

Characterization of Nicalon fibres with varying diameters

Part II *Modified Weibull distribution*

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Diameters vary significantly in a tow of commercial Nicalon™ fibres, which is one of the most attractive ceramic reinforcements for structural composites. It was found that the strength distribution of Nicalon fibres could not be adequately characterized using either single- or bi-modal Weibull distribution. A recently proposed modified Weibull distribution can account for the effect of varying diameter in the characterization of fibre strength. To verify the validity of the modified Weibull distribution, comprehensive mechanical testing and fractographic studies have been conducted on Nicalon SiC fibres with diameters varying from 8 to 22 μm . The experimental results have been reported in Part I. Part II of this paper further modifies the derivation of the modified Weibull distribution to yield a relationship which is similar in form, but soundly based on experimental findings. Factors considered in the modified Weibull distribution include the dependence of fracture toughness and flaw density on fibre diameter, both of which may vary with fibre diameter, as reported in Part I. Comparison with experimental data shows that the current modified Weibull distribution works very well, while both single-modal and bi-modal Weibull distributions are inadequate for describing Nicalon fibres with varying diameters. © 1998 Chapman & Hall

1. Introduction

Nicalon silicon carbide fibre is increasingly being used as reinforcement for advanced composite materials [1–7]. Fibre strength is a major factor in determining the strength of fibre-reinforced composite materials. In order to predict the strength of a fibre-reinforced composite, it is essential to understand and accurately characterize the distribution of fibre strength. Nicalon fibres exhibit brittle fracture under tensile stress [1, 3], which is typical of advanced ceramic fibres. The strength of Nicalon fibre, similar to that of other ceramic fibres, is size dependent [8–11], and displays a range of experimental values for a given configuration.

A tow of commercial Nicalon fibres typically has a range of fibre diameters [12–17]. For example, the Nicalon (SiC) fibre spool (Dow Corning) used in this study contains fibres with individual diameter ranging at least from 8 to 22 μm . When Nicalon fibres with such a large diameter variation are used as reinforcements for a composite component, a practical question arises as to how to characterize the strength of the fibres so that the composite strength can be predicted

during the design stage. The Weibull distribution [18] is the first and most important function [19–20] that has been widely used to characterize brittle ceramic fibres for their strength distribution and dependence on gauge length [21–24]. However, it has been found inadequate for characterizing the diameter dependence of fibre strength, because factors such as fracture mechanics and material structure, which are not considered in the Weibull distribution, also affect the ceramic fibre strength [15, 20].

A bi-modal Weibull distribution can be used to characterize the strength of ceramic fibres when two distinct flaw populations are presumed to exist. However, as reported in Part I of this paper, as well as in a previous study [1], more than two types of flaws are found to exist in Nicalon fibres, suggesting that the bi-modal Weibull distribution may not be appropriate in this situation. In addition, the experimental results reported in Part I of this paper indicate that the apparent fracture toughness of Nicalon fibres increases with decreasing diameter. The bi-modal Weibull distribution cannot take into account the fracture toughness variation with varying diameter,

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and therefore may yield misleading results when used to describe the statistical strength of Nicalon fibres. It will be shown later that the bi-modal Weibull distribution is indeed inappropriate for characterizing the strength of Nicalon fibres.

The current ASTM standard [25] does not account for the effect of diameter variation on fibre strength. It only recommends using the average fibre diameter in calculating fibre strength, which may be reasonable if the fibre diameter does not vary much, but is certainly not appropriate for Nicalon fibres because of their large diameter variation. Watson *et al.* [26] and Andersson *et al.* [17, 27] realized the problem associated with varying fibre diameter in using the Weibull statistics, but did not try to solve it.

