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The Effect of Processing on Strength of Nicalon Fibers in Nicalon Fiber-SiC Matrix Composites*

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THE EFFECT OF PROCESSING ON STRENGTH OF NICALON FIBERS IN NICALON FIBER–SiC MATRIX COMPOSITES*

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Degradation of strength of Nicalon** (silicon carbide) fibers during processing of Nicalon fiber-SiC matrix composites was studied. Strength distribution of as-fabricated Nicalon fibers was obtained via bundle tests. Whereas, strengths of fractured fibers in Nicalon fiber-reinforced SiC matrix composite specimens were estimated by measuring fracture mirror radii. Comparison of fracture probability plots indicate significant differences in the behavior of the as-fabricated fibers and those in the composite. Possible causes leading to these differences are discussed.

Introduction

Continuous fiber-reinforced ceramic matrix composites have become an important

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** Manufactured by Nippon Carbon Co., Tokyo, Japan.

class of materials for structural applications because of their improved flaw tolerance, large work of fracture and noncatastrophic mode of failure[1-3]. Fracture behavior of fiber-reinforced ceramic composites is strongly influenced by the mechanical properties of reinforcing fibers and matrix, fiber/matrix interfacial characteristics, and residual stresses arising from thermal expansion mismatch of fibers and matrix. High strength of fibers and weak fiber/matrix interfaces along with tensile radial residual stresses are requisites for a 'tough' ceramic composite[4]. Strength of reinforcing fibers is critical because once a matrix crack initiates and extends there is a load transfer from matrix to fibers in the wake of the crack. Weak fibers fracture, leading to a catastrophic failure of the composite, whereas, strong fibers accomodate the stresses. Thouless et al.[5] have shown by theoretical analysis and experimental observations that the amount of fiber pullout, which contributes to the toughening of the composite, is strongly influenced by the mean strength and as well by the variability in strength of the reinforcing fibers. Also, the ultimate load bearing capacity of the composite is determined by the fiber strength characteristics[6]. It is, therefore, clear that strength of fibers is an important parameter for the design and development of fiber-reinforced ceramic matrix composites with superior mechanical properties.

Recently, research and development of thermally stable and oxidation resistant continuous silicon carbide (SiC) fibers, such as commercially manufactured Nicalon, has led to the fabrication of composites for elevated temperature applications. Nicalon fibers are currently being used as reinforcements in such matrices as silicon carbide, silicon nitride and glasses[7-9]. Over the past few years there has been an extensive mechanical and microstructural characterization of ceramic fibers[10-12] over a range of temperatures and enviornments. Clark et al.[12] have shown that SiC fibers are susceptible to thermal degradation. Moreover, it is possible to introduce surface defects or damage during handling of fibers during composite fabrication, and hence, further contribute towards strength degradation. Therefore, it becomes important to recognize and establish factors leading to strength degradation during fabrication processes and subsequently account for it in the prediction of the composite mechanical properties.

Results of an investigation into the effect of processing on the strength distribution of Nicalon fibers are reported in this paper. Single fiber strength distribution of as-received Nicalon fibers were obtained from bundle tests. Strength distribution of fractured fibers in a Nicalon fiber-reinforced SiC matrix composite were assessed from measurements of fracture mirror radii. Two-parameter Weibull distribution function was found to adequately describe the strength distributions for the two cases. Scale parameter for the as-received fibers was found to be larger compared to the fibers incorporated in the composite. However, there is no significant difference in the Weibull moduli for the two cases. Strength degradation of Nicalon fibers in composites is believed to be due to thermal degradation and mechanical damage to the fibers during fabrication.

Strength Distribution Function

For brittle materials, such as most ceramics, strength distribution is commonly described by the two-parameter Weibull distribution function which is expressed as follows[13]:

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

In equation 1, $F(\sigma)$ is the cumulative failure probability at an applied stress σ, σ_0 is the scale parameter signifying a characteristic strength of the distribution, and m is referred to as the Weibull modulus that characterizes the flaw distribution in the material. It should be noted that the above representation is based on the assumption that there is no interaction among the flaws in the material and failure of ceramics follows the weakest–link principle, i.e., failure occurs at the most severe flaw. Therefore, based on weakest–link principle, there is a size dependence of fracture strengths of ceramics since the severity of the critical flaw increases as the size of the specimen under stress increases. However, Weibull modulus does not change with specimen size provided the flaw population remains the same. Such experimental observations for brittle ceramic materials are well documented in literature[13,14].

