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Title: ## ON THE STATISTICAL STRENGTH OF NICALON FIBERS AND ITS CHARACTERIZATION

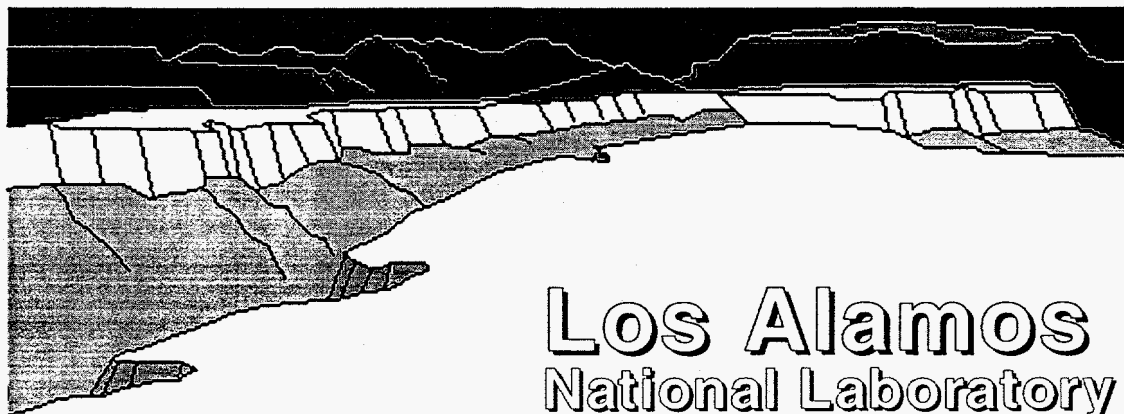RECEIVED
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ON THE STATISTICAL STRENGTH OF NICALON FIBERS AND ITS CHARACTERIZATION

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ABSTRACT

Nicalon fibers are one of the most attractive ceramic fibers for reinforcing high temperature structural composites. Experimental results show that the diameter variation (from 8 to 22 μm) in a tow of commercial Nicalon fibers has an effect on the statistical strength distribution of Nicalon fibers. Therefore, an appropriate characterization of the statistical distribution of fiber strength, capable of accounting for the effect of diameter variation, is required. The two-parameter single-modal Weibull distribution is found inadequate for characterizing the strength of Nicalon fibers. In this work, we demonstrate that a three-parameter modified-Weibull distribution can successfully characterize the strength of Nicalon fibers. The three-parameter modified-Weibull distribution yields a higher β value, which indicates less scatter in fiber strength than would be found using the two parameter single-modal Weibull distribution. It more accurately treats the strength variation caused by diameter variation. In addition, it is also much easier to use than the four-parameter bi-modal Weibull distribution. Moreover, it is seen to more accurately treat the strength variation caused by diameter variation than the single modal two parameter analysis.

INTRODUCTION

Nicalon[TM] silicon carbide fiber has attracted great interest in recent years due to its potential as a reinforcement for advanced composite materials. The strength of reinforcing fibers is one of the most prominent mechanical properties that governs the strength and toughness of composites, and its characterization is essential for predicting the mechanical properties of composites during composite design. The strength of Nicalon fibers is size dependent and scattered, which is typical for ceramic fibers. In addition, a tow of commercial Nicalon fibers

typically exhibits a range of fiber diameters. For example, the Nicalon (SiC) fiber spool used in this study contains fibers with diameters ranging from 8 to 22 μm.

The large diameter variation of Nicalon fibers complicates the characterization of fiber strength. Although the strength and fracture behavior of Nicalon fibers have been studied extensively in recent years [1-6], no thorough study has been done to investigate the effect of fiber diameter variation on the characterization of their statistical strength. Traditionally, the single-modal Weibull distribution has been used to characterize brittle ceramic fibers for their strength distribution and dependence on gauge length. However, the Weibull distribution cannot account for the effect of fiber diameter on fiber strength. It cannot differentiate the strength variation caused by the fiber diameter change from that caused by the real strength scatter. As a result, the Weibull distribution has been found invalid for characterizing the strength of fibers with varying diameters [7].