Batdorf [20] proposed that the fracture statistics should be based on a proper consideration of three elements: extreme value statistics (e.g. weakest link theory), fracture mechanics, and materials structure. Statistical theories incorporating these elements are as yet in their infancy. Batdorf [20] believes that such theories offer the greatest long-range promise for future progress. The Weibull theory is almost exclusively based on the weakest link theory, which explains its failure in characterizing Nicalon fibres with varying diameters. To overcome some of the shortcomings of the Weibull distribution, Wagner [28] assumed that there is a deterministic functional relationship between the Weibull scale parameter and the diameter of polymer fibres. He suggested that such a relationship could arise from the microstructure variation with varying fibre diameters. By relating the scale parameter of the Weibull distribution with the fibre diameter in such a way, Wagner effectively incorporated the fibre structure variation into the Weibull distribution. However, Wagner's theory does not account for the effect of fracture mechanics.

We [15, 29] have proposed a modified Weibull distribution to characterize the strength of ceramic fibres with varying diameters, which can account for all three factors proposed by Batdorf [20]. However, several assumptions made during the derivation of the modified Weibull distribution need to be verified experimentally.

To verify the validity of the modified Weibull distribution for the characterization of the strength of Nicalon SiC fibres, extensive mechanical testing and fractographic analysis have been conducted in this investigation. The experimental results have been reported in Part I of this paper [30]. Based on these findings, Part II of this paper will further validate and modify the derivation of the modified Weibull distribution. Comparison with experimental data will be made to show the validity of the modified Weibull distribution in characterizing the strength of Nicalon fibres with varying diameters, and also to show the limitation of the original Weibull distribution.

2. Modified Weibull distribution

Several different types of flaws, such as individual pores, pore clusters, granular defects, and surface flaws, have been found to cause failure in Nicalon

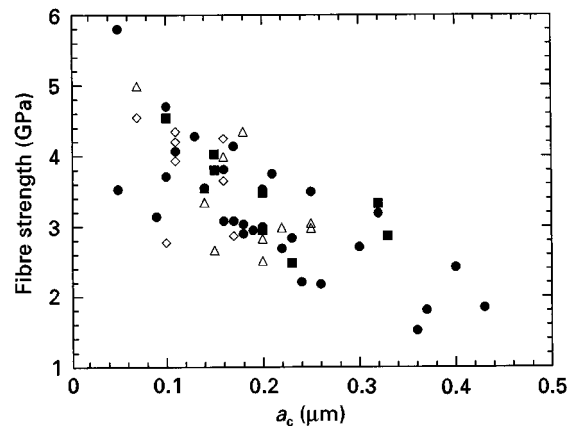


Figure 1 Effects of flaw size and flaw type on the strength of Nicalon fibres. The flaw size has much more effect on the strength of Nicalon fibres than flaw type. (●) Surface flaw; (■) pore cluster; (△) granular defect; (◇) individual pore.

fibres [30]. Different flaw types may have different ranges of size distribution. Fig. 1 shows the effect of both the flaw size and flaw type on the strength of Nicalon fibres. It can be seen that the flaw size has much more effect on the fibre strength than does flaw type. Therefore, only the general flaw size distribution will be considered in the following derivation. Let us define the expected number of critical size flaws under stress σ in the fibre volume under testing as P . For the simplest approximation, one may assume that failure occurs at which $P = 1$ [10]. If P is less than 1, it may be considered as the probability that a flaw will occur [20]. At a fixed fibre diameter, P will be proportional to the gauge length, if the fibre-processing parameters and subsequent handling conditions do not vary with fibre length. At a fixed fibre gauge length, P can be generally assumed to be proportional to d^e , where e is a constant and d is the fibre diameter. If the flaw density follows ideal Weibull statistics, e equals 2 for volume flaws, and 1 for surface flaws [16]. However, e could be any positive value if the fibre diameter affects the flaw density. Based on the above discussion, the expected number of critical size flaws, P , in a fibre of length L and diameter d can be expressed as

$$P = CLd^e \quad (1)$$

where C is a constant.

Derived from weakest link theory, Weibull statistics assumes that the probability of encountering a critical flaw, and hence the probability of fibre failure is proportional to fibre volume for fibres failing due to volume flaws [16, 20]. This yields a Weibull distribution in the form

$$F(\sigma) = 1 - \exp\left[-\left(\frac{\sigma}{\sigma_v}\right)^{\beta_v} V\right] \quad (2)$$

where $F(\sigma)$ is the probability of a fibre having a strength less than or equal to σ , σ_v and β_v are Weibull scale and shape parameters related to volume, and V is fibre volume. However, it is the expected number of critical size flaws, and not the fibre volume, that ultimately determines fibre failure [15, 29].