In the present study, since strength distribution of fibers has been investigated on various fiber gage-lengths it is appropriate to account for the size effect and represent Weibull distribution function at a standard gagelength. Therefore, equation 1 was modified and represented as follows[10]:

$$F(\sigma) = 1 - \exp\left[-\frac{L}{L_o}\left(\frac{\sigma}{\sigma_o}\right)^m\right] \qquad (2)$$

where L is the gagelength of fiber stressed, L_o is the standard gagelength, and rest of the notations are as described above. Thus, by using the Weibull distribution function as given by equation 2, it is possible to compare Weibull scale parameters at equivalent fiber gagelengths.

Experimental Procedure

Material

Carbon-coated ceramic grade Nicalon fibers were used to study the effect of processing on the fiber strength distribution. SiC fibers were chosen for this study because of their high temperature stability and successful incorporations as reinforcements in composites for commercial applications[7-9]. Tows of Nicalon fibers were used to determine single fiber strength distribution via bundle tests. Nicalon fiber tows of various lengths were carefully extracted from Nicalon fiber mats*** . Typically, each tow consisted of 500 individual fibers . Nominal diameter of the fibers ranged from 10-15 μm. Polymer-derived Nicalon fibers consist

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*** Distributed by Dow Corning Corp., Midland, MI.

primarily of β-SiC crystallites of an average size of 1.7 nm along with excess carbon and SiO[15,16]. Reported[17] density and elastic modulus of Nicalon fibers are 2.55 g/c.c. and 210 GPa, respectively.

Nicalon fiber-reinforced silicon carbide (SiC) matrix composites were used for the evaluation of strength distribution of fibers in the composites. Composites were fabricated at Oak Ridge National Laboratory by densifying multiple layers of Nicalon mats stacked in graphite die. Chemical vapor infiltration (CVI) technique, under forced conditions of thermal and pressure gradients, was used for densification of the preforms with SiC. Resulting composites were close to 90% dense. Details of specimen fabrication and mechanical properties of the composite are described elsewhere[7,18].

Bundle and Flexure Tests

Fiber bundle test, originally developed by Manders and Chou[19], was employed to determine single fiber strength distribution of as-fabricated Nicalon fibers. Weibull parameters, needed to describe the strength distribution of fibers, were estimated from the load versus strain plots of a fiber bundle loaded in uniaxial tension[20]. Bundle tests were conducted on a universal testing system# using the experimental set-up shown schematically in Figure 1. Tests were conducted on fiber tows of various gage lengths ranging from 27 mm to 100 mm in ambient conditions and at a loading rate of 0.5 mm/min.

Model 4505, Instron Corp., Canton, MA.

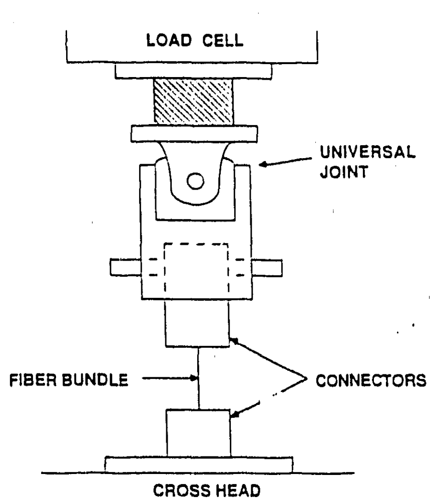

Figure 1 Schematic of the Experimental Set-up Used for Bundle Tests.

Continuous Nicalon fiber-reinforced SiC composites were fractured in four-point-bend mode on the universal testing system. Inner and outer loading spans were 9.53 mm and 19.05 mm, respectively. Typical dimensions of the flexure bar specimens were 2.9 mm x 4.2 mm x 51.0 mm. Flexure tests were conducted at a loading rate of 1.27 mm/min in ambient conditions. Fractured composite specimens were examined on a scanning electron microscope## (SEM) to locate the failure origin and establish the associated characteristic fracture surface morphology for the fibers.

Results and Discussion

Figure 2 shows a typical load versus strain plot obtained from a bundle test. Strain or the displacement in the fiber bundle, at a particular load, was determined by subtracting the system (grips, connectors, etc.) displacement from the absolute displacement of the crosshead of the testing machine. Displacement contribution due the system accessories was obtained by estimating system compliance following the procedure described in ASTM D 3379 – 75[21].

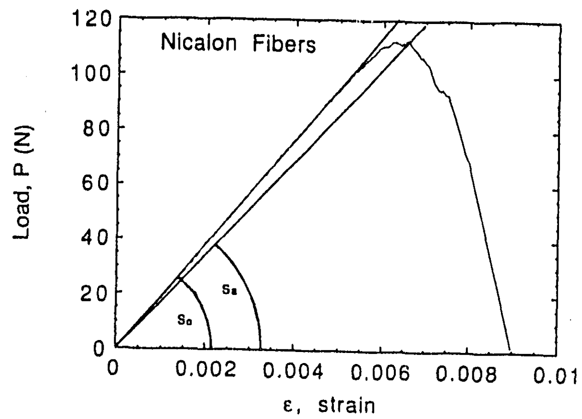

Figure 2. Typical Load–Strain Variation Obtained From a Bundle Test of Nicalon Fibers.