In this work, extensive mechanical testing has been conducted on Nicalon fibers, and a three-parameter modified Weibull distribution proposed previously by us [7, 8] has been successfully used to characterize the strength of Nicalon fibers. It will be shown that the modified Weibull distribution is much more accurate in characterizing fiber strength than the single-modal two parameter Weibull distribution, and is also much easier to use than the bi-modal four parameter Weibull distribution.

EXPERIMENTAL PROCEDURE

Individual Nicalon™ fibers (Dow Corning) were glued at the ends onto stiff paper mounts. Mounted fibers were secured into an alignment jig which was then attached to the grips of a fiber testing machine (MicroPull Science, Thousand Oaks, CA). Fibers were tested at three different gauge lengths (10, 25 and 50 mm), with data sets for each gauge length consisting of about 50 individual fiber tests. All of these tests were conducted in mineral oil to permit consistent recovery of the fractured fiber ends for fractographic analysis and diameter measurement on a JEOL 6300FVX Scanning Electron
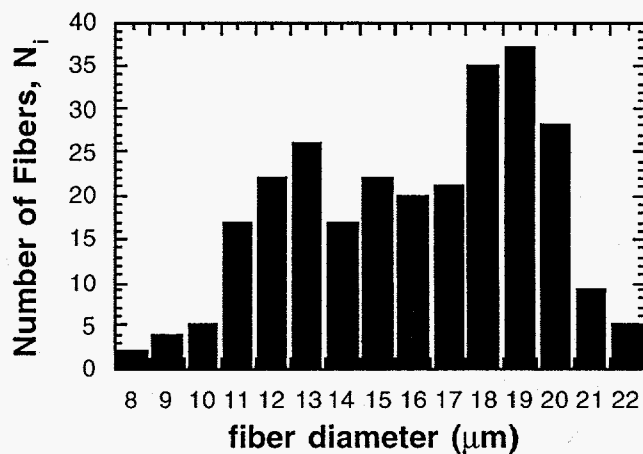

Fig. 1 Distribution of fiber diameters.

Microscope (SEM). Fibers breaking outside of the gauge length, or at the extreme ends of the fiber, were discarded. Peak load was used to calculate fiber strength.

EXPERIMENTAL RESULTS

Figure 1 shows the distribution of fiber diameters for all samples tested. Individual fiber diameters vary significantly within the tow, ranging from approximately 8 to 22 μm. From this distribution, the average fiber diameter is calculated as 16.0 μm. Several different flaw populations, such as individual pores, pore clusters, granular defects, and surface flaws, have been found to cause failure of the Nicalon fibers (see reference 9 for more information). Figure 2 shows the measured fiber strength vs. fiber diameter for all three gauge lengths (10, 25, and 50 mm). It can be seen that fiber strength increases with decreasing fiber diameter.
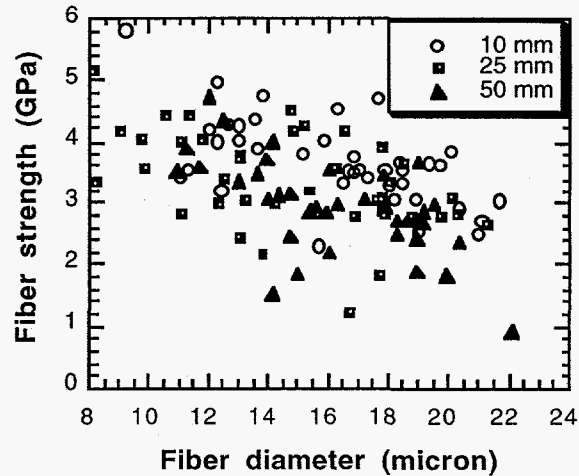
Fig. 2 The measured fiber strength value plotted against fiber diameter for all three gauge lengths (10, 25, and 50 mm).