Therefore, a more general form of the Weibull distribution should use the probability of encountering a critical flaw P instead of the fibre volume. Thus, we can write the probability-based Weibull distribution as

$$F(\sigma) = 1 - \exp\left[-\left(\frac{\sigma}{\sigma_P}\right)^{\beta_P} P\right] \quad (3)$$

where σ_P and β_P are Weibull scale and shape parameters related to P . Substituting Equation 1 into Equation 3 yields

$$F(\sigma) = 1 - \exp\left[-C\left(\frac{\sigma}{\sigma_P}\right)^{\beta_P} Ld^e\right] \quad (4)$$

As pointed out in our previous work [15, 29], fracture mechanics and microstructure can also affect the fibre strength. Our experimental data in Part I of this paper indicate that the fracture toughness and mirror constant of Nicalon fibres increases with decreasing diameter (see Fig. 2), which could have been caused by the combination of both fracture mechanics and microstructure. Nicalon fibres with smaller diameters may have higher density and less flaws [30, 31] because of more effective pyrolysis, which will result in higher fracture toughness. From Fig. 2a, it is reasonable to assume that a power relationship exists between the mirror constant and the fibre diameter (Fig. 2), i.e.

$$A_m = \sigma_f r_m^{1/2} = Bd^{-u} \quad (5)$$

where A_m is the mirror constant, σ_f is the fracture strength of fibre, and r_m is the fracture mirror radius, and B and u are constant.

It has been shown in Part I [30] that r_m/r , the ratio of fracture mirror radius to fibre radius, does not change with varying fibre diameter, although the

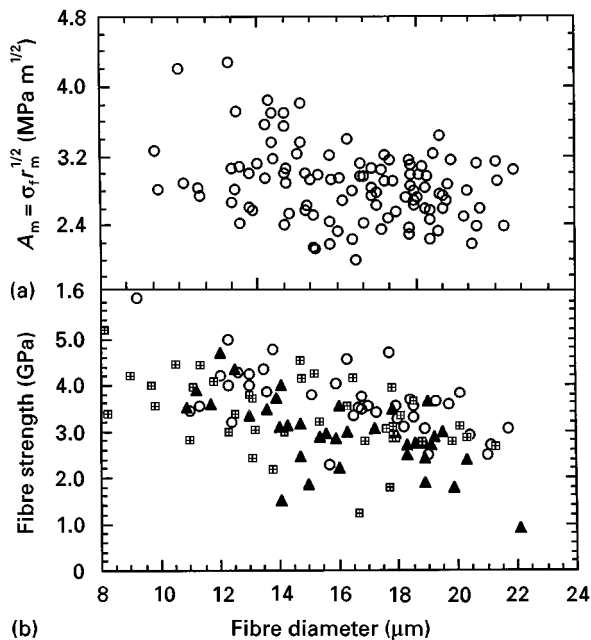


Figure 2 (a) Student's t -testing indicates with 99% confidence that mirror constants $A_m = \sigma_f r_m^{1/2}$ increases with decreasing fibre diameter. (b) Nicalon fibres tested with gauge lengths of 10 (○), 25 (■) and 50 mm (▲) in mineral oil show the increase of strength with decreasing diameter.

value of r_m/r exhibit scatter in the range of 0.04 to 0.28, that is

$$\frac{r_m}{r} = R \quad (6)$$

where R is a constant. Substituting Equation 6 into Equation 5, taking into account $r = d/2$, and rearranging yields

$$\sigma_f = Ed^{-b} \quad (7)$$

where E and b are constants and can be expressed as

$$E = (2/RB)^{1/2} \quad (8)$$

and

$$b = u + 1/2 \quad (9)$$

Of the two Weibull distribution parameters in Equation 4, σ_P is a measure of the average fibre strength and β_P is a measure of scatter in strength. Since σ_f in Equation 7 is non-statistical, it should only affect average fibre strength. Therefore, we can incorporate Equation 7 into Equation 4 by multiplying σ_P with another parameter σ_f

$$F(\sigma) = 1 - \exp\left[-C\left(\frac{\sigma}{\sigma_P \sigma_f}\right)^{\beta_P} Ld^e\right] \quad (10)$$