Model JXA-840A, JEOL Co., Ltd., Tokyo, Japan.

Weibull parameters (σ_o and m) of the as–recieved Nicalon fibers were obtained from the maximum load P_{max}, slopes S_o and S_A, (as indicated on Figure 2) and the specimen gagelength, L, via the following equations[19]:

$$m = \frac{1}{\ln\left[\frac{S_o}{S_A}\right]} \qquad (3)$$

$$P_{max} = S_o \varepsilon_o \left(\frac{1}{2.7183Lm}\right)^{1/m} \qquad (4)$$

$$\sigma_o = E_f \varepsilon_o \qquad (5)$$

Results of seven tests conducted on fiber bundles with various gagelengths gave an average value for Weibull modulus as 7.1. The average value for the scale parameter, after correcting it for a gagelength of 10 mm via equation 2, was 3.45 GPa. These results are in accordance with the reported values in the literature for Nicalon fiber strength distribution. Goda and Fukunaga[10] obtained strength distribution for Nicalon fibers by single fiber testing on fibers of gagelength of 10 mm. Their reported values for the Weibull modulus and scale parameter are 4.7 and 4.24 GPa, respectively.

SEM investigation of the fibers showed that most failed from defects or flaws located at the fiber surface. Typical surface morphology of a fractured fiber in Nicalon fiber–reinforced SiC composite tested in four–point–bend mode is shown in Figure 3. Characteristic features associated with brittle failure such as mirror (smooth region around the fracture origin) and hackle (region of multiple fracture planes) are clearly observed on the surface of fractured fibers.

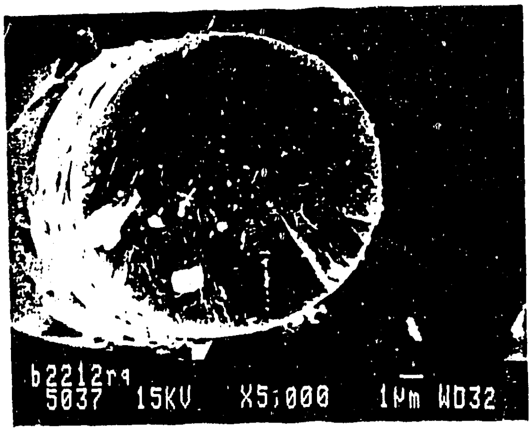

Figure 3. Surface Morphology of a Fractured Fiber in a Nicalon Fiber–Reinforced SiC Composite.

It is well known for glasses and ceramics that such fracture surface features as the mirror radii can be correlated to the tensile strength through an empirical relationship[22,23]:

$$\sigma_f r_m^{1/2} = A_m \qquad (6)$$

where r_m is the mirror radii, σ_f is the tensile strength and A_m is the mirror constant and is related to the fracture toughness of the material. In the present study, A_m is taken as 3.5 $MPam^{1/2}$ following the work of Thouless et al.[5]. Strengths of over thirty Nicalon fibers from five different fractured composite specimens were determined by measuring their fracture mirror radii and using equation 6. Figure 4 shows the linearized Weibull strength distribution plot for Nicalon fibers in a SiC matrix composite. Weibull modulus and the corresponding scale parameter were 6.0 and 2.3 GPa, respectively.

Figure 5 compares the strength distribution of Nicalon fibers in the as–fabricated state with those incorporated in the composite. As–received fibers exhibit an average strength of more than one and a half times than that of fibers incorporated in composites. Also shown in Figure 5, for comparison purposes, is the strength distribution obtained by single fiber tests of Nicalon fibers[10]. Reduction of strength of fibers suggests that either new flaws are generated or pre–existing flaws become more severe during processing. SEM examination of the fractured fibers revealed some interesting features. Figure 6 shows a fractured fiber with a distinct surface flaw believed to be introduced by mechanical damage to the fiber. Possiblities of this to occur are during the handling and stacking of fiber mats prior to densification by vapor infiltration.