CHARACTERIZATION OF THE STRENGTH OF NICALON FIBERS

Strength Characterization Using Weibull Distribution

Weibull statistics assumes that the probability of encountering a critical flaw, and hence the probability of fiber failure, is proportional to fiber volume for fibers failing due to volume flaws [10]. This yields the Weibull distribution in the form:

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

where $F(\sigma)$ is the probability of a fiber having a strength less than or equal to σ. Note σ_v and β_v are Weibull scale and shape parameters related to volume, and V is fiber volume.

The average fiber strength as a function of fiber diameter can be obtained from Eq. 1 as:

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

(2)

Equation 2 can be rewritten as

$$\ln \bar{\sigma} = \ln K_V - \frac{1}{\beta_V} \ln V \qquad (3)$$

Fig. 3 Fitting Eq. 3 (single modal Weibull distribution) to the experimental fiber strength data.

Fitting Eq. 3 to the experimental strength data for Nicalon fibers (see Fig. 3) yields K_v = 15.986 and β_v = 5.209, from which we also obtain σ_v = 17.370.

Rearranging Eq. 1, we can obtain another way to calculate the Weibull parameters:

$$\ln \ln \frac{1}{1 - F(\sigma)} - \ln V = -\beta_V \ln \sigma_V + \beta_V \ln \sigma \qquad (4)$$

Equation 4 is the most common form used to calculate Weibull parameters. $F(\sigma)$ can be calculated from experimental data as [10]

$$F(\sigma_i) = \frac{i}{N+1} \qquad (5)$$

where N is the total number of tests, and the observed fiber strength values, $\sigma_1 \ldots \sigma_N$, are ranked in an ascending order. Plotting the experimental data of $\ln \ln\{1/[1-F(\sigma_i)]\}$ - $\ln V_i$ against $\ln \sigma_i$ for Nicalon fibers and fitting Eq. 4 to the plot (see Fig. 4) yields β_v = 6.022 and σ_v
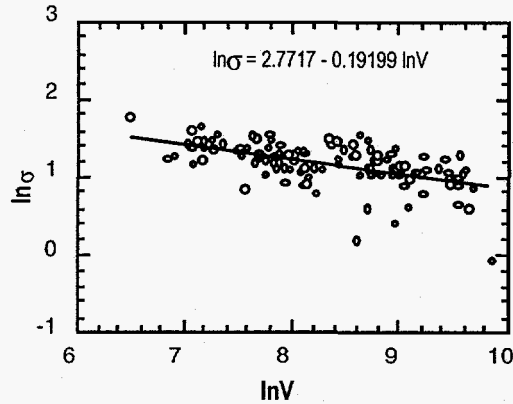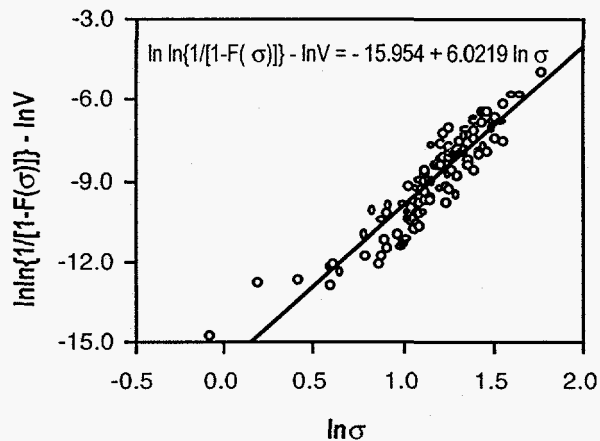

Fig. 4 Fitting Eq. 4 (derived from the single modal Weibull distribution) to the strength data.

= 14.144. It is obvious that the Weibull parameters obtained from Eq. 3 and Eq. 4 are not consistent, although both equations are derived from Eq. 1. This suggests that the single modal Weibull distribution is inadequate for characterizing the strength of Nicalon fibers with varying diameters.