Substituting Equation 7 into Equation 10 yields

$$F(\sigma) = 1 - \exp\left[-\left(\frac{\sigma}{\sigma_0}\right)^{\beta_P} Ld^h\right] \quad (11)$$

where

$$\sigma_0 = C^{-1/\beta_P} E \sigma_P \quad (12)$$

and

$$h = e + b\beta_P \quad (13)$$

where h can be considered as the diameter dependency factor, which describes the effect of diameter on fibre strength.

The average fibre strength as a function of fibre diameter and length can be derived from Equation 11 as [32]

$$\bar{\sigma} = \sigma_0 \Gamma\left(1 + \frac{1}{\beta_P}\right) L^{-1/\beta_P} d^{-h/\beta_P} \quad (14)$$

where $\Gamma(1 + 1/\beta_P)$ is a gamma function. Equation 14 can also be written as

$$\bar{\sigma} = KL^{-m} d^{-n} \quad (15)$$

where the values of K , m and n can be obtained by fitting Equation 15 to experimental data, and the parameters for the modified Weibull distribution can be subsequently calculated as

$$\beta_P = \frac{1}{m} \quad (16)$$

$$h = \beta_P n \quad (17)$$

and

$$\sigma_0 = \frac{K}{\Gamma(1 + 1/\beta_P)} \quad (18)$$

3. Comparison with experimental data

3.1. Modified Weibull distribution

The following two equations can be derived from Equation 15

$$\ln \bar{\sigma} + m \ln L = \ln K - n \ln d \quad (19)$$

$$\ln \bar{\sigma} + n \ln d = \ln K - m \ln L \quad (20)$$

Assuming an initial value for m , Equation 19 can be fitted to experimental strength data for Nicalon fibres (Fig. 2b) to obtain a value of n . The n value can then be used to fit Equation 20 to experimental data to yield a new m value. The above calculation is iterated if the m or n value is different from the previous m or n value used in the calculation, until consistent m and n values are obtained. Fig. 3a and b show the final round of curve fittings for the strength data of Nicalon fibres (see Fig. 2b), which yield $m = 0.14909$, $n = 0.56433$, and $K = 23.8456$. The modified Weibull distribution parameters can be calculated using Equations 16–18 as $\beta_P = 6.707$, $h = 3.875$, and $\sigma_0 = 25.696$.

The Weibull parameters σ_0 and β_P can also be directly obtained by fitting Equation 11 to experimental data. Equation 11 can be rearranged as

$$\ln \ln \frac{1}{1 - F(\sigma)} - \ln L d^h = -\beta_P \ln \sigma_0 + \beta_P \ln \sigma \quad (21)$$

where $F(\sigma)$ is calculated from experimental data as [33]

$$F(\sigma_i) = \frac{i - 0.5}{N} \quad (22)$$

where N is the total number of tests, and the observed fibre strength values, $\sigma_1 \dots \sigma_N$, are ranked in ascending order. Fig. 4 shows the fit of Equation 21 with experimental data using $h = 3.875$, which yields $\beta_P = 6.713$ and $\sigma_0 = 25.967$. Since both Equations 15 and 21 are derived from Equation 11, and Equations 19 and 20 are derived from Equation 15, the β_P and σ_0 values obtained from the two methods discussed above should be consistent if Equation 11 is valid for characterizing the strength of Nicalon fibres with varying diameters. It is obvious that very consistent β_P and σ_0 values have been obtained, which validate the present modified Weibull distribution. Note that

h represents the effect of diameter on fibre strength. For the experimental strength data reported in this paper, the h value of 3.875, obtained by fitting of Equations 19 and 20 to the data, is a unique value that makes Equation 21 and Equations 19 and 20 yield consistent β_P and σ_0 values. Any other h values will yield inconsistent β_P and σ_0 values.