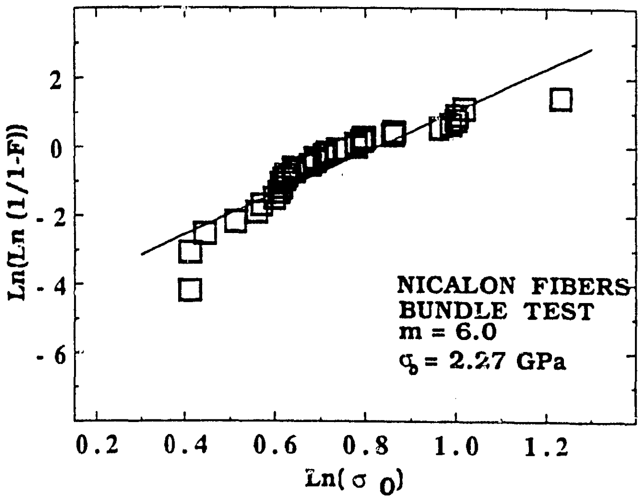

Figure 4. Linearized Weibull Plot for Fractured Nicalon Fibers in SiC Matrix Composites.

Strength degradation of Nicalon fibers due to exposure to high temperatures is well documented in the literature[12]. Loss of tensile strength by more than 30% and 70% has been reported for ceramic grade Nicalon fibers by exposure at temperatures 1000 °C and 1200 °C, respectively, for 12 hours in a wet-air atmosphere[12]. This is attributed to microstructural and stochiometric changes that occur in the fiber at elevated temperatures. Similar changes are expected, however, to a lesser degree for the carbon–coated Nicalon fibers investigated in this study. Due to the higher processing temperatures in the range of 1200 C and larger exposure times of over 24 hours used in the farbrication of the composites makes it difficult to prevent fiber degradation. Okamura et al.[24] have shown that formation of SiO_2 film can also contribute to the reduction in both the tensile strength and as well the Young's modulus of the fibers. To establish the effects of thermal degradation on the Nicalon fibers used in composites a quantitative microstructural and phase analysis of the fibers is currently in progress. Also, since there is no significant change observed in the Weibull modulus, it implies the flaw population remains same except the flaws become more severe due to degradation in the inherent material properties. This again is related to thermal degradation of the fiber material.

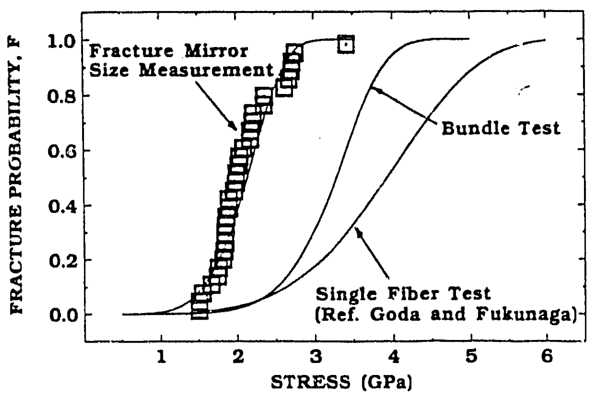

Figure 5. Comparison of Weibull Strength Distribution of Nicalon Fibers in As–Recieved State and After Processing.

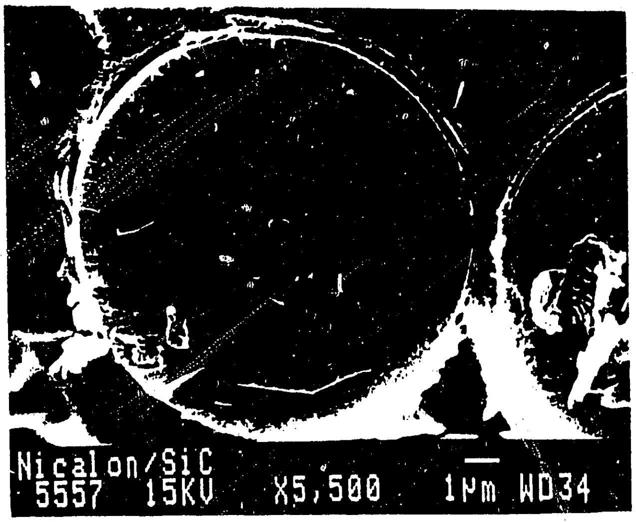

Figure 6. Fractured Nicalon Fiber in SiC Matrix Composite Showing a Surface Defect as the Flaw Origin .

Conclusions

1. Strength distribution of as–received Nicalon fibers was obtained using bundle test procedure, whereas, fracture mirror radii measurements were made to estimate strengths of fibers in composites.
2. Results indicate a significant decrease in the strength of the Nicalon fibers in composites as compared to as-recieved fibers. However, Weibull moduli for the two cases were similar.
3. Decrease in the average strengths of Nicalon fibers after incorporation in composites is attributed to thermal and mechanical degradation of fibers encountered during the processing procedures.

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