Strength Characterization Using Modified Weibull Distribution

We have proposed a three-parameter modified Weibull distribution [1,8] in the form:

$$F(\sigma) = 1 - \exp\left[-\left(\frac{\sigma}{\sigma_0}\right)^{\beta_p} Ld^h\right] \tag{6}$$

The average fiber strength as a function of fiber diameter and length can be derived from Eq. 6 as [11]:

$$\bar{\sigma} = \sigma_0 \Gamma\left(1 + \frac{1}{\beta_P}\right) L^{-1/\beta_P} d^{-h/\beta_P} \tag{7}$$

where $\Gamma(1+1/\beta_p)$ is a gamma function. Eq. 7 can also be written as

$$\bar{\sigma} = KL^{-m}d^{-n} \tag{8}$$

where the values of K, m and n can be obtained by fitting Eq. 8 to experimental data, and the parameters for the modified Weibull distribution can be subsequently calculated as

$$\beta_P = 1/m \tag{9}$$

$$h = \beta_P n \tag{10}$$

and

$$\sigma_0 = \frac{K}{\Gamma(1 + 1/\beta_P)} \tag{11}$$

The following two equations can be derived from Eq. 8

$$\ln\bar{\sigma} + n\ln d = \ln K - m\ln L \tag{12}$$

$$\ln\bar{\sigma} + m\ln L = \ln K - n\ln d \tag{13}$$

Assuming an initial value for n, Eq. 12 can be fitted to experimental strength data for Nicalon fibers to obtain a value of m. The m value can then be used to fit Eq. 13 to experimental data to yield a new n value. The above calculation is iterated if the m or n value is different from the previous m or n value, until consistent values are obtained. Table II lists the m and n values during an iteration. The initial n value was assumed to be 0.7. Figures 5a and 5b show the final round of curve fittings, which yielded $m = 0.14909$, $n = 0.56433$, and $K = 23.8456$. The modified Weibull distribution parameters can be calculated using Eqs. 9-11 as $\beta_p = 6.707$, $h = 3.875$, and $\sigma_0 = 25.696$. Note that the above calculation was done using basic spreadsheet software. The calculation is much simpler than that necessary for the bi-modal Weibull distribution, which requires more advanced software or computer programs.
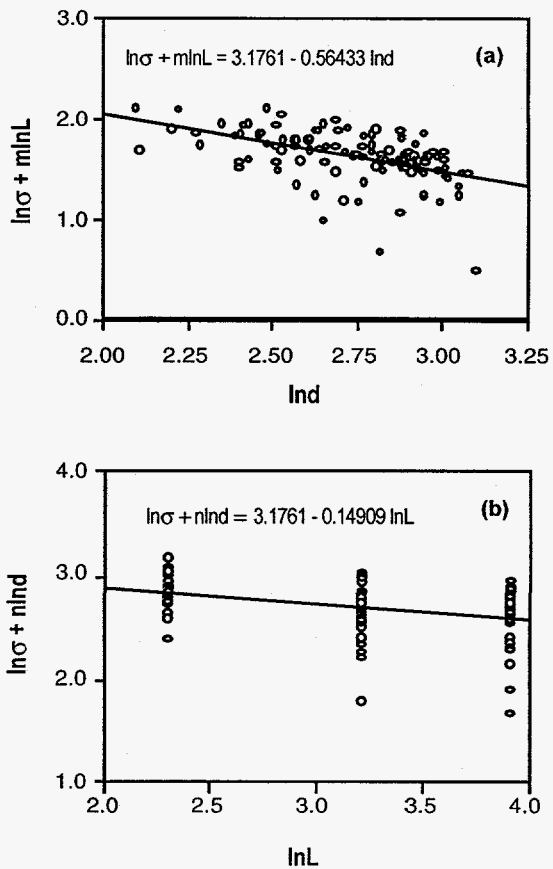
Fig. 5 a) Fitting to Eq. 12 yields an n value of 0.56433, while b) fitting to Eq. 13 yields an m value of 0.14909. Both fits yield a K value of 23.8456.

