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EFFECTS OF IN SITU FIBER STRENGTH CHARACTERISTICS ON MECHANICAL PROPERTIES OF SiC(f)/SiC COMPOSITES*

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June 1994

To appear in the Proceedings of the Ceramic Matrix Composites Symposium held at the 96th Annual Meeting & Exposition, Indianapolis, April 24-28, 1994

*Work supported by U.S. Department of Energy, Advanced Research and Technology Development, Fossil Energy Material Program, under Contract W-31-109-Eng-38.

EFFECTS OF IN SITU FIBER STRENGTH CHARACTERISTICS ON MECHANICAL PROPERTIES OF SiC(f)/SiC COMPOSITES*

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Abstract

Nicalon-fiber-reinforced silicon carbide (SiC) matrix composites were tested in flexure at room and elevated temperatures. The measured strength of composites decreased slightly from a room temperature value of 400 MPa to 380 MPa at 1200°C. However, at 1300°C strength decreased significantly to 290 MPa. The rapid decrease in strength over 1300°C is believed to be due to degradation in strength of the reinforcing fibers. In situ fiber strength and fiber pullout distribution in fractured composites were estimated by fractographic techniques. Correlations were made between the measured strengths of composites to the in situ fiber strength characteristics to explain the mechanical properties of composites at room and elevated temperatures.

Introduction

Strength of reinforcing fibers is an important parameter which controls fracture behavior of continuous fiber ceramic composites (CFCC). High strength of fibers is required to facilitate successful transfer of load from matrix to fibers once a matrix crack is initiated and extended.[1] In addition, the amount of fiber pullout (which contributes to the toughening of the composite) is strongly influenced by the mean strength and the variability in strength of the reinforcing fibers.[2] Also, the ultimate load–bearing capacity of the composite is determined by fiber strength characteristics.[3] Therefore, fiber strength at room temperatures and its retention at elevated service temperatures are important in the design and development of CFCCs with superior mechanical properties.

In the present study, mechanical properties of Nicalon-fiber-reinforced SiC matrix composites were tested in flexure at room and elevated temperatures. Single–fiber strength distribution of as–fabricated Nicalon fibers was obtained from bundle tests. Strength distributions of in situ fractured fibers in Nicalon/SiC composites tested at room and elevated temperatures were assessed by measuring fracture mirror radii. Variations in the mechanical properties of composites evaluated as a function of test temperatures were correlated to the in situ fiber strength characteristics.

Experimental Procedure

Material

Carbon–coated ceramic–grade Nicalon fibers were used to study the effect of fiber strength distribution on room and elevated temperature properties of Nicalon fiber-reinforced SiC composites. Tows of Nicalon fibers were used to determine single–fiber strength distribution using bundle tests. Typically, each tow consisted of 500 individual fibers with diameters ranging from 10–15 μm. Polymer–derived Nicalon fibers consist primarily of β–SiC crystallites with an average size of 1.7 nm, along with excess carbon and SiO.[4] The reported density and elastic modulus (E_f) of Nicalon fibers are 2.55 g/cm^3 and 210 GPa, respectively.[5]

Nicalon–fiber–reinforced SiC matrix composites were used to evaluate the strength distribution of fibers in the composites, which were fabricated at Oak Ridge National Laboratory by densifying multiple layers of Nicalon mats stacked in a graphite die. Chemical vapor infiltration (CVI), under forced conditions of thermal and pressure gradients, was used to densify the preforms with SiC.[6] The fiber content in the composites was and the resulting composites were ≈90% dense. For elevated temperatures tests, composites were overcoated with a 35 μm layer of SiC to prevent oxidation of exposed carbon coating on the fiber surface caused by cutting of composite specimens.

Bundle and Flexure Tests

The fiber bundle test was used for the determination of the single–fiber strength distribution of as–fabricated Nicalon fibers. Weibull parameters, needed to describe the strength distribution of fibers, were estimated from load–vs.–strain plots of a fiber bundle loaded in uniaxial tension.[7] Bundle tests were conducted on a universal testing system** with the experimental setup shown schematically in Fig. 1. Tests were conducted on fiber tows with gage lengths ranging from 27 to 100 mm under ambient conditions and at a crosshead speed of 0.5 mm/min.