3.2. Single-modal Weibull distribution

The average fibre strength as a function of fibre diameter can also be obtained from the single-modal Weibull distribution (Equation 2)

$$\bar{\sigma} = \sigma_V \Gamma \left(1 + \frac{1}{\beta} \right) V^{-1/\beta} = K_V V^{-1/\beta} \quad (23)$$

Equation 23 can be rewritten as

$$\ln \bar{\sigma} = \ln K_V - \frac{1}{\beta} \ln V \quad (24)$$

Fitting Equation 24 to the experimental strength data for Nicalon fibres (see Fig. 5) yields $K_V = 15.986$ and $\beta = 5.209$, from which we also obtain $\sigma_V = 17.370$.

Rearranging Equation 2, we can obtain another way to calculate the Weibull parameters

$$\ln \ln \frac{1}{1 - F(\sigma)} - \ln V = -\beta \ln \sigma_V + \beta \ln \sigma \quad (25)$$

Fitting Equation 25 to the experimental data for Nicalon fibres (see Fig. 6) yields $\beta = 6.022$ and $\sigma_V = 14.144$. It is obvious that the Weibull parameters obtained from Equations 24 and 25 are not consistent, although both equations are derived from Equation 2. This suggests that the single modal Weibull distribution is inadequate for characterizing the strength of Nicalon fibres with varying diameters.

The fibre volume can be calculated as

$$V = \frac{\pi}{4} d^2 L \quad (26)$$

Substituting Equation 26 into Equation 2 yields

$$F(\sigma) = 1 - \exp \left[-\frac{\pi}{4} \left(\frac{\sigma}{\sigma_V} \right)^{\beta_V} L d^2 \right] \quad (27)$$

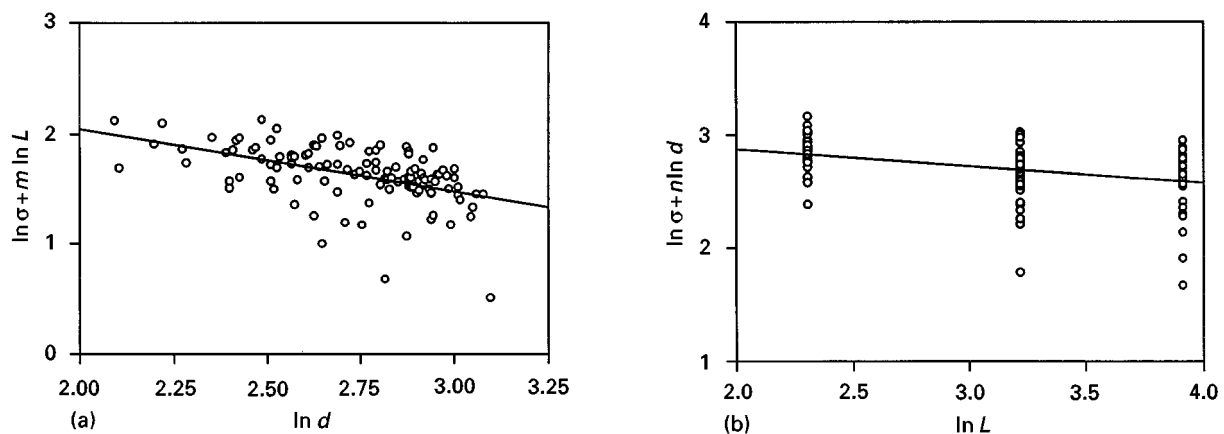


Figure 3 The final fitting of Equations 19 and 20 to the experimental strength data for Nicalon fibres. (a) Fitting to Equation 19 yields an n value of 0.56433, while (b) fitting to Equation 20 yields an m value of 0.14909. Both fits yield a K value of 23.8456.