Table II The m and n values during the iteration

n as obtained using Eq. 13 $\ln \overline{\sigma} + m \ln L = \ln K - n \ln d$	m as obtained using Eq. 12 $\ln \overline{\sigma} + n \ln d = \ln K - m \ln L$
$n_0 = 0.7$	$m_1 = 0.14809$
$n_1 = 0.56439$	$m_2 = 0.14909$
$n_2 = 0.56433$	$m_3 = 0.14909$
$n_3 = 0.56433$	

We can also derive another way to obtain the Weibull parameters σ_0 and β_p by rearranging Eq. 6 as

$$\ln \ln \frac{1}{1-F(\sigma)} - \ln L d^h = -\beta_p \ln \sigma_0 + \beta_p \ln \sigma \qquad (14)$$

Fitting Eq. 14 to the experimental data (Fig. 6) using $h = 3.875$ yields $\beta_p = 6.713$ and $\sigma_0 = 25.967$. It is obvious that the β_p and σ_0 values obtained from Eq. 14 are very consistent with that obtained from Eq. 12 and 13. Equations 12, 13 and 14 are all derived from Eq. 6. Therefore, they should yield consistent β_p and σ_0 values if Eq. 6 is valid. The fact that very consistent β_p and σ_0 values have been obtained validates the three-parameter modified Weibull distribution.
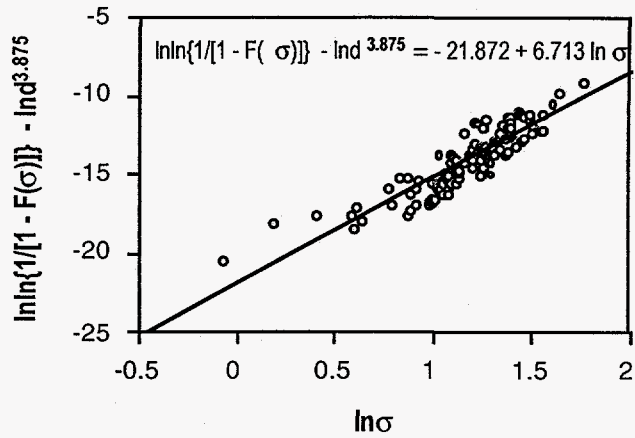

Fig. 6 Fitting Eq. 14 to experimental strength data for Nicalon fibers.

It is clear that the three-parameter modified-Weibull distribution can satisfactorily characterize the strength of Nicalon fibers, while the single-modal Weibull distribution is inappropriate for the task due to the large variation in Nicalon fiber diameter. In practice, the single-modal Weibull distribution is often used to characterize the strength of Nicalon fibers due to its simplicity, with most users unaware of the aforementioned complication associated with varying diameter. In addition to the inconsistency of β values obtained from the single-modal Weibull distribution, another error caused by using it is the low calculated β value (large strength scatter). The single modal Weibull distribution cannot take into account the effects of diameter variation on fiber strength. Instead, it treats these effects as scatter of fiber strength, which will erroneously result in larger strength scatter and consequently a smaller β value. For example, we have obtained a β of 5.21 using Eq. 3 and 6.02 using Eq. 4 (both equations are derived from the single-modal Weibull distribution). The β value for Nicalon fibers has been reported to be in the range of 4 to 6, which were obtained using the single-modal Weibull distribution [12]. These values are below the β value obtained using the three-parameter modified-Weibull distribution, which is 6.71, because the modified Weibull distribution can, in fact, account for the effects of diameter variation on fiber strength.

CONCLUSIONS

The three-parameter modified Weibull distribution has shown excellent consistency in characterizing the strength of Nicalon fibers with varying diameters. The single-modal Weibull distribution is found to be inadequate for characterizing the strength of Nicalon fibers with varying diameters, and is found to yield an erroneously low Weibull modulus, β. The present modified Weibull distribution is also mathematically much simpler than a multi-modal Weibull distribution.

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