Flexural tests on bars (2.9 x 4.2 x 28.0 mm) of continuous Nicalon–fiber–reinforced SiC composites were conducted in the four–point–bend mode at room and elevated temperatures on the universal testing system. For room–temperature tests, loading and support spans were 9.53 and 19.05 mm, respectively, and tests were conducted at a crosshead speed of 1.27 mm/min at ambient conditions. For tests at elevated temperatures, SiC fixtures with loading and support spans of 12.7 mm and 25.4 mm, respectively, were used. The crosshead speed for these tests was 1 mm/min. The composite bars were held at the test temperature for 15 minutes in air prior to fracturing them in vacuum. All specimens were loaded perpendicular to the layers of mats. Fractured composite specimens were examined

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**Model 4505, Instron Corp., Canton, MA.

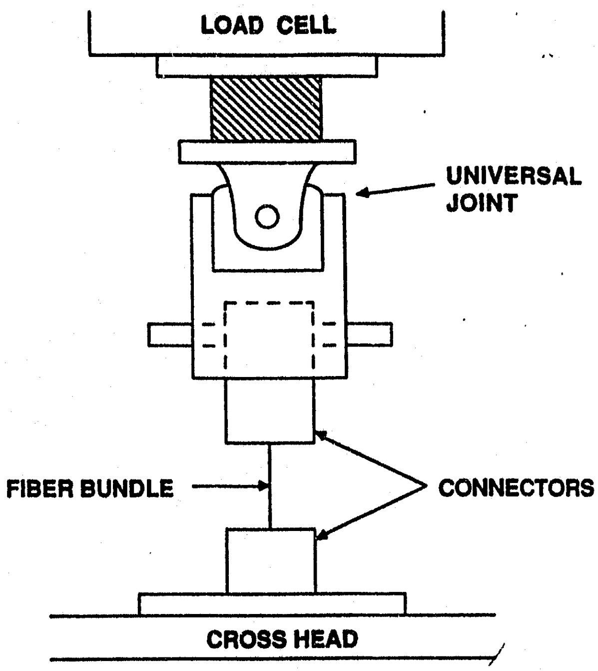


Figure 1. Schematic Representation of Experimental Set–Up Used for Bundle Tests.

by scanning electron microscopy (SEM)† to locate the origin of failure and establish the associated characteristic fracture surface morphology of the fibers.

Results and Discussion

Strength Distribution of As–Fabricated Nicalon Fibers

In the present study, distribution function used to describe Nicalon fiber strength is given in Eqn. 1. This formulation was used because strength distributions of fibers were obtained from fibers having various gage lengths and it was appropriate to account for the size effect and represent the Weibull strength distribution at a standard gage length, L, taken to be 10 mm.

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

In Eq. 1, $F(\sigma)$ is the cumulative failure probability at an applied stress σ, σ_o is the scale parameter signifying a characteristic strength of the distribution, and m is the

†Model JXA–840A, JEOL Co., Ltd., Tokyo, Japan.

Weibull modulus that characterizes flaw distribution in the material, and L_o is the fiber gage length at which the Weibull parameters are determined.

Figure 2 shows a typical load–strain plot obtained from a bundle test. Strain or 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 due to system accessories was obtained by estimating system compliance. Weibull parameters (σ_o and m) of the as–fabricated Nicalon fibers were obtained from the maximum load P_{max}, slope of the linear portion of the load–strain behavior, S_o, slope of the line joining the maximum load to the origin, S_A, (as indicated on Fig. 2) and the specimen gage length, L_o, by solving the following equations:[7]

$$m = \frac{1}{\ln\left[\frac{S_o}{S_A}\right]} \tag{2}$$

$$P_{max} = S_o \varepsilon_o \left(\frac{1}{2.7 L_o m}\right)^{\frac{1}{m}} \tag{3}$$

$$\sigma_o = E_f \varepsilon_o \tag{4}$$

Results of seven tests conducted on fiber bundles with various gage lengths gave an average value for the Weibull modulus as 7.1. The average value for the scale parameter, after correcting it for a gage length of 10 mm using Eq. 1, was 3.45 GPa. These results are in accordance with reported values in the literature for Nicalon fiber strength distribution.[8]

Bulk Mechanical Property Characterization

Figure 2 shows the typical load-displacement behavior obtained from the flexure tests conducted at room temperature. Room temperature tests showed more extensive fiber pullout and larger work-of-fracture compared to the elevated temperature tests. The ultimate strength is obtained from the peak load. The results of mechanical property measurements are shown in Figure 4. Measured strength shows a small decrease from a room temperature value of 400 MPa to 380 MPa at 1200°C. However, at 1300°C strength decreases significantly to 290 MPa. This rapid decrease in strength over 1300°C is believed to be due to degradation in strength of the reinforcing fibers.