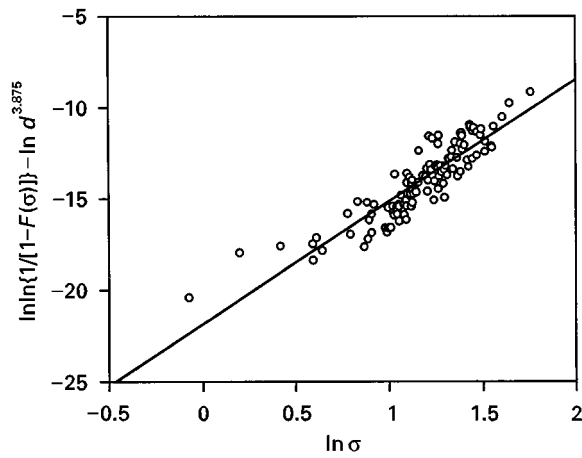


Figure 4 Fit of Equation 21 to experimental strength data for Nicalon fibres using $h = 3.875$ yields $\beta_p = 6.713$ and $\sigma_0 = 25.967$, which are consistent with the Weibull parameters obtained from Equations 19 and 20.

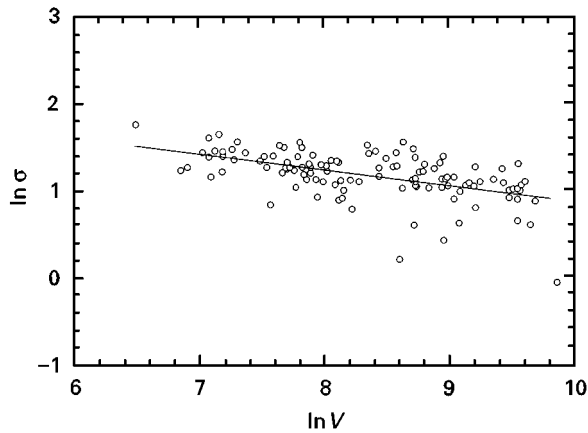


Figure 5 Fit of Equation 24 (derived from the single-modal Weibull distribution) to the experimental fibre strength data yields $K = 15.986$ and $\beta = 5.209$, from which we also obtain $\sigma_v = 17.370$.

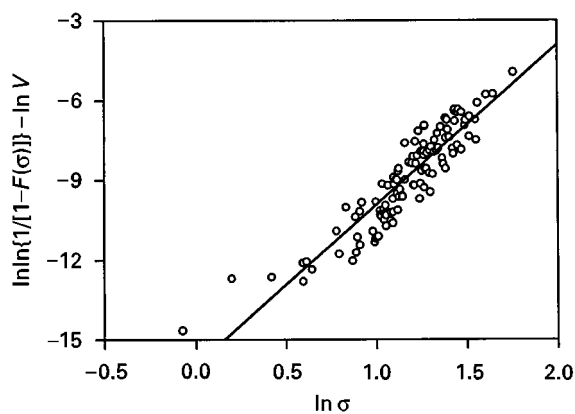


Figure 6 Fit of Equation 25 (derived from the single modal Weibull distribution) to the strength data for Nicalon fibres yields $\beta = 6.022$ and $\sigma_v = 14.144$.

Comparing Equation 27 with Equation 11, it is obvious that the volume flaw-based single modal Weibull distribution can be considered as a special case of the modified Weibull distribution in that they are equivalent to each other when $h = 2$. The deviation of the

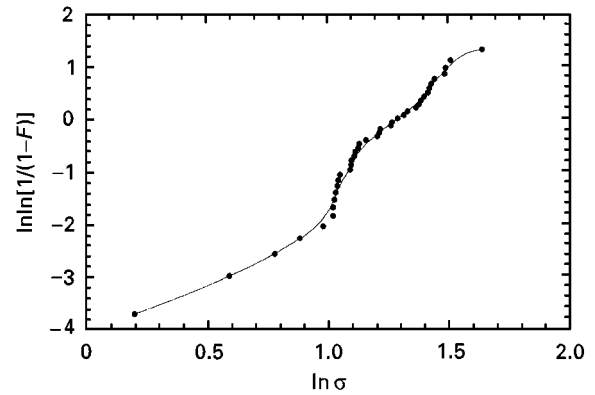


Figure 7 Plot of $\ln\{\ln[1/(1-F)]\}$ versus $\ln\sigma$ for fibres with a gauge length of 25 mm. The curve shows more than one knee, indicating that a bi-modal Weibull distribution is not adequate for characterizing the Nicalon fibres with varying diameters.

h value from 3.875 in the single modal Weibull distribution has caused the inconsistent β_p and σ_0 values.

