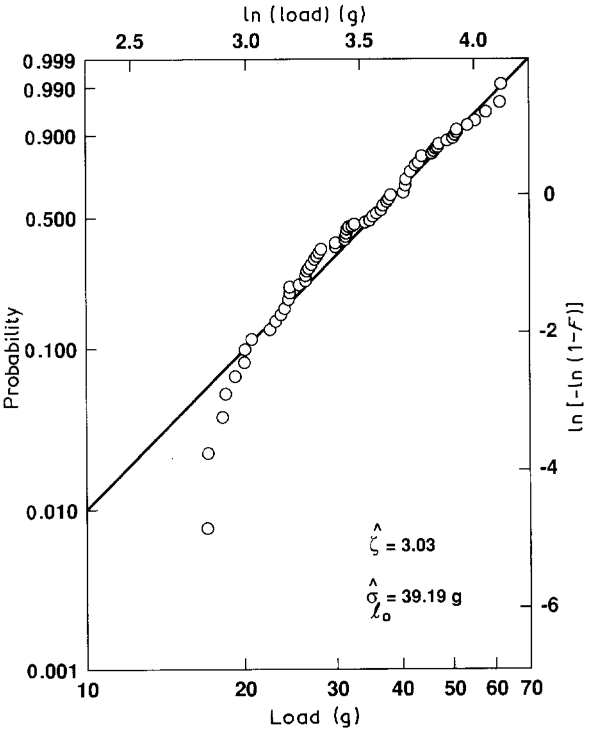

Figure 1 Weibull probability plot for the failure load of type I heat-cleaned single Nicalon fibres.

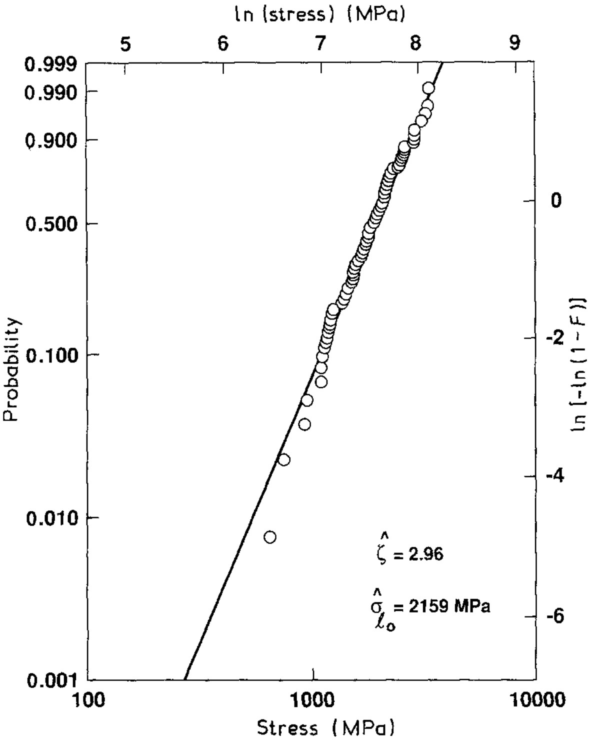

Figure 2 Weibull probability plot for the failure stress of type I heat-cleaned single Nicalon fibres.

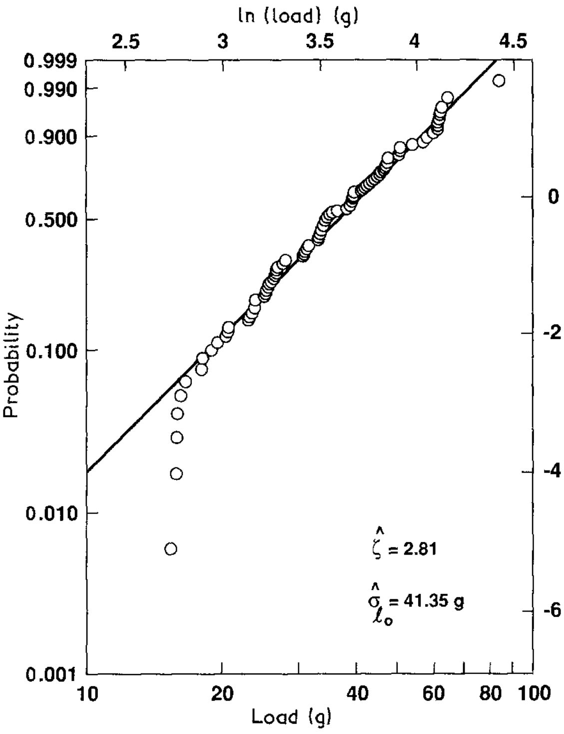

Figure 3 Weibull probability plot for the failure load of type II heat-cleaned single Nicalon fibres.