In situ Fiber Strength Evaluations

SEM was used for fractographic evaluation to estimate in-situ fiber strengths in composites tested at room and 1300°C. Specifically, critical flaws and the associated characteristic fracture markings were identified. In-situ fiber strength in the composites was evaluated from measurements of these characteristic markings. Typical fracture surface of a fiber in a Nicalon-fiber–reinforced SiC composite,

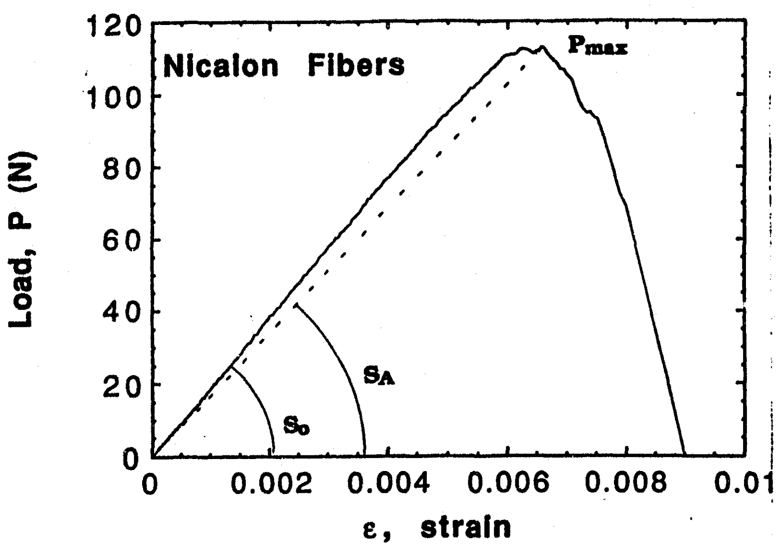


Figure. 2. Typical Load–Strain Variation Obtained from a Bundle Test of Nicalon Fibers.

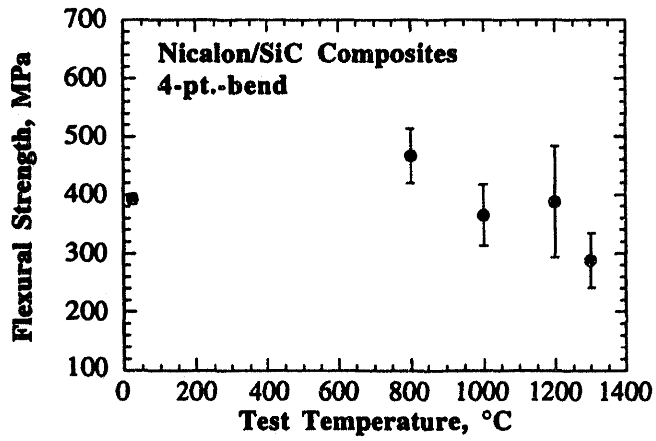


Figure 3. Variation of Ultimate Strength with the Test Temperature for Nicalon Fiber-Reinforced SiC Composites.

tested in the four–point–bend mode, is shown in Fig. 4. Characteristic features associated with brittle failure, such as mirror (a smooth region around the fracture origin) and hackle (a region of multiple fracture planes), are clearly observable on the surface of fractured fibers. SEM investigation showed that most fibers failed from defects or flaws at the fiber surface.