3.3. Bi-modal Weibull distribution

Part I of this paper has identified four types of flaws in Nicalon fibres: pore clusters, granular defects, individual pores and surface flaws [30]. Theoretically, the bi-modal Weibull distribution can only characterize the strength of ceramic fibres with two distinct flaw populations. However, it can still be approximately applied to the Nicalon fibres if the collective flaw populations found in the fibres act like two distinct flaw types. Unfortunately, this is not the case as seen in Fig. 7, which shows a plot of $\ln\{\ln[1/(1-F)]\}$ versus $\ln\sigma$ for fibres with gauge length of 25 mm. Johnson [33] has indicated that the curve should consist of two straight lines with two different slopes and a single knee with positive curvature near the intersection of the two straight lines. Clearly, this behaviour is not observed in the curve in Fig. 7, which suggests that a tri-modal (or higher) Weibull distribution is needed. The bi-modal or multi-modal Weibull distribution could be misleading or erroneous if used to characterize the strength of Nicalon fibres with varying diameters, because it cannot take into account factors such as the effect of fracture mechanics and the possible flaw density variation with fibre diameter. Therefore, it is inappropriate to use the bi-modal Weibull distribution to characterize the strength of Nicalon fibres with varying diameters.

It is obvious from the above discussions that the present modified Weibull distribution can satisfactorily characterize the strength of Nicalon fibres with varying diameters, while neither a single-modal nor a bi-modal Weibull distribution is appropriate for the task. Nevertheless, the single-modal Weibull distribution is often used to characterize the strength of Nicalon fibres because of its simplicity, despite the aforementioned complication associated with varying diameter [17,26,27]. One of the errors caused by using the single modal Weibull distribution is the low calculated β value (large scatter in strength). The increases in fracture toughness with decreasing fibre diameter, as found in this study, will make the strength

of Nicalon fibres increase faster with decreasing fibre diameter than the case of constant K_{Ic} . The single-modal Weibull distribution cannot account for the effects of fracture toughness variation and possible flaw density variation with fibre diameters. Instead, it treats these effects as scatter of fibre strength, which will erroneously result in larger strength scatter and consequently smaller β value. The β value for Nicalon fibres obtained using the single-modal Weibull distribution has been reported to be in the range of 4 to 6 [16]. Using the single modal Weibull distribution, we have obtained the β value as 5.2 from Equation 24, and 6.0 from Equation 25, both of which fall into the reported range. However, the present modified Weibull distribution yields a β value of 6.7, higher than the reported range. This is because the present theory can account for the fracture toughness variation and possible flaw density variation with fibre diameter, further demonstrating the usefulness of the present modified Weibull distribution.

4. Conclusions

Based on experimental findings for the strength and fracture of Nicalon fibres, a modified Weibull distribution has been derived for Nicalon fibres with varying diameters. The modified Weibull distribution can take into account the fracture toughness variation and possible flaw density variation with varying fibre diameters, and shows excellent consistency in characterizing the strength of Nicalon fibres. The single-modal Weibull distribution is found to be inadequate for describing the strength of Nicalon fibres with varying diameters, and is found to yield an erroneously low Weibull modulus β . The bi-modal Weibull distribution is also found inadequate for characterizing the strength of Nicalon fibres. The present modified Weibull distribution is mathematically simpler than a multi-modal Weibull distribution, and more accurate than the single-model Weibull distribution. The applicability of the modified Weibull distribution to other ceramic fibres with varying diameters needs to be further investigated.

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