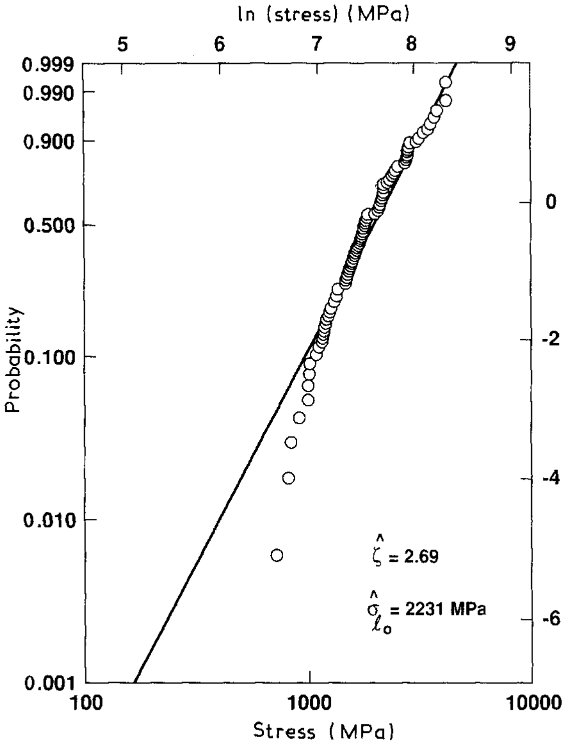

Figure 4 Weibull probability plot for the failure stress of type II heat-cleaned single Nicalon fibres.

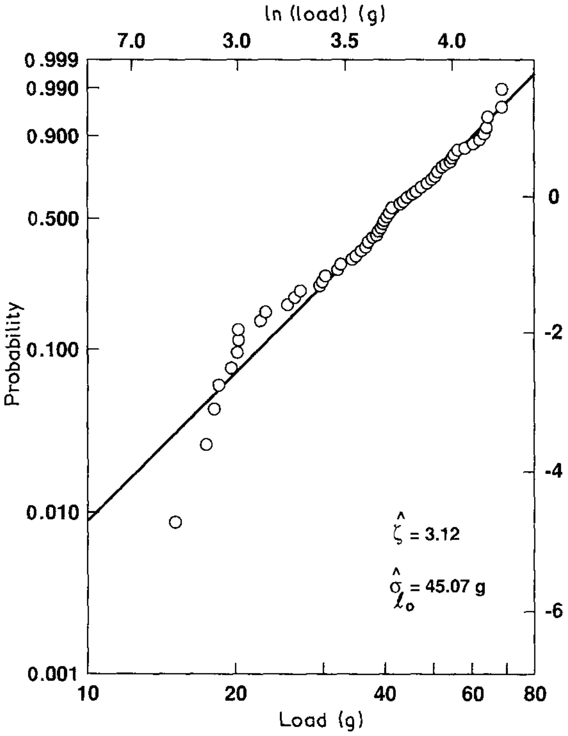

Figure 5 Weibull probability plot for the failure load of type III heat-cleaned single Nicalon fibres.

scale parameters for failure stress of the type III heat-cleaned Nicalon fibres for the various gauge lengths. Fig. 9 shows plots of the tension-test results for the single filaments on Weibull co-ordinates. For these specimens the mean and coefficient of variation (c.v.) of the filament cross-sectional area were 1.87×10^{-6} cm^2 and 17.8%, respectively. In a weakest link setting ($\alpha = 1$), a log–log plot of the scale parameter against gauge length should produce a straight line with a negative slope of one over the Weibull shape parameter, ζ. Fig. 10 shows such a plot together with 95% confidence intervals on the scale parameter estimates, and the least squares linear fit yields a shape parameter estimate of $\zeta = 3.20$. The statistical analysis obtained from the analysis of variance procedure shows an insignificant difference in strength between