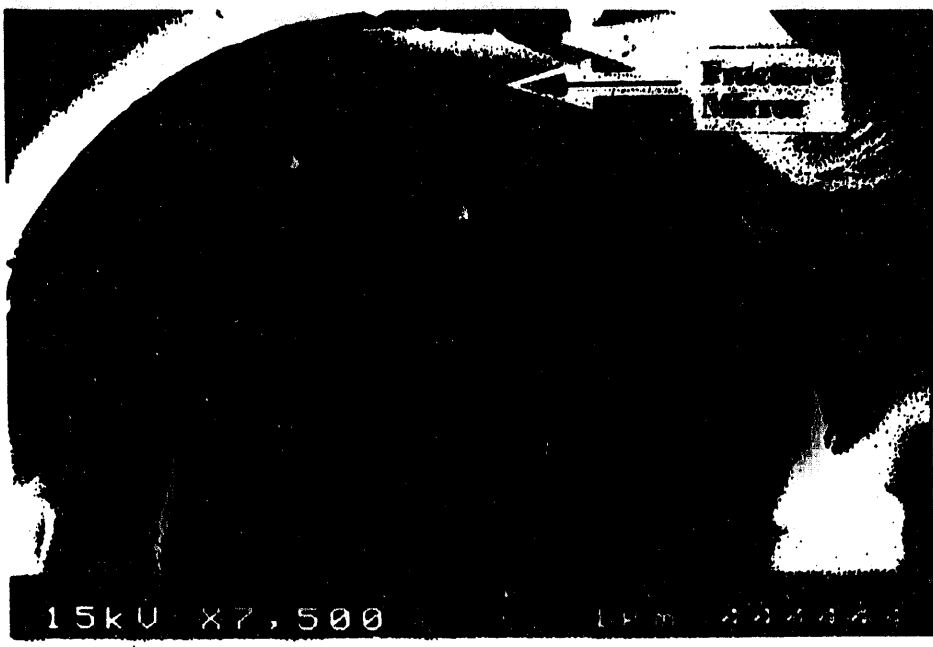
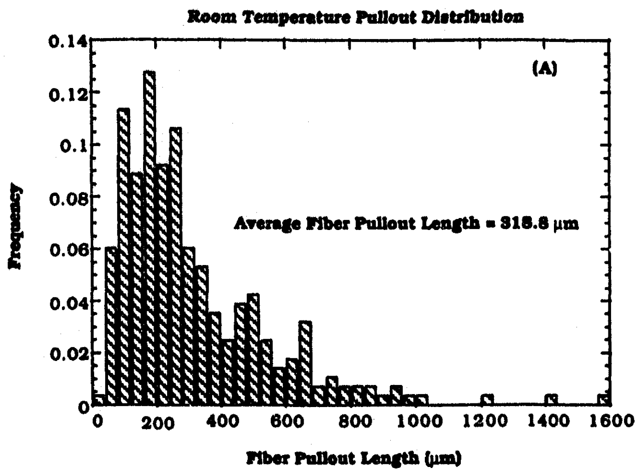


Figure 4. Characteristic Features on a Fractured In Situ Nicalon Fiber

For glasses and ceramics, fracture surface features such as mirror radii can be correlated to tensile strength through the empirical relationship:[9]

$$\sigma_f\sqrt{r_m} = A_m \tag{5}$$

where r_m represents the mirror radii, σ_f is the tensile strength, and A_m is the mirror constant, which is related to the fracture toughness of the material. In the present study, A_m is taken as 3.5 $MPam^{1/2}$.[2] Strengths of more than 30 Nicalon fibers for each set of composite specimens were determined by measuring their fracture mirror radii and using Eq. 5. However, it should be noted that the Weibull scale parameter has to be corrected to account for effective fiber gage length, of the in situ fibers, over which the stresses are acting. Therefore, it was assumed here that the in situ fiber gage length are of the order of the fiber pullout lengths. To this end, fiber pullout lengths were measured on the room and 1300°C tested specimens and are shown in Fig. 5A&B. The average values for pullout lengths for composites tested at room and elevated temperatures were ≈320 µm and 200 µm, respectively. Using the values of average pullout lengths, scale parameters were evaluated at the standard gage length of 10 mm. Table 1 lists the various Weibull strength distribution parameters. It should be noted that the values for scale parameters reported in Table 1 is at a gage length of 10 mm.



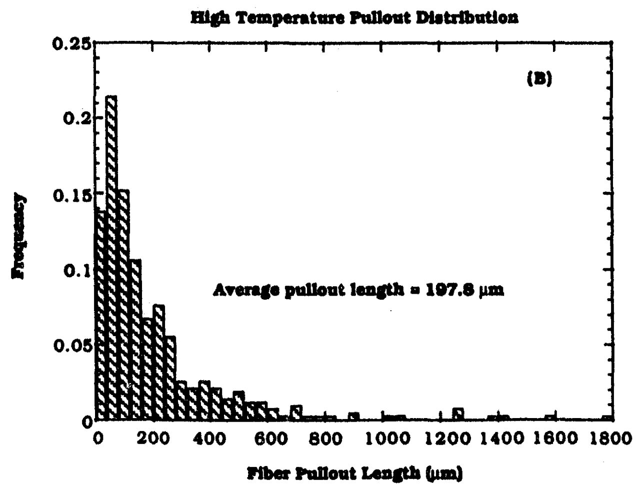


Figure 5. Fiber Pullout Length Distribution in Composites Tested at Room and 1300°C

Table 1. List of Weibull Strength Parameters of As-Fabricated and In situ Nicalon Fibers in Composites Tested at 24°C and 1300°C.