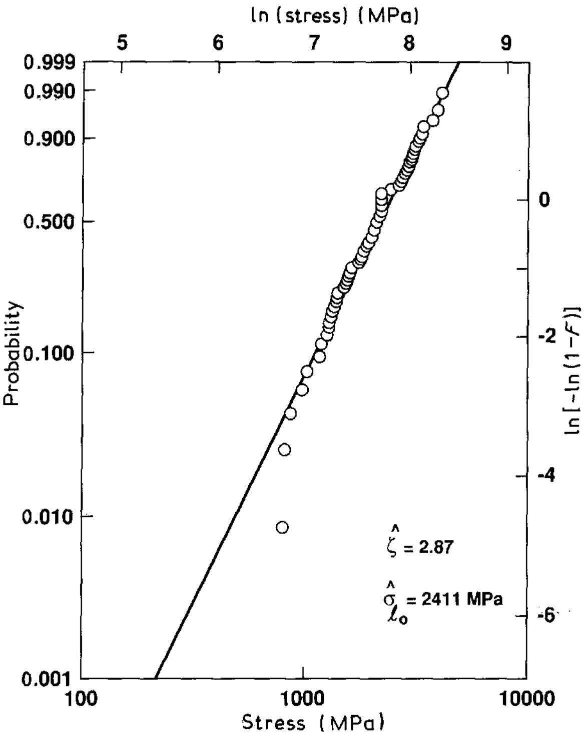

Figure 6 Weibull probability plot for the failure stress of type III heat-cleaned single Nicalon fibres.

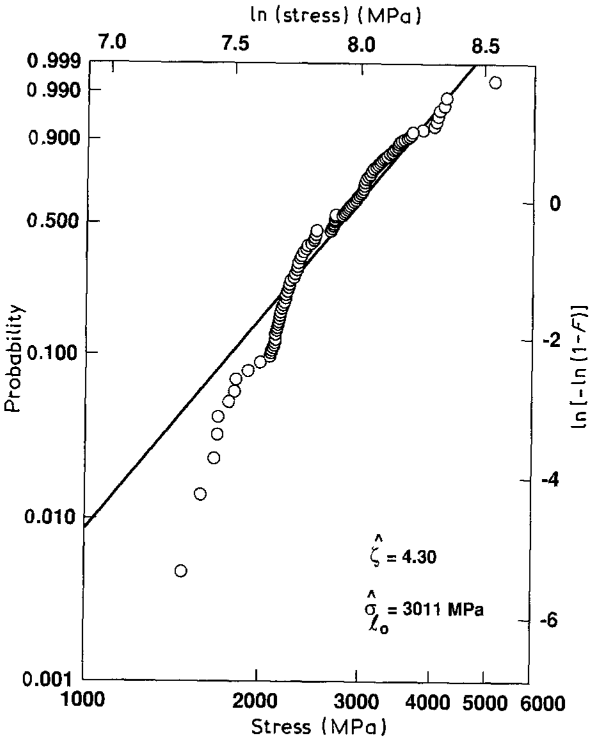

Figure 8 Weibull probability plot for the failure stress of type IV heat-cleaned single Nicalon fibres.

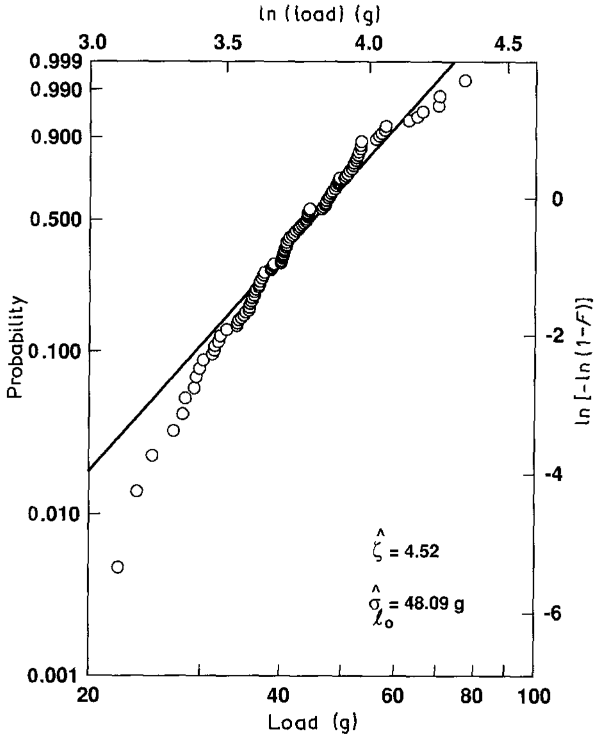

Figure 7 Weibull probability plot for the failure load of type IV heat-cleaned single Nicalon fibres.

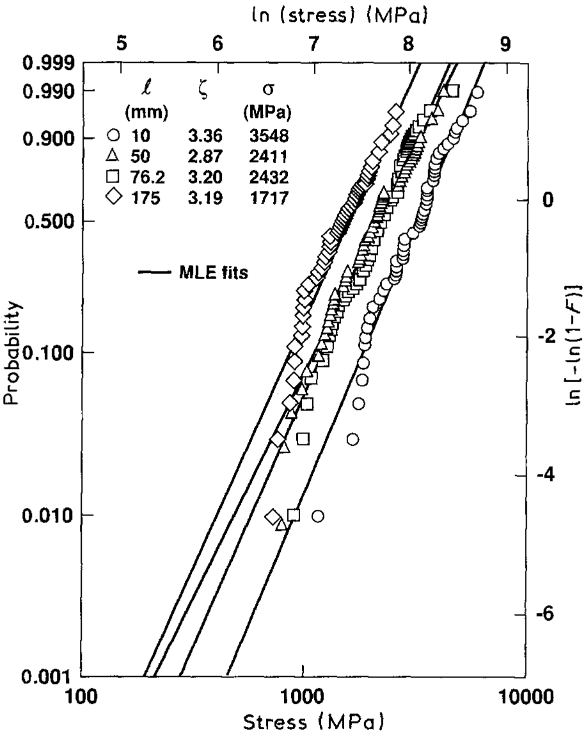

Figure 9 Type III heat-cleaned single Nicalon fibre strength at four gauge lengths on Weibull probability co-ordinates.

two gauge lengths of 50 and 76.2 mm (see Table IV). Also in this setting, if we were to compare the strength distributions $F_{l_1}(\sigma)$ and $F_{l_2}(\sigma)$ for two different gauge lengths l_1 and l_2 we should find that $F_{l_2}(\sigma) = 1 - [1 - F_{l_1}(\sigma)]^R$ where $R = l_2/l_1$. Using these data, we show in Fig. 11 a rescaling of all distributions to the shortest gauge length (10 mm) to yield a 'master' Weibull distribution with a shape parameter of 3.08 and a scale parameter of 3640 MPa. This curve, when compared with Fig. 9, is remarkably continuous, smooth and straight.

Taken together, these results suggest that the Weibull/weakest-link model with $\alpha = 1$ is quite adequate for representing the fibre strength over a wide range of lengths. There is perhaps an indication of slightly less than one ($\alpha = 0.77$) at the longest gauge

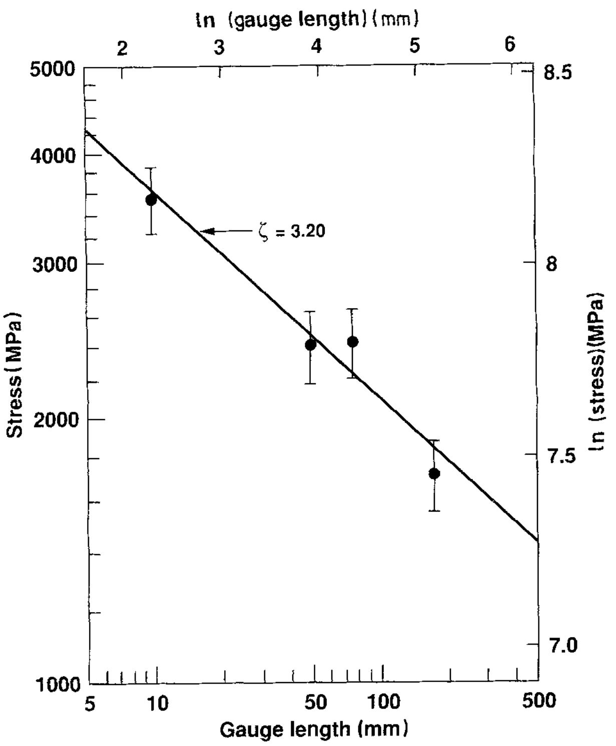

Figure 10 Weibull scale parameter for the type III heat-cleaned single Nicalon fibre strength against gauge length on log–log scale.