Fiber Condition	Test Temperature (°C)	Scale Parameter, σ_0 (GPa)	Weibull Modulus, m
As-Fabricated	24	3.45	7.1
In situ	24	1.28	6.0
In situ	1300	0.96	5.2

Figure 6 shows the Weibull strength distribution plots, for Nicalon fibers in the three different states, constructed using the data in Table 1 and using Eqn. 1. It is clear from this figure that there is a significant decrease (≈60%) in the strength of Nicalon fibers from its as-fabricated state to after composite fabrication and their incorporation in composites. There is additional 25% drop in the strength of Nicalon fibers when the composites are exposed to 1300°C. These drop in strength is believed to be due to the mechanical and/or thermal damage that the fibers undergo during processing. Strength degradation of Nicalon fibers due to exposure to high temperatures is well documented in the literature. Loss of tensile strength of more than 30 and 70% has been reported for ceramic–grade Nicalon fibers by exposure at temperatures 1000 and 1200 °C, respectively, for 12 h in a wet–air atmosphere.[10] This loss of tensile strength is attributed to microstructural and stochiometric changes that occur in the fibers at elevated temperatures. Similar changes, although not as severe, are expected for the carbon–coated Nicalon fibers investigated in this study. Higher processing temperatures (in the range of 1200 °C) and longer exposure times (over 24 h) used in the fabrication of the composites make it difficult to prevent fiber degradation. Okamura et al.[11] have shown that formation of an SiO_2 film can also contribute to the reduction of both the tensile strength and the Young's modulus of the fibers.

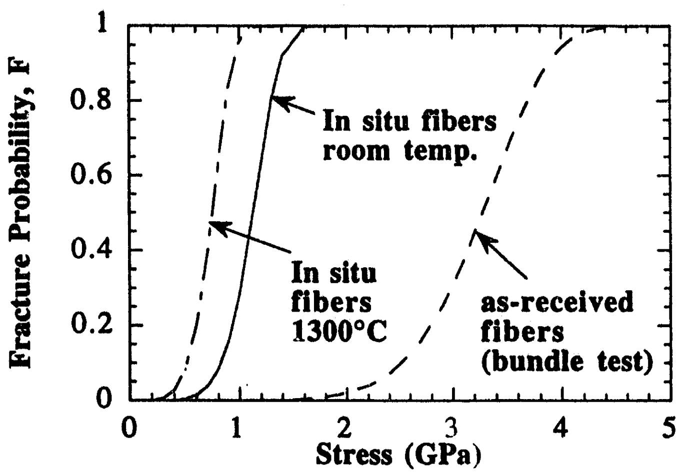


Figure 6. Weibull Strength Distribution Plots of Nicalon Fibers in As-Fabricated State, After Incorporation in Composites, and After Exposure of Composites to 1300°C.

Conclusions

1. Strength distribution of as-fabricated Nicalon fibers was obtained from bindle tests, whereas the strength of in situ fibers were evaluated from fracture mirror sizes.

2. In situ strength of Nicalon fibers in composites is lower than the strength of as-fabricated Nicalon fibers.

3. Decrease in strength of Nicalon fibers after incorporation in composites is attributed to thermal and mechanical degradation of fibers during fabrication.

4. High-temperature exposure (1300°C) further decreases the in situ fiber strength of Nicalon fibers which is reflected in the drop in the composite ultimate strength at those temperatures.

Acknowledgments

This work was supported by the U.S. Department of Energy, Advanced Research and Technology Development, Fossil Energy Materials Program, under Contract W–31–109–Eng–38. The authors thank D. P. Stinton of Oak Ridge National Laboratory for providing the composite specimens. The authors are grateful to Dr. Jules Routbort for permitting us to use the high temperature testing facility and Martha Boling–Risser for providing assistance in using the high temperature test facility.

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