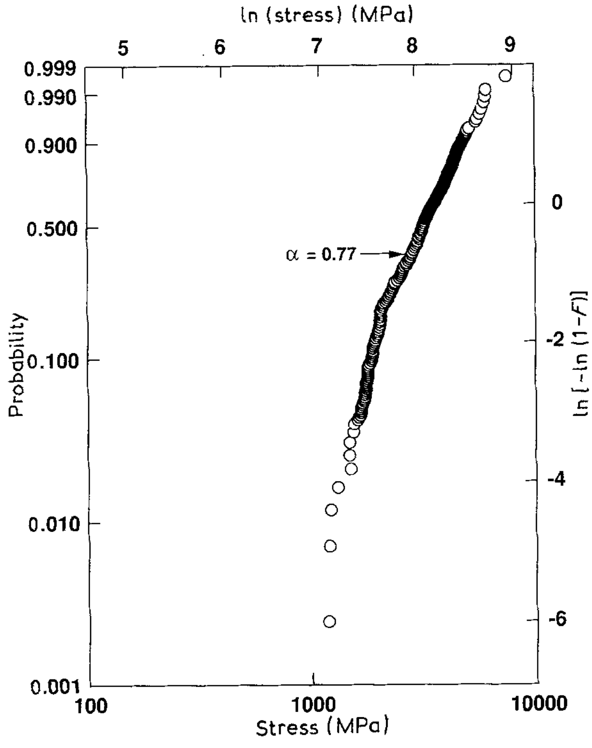

Figure 11 "Master" distribution of all the type III heat-cleaned fibre strength distributions scaled to a 10-mm gauge length.

length, and that the Weibull shape parameter, ζ, increases slightly with decreasing gauge length. The logarithmic strength–length plots show that the strength of single Nicalon fibres follows the classical weakest-link rule.

TABLE III Effect of gauge length on strength and Weibull parameters for type III of heat-cleaned single Nicalon fibres (strain rate = 0.02 min^{-1}) (see Equation 1)

Gauge length (mm)	Strength (MPa) (c.v., %)	$\hat{\zeta}$	$\hat{\sigma}_{l_0}$(MPa)
10	3184 (32.52)	3.36	3548
50	2145 (38.03)	2.87	2411
76.2	2182 (33.19)	3.20	2432
175	1535 (34.22)	3.19	1717

5. Conclusions

1. The strengths of single Nicalon fibres for the three types of heat-cleaning procedures are shown to be less than that of as-received unsized fibres by 22–30%. This suggests that the extra handling in removing M-sizing from the original sized bundle may change the fibre surface properties or cause some fibre damage.

2. The strength of single Nicalon fibres fits well to a two-parameter Weibull distribution. The scatter of the fibre strength can be represented by a Weibull shape parameter. It was found that the larger the shape parameter, the less variability in strength.

3. The strength–length relationship of single Nicalon fibres (type III heat-cleaning procedure) follows the weakest-link rule. This relationship can be used as an interpolation or extrapolation to the single fibre strength.

The present study lays a foundation for theoretical predictions of the strengths of dry and impregnated Nicalon fibre bundles and eight-harness satin Nicalon cloths, studies of which will be reported in the near future.

TABLE IV Results of analysis of variance for the type III heat-cleaned single Nicalon fibre strengths between two gauge lengths of 50 and 76.2 mm

Source of variation	Sum of squares	d.f.	Mean squares	*F* ratio	Significance level
Model	35323.54	1	35323.54	0.06	0.8086
Error	62285386.38	104	598897.95		
Total (Corrected)	62320709.92	105			

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