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EFFECT OF FIBER ARCHITECTURE ON MECHANICAL BEHAVIOR OF SiC(f)/SiC COMPOSITES*

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DISCLAIMER

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ABSTRACT

We evaluated mechanical properties (first matrix cracking stress, strength, and work-of-fracture) of Nicalon-fiber-reinforced silicon carbide matrix composites with three different fiber lay-up sequences (0°/20°/60°, 0°/40°/60°, and 0°/45°) at various temperatures from room to 1300°C. Up to 1200°C, ultimate strength and work-of-fracture for the 0°/40°/60° and 0°/45° composites increased, but then declined at 1300°C. The decreases were correlated to in-situ Nicalon fiber strength and fiber/matrix interface degradation. However, for the 0°/20°/60° composites, ultimate strength and work-of-fracture reached their a minima at 1200°C. These measured ultimate strengths at room and 1300°C were correlated to the predictions made with an analytical model and to in-situ fiber strength characteristics. The large difference in room-temperature ultimate strengths between the three sets of composites is attributed to the relative contributions of the off-axis fibers to the load-bearing capacity of each composite.

INTRODUCTION

Continuous fiber–reinforced ceramic matrix composites (CFCCs) are being pursued as materials for structural applications because they provide a unique combination of high strength and toughness at both room and elevated temperatures [1]. Mechanical properties of CFCCs, for a fixed fiber content, is largely controlled by intrinsic composite parameters, including strengths of the reinforcing fibers and matrix [2,3], fiber/matrix interface characteristics [4], and internal residual stresses [5]. However, in recent years, demanding material-performance needs such as multiaxial stress states have led to development of CFCCs with increasingly complex designs. To date, few experimental studies have been conducted to establish correlations between material design parameters such as fiber orientation and composite properties.

In the present study, SiC-fiber-reinforced SiC matrix composites were used in evaluating mechanical properties with three different fiber lay-up sequences. Rectangular bars of composites were tested in flexural mode at room and elevated temperatures. Mechanical properties such as first matrix cracking stress, ultimate strength, and work-of-fracture were evaluated as functions of test temperature. Strength of in-situ fibers in composites at room and elevated temperatures were measured by fractographic techniques. Correlations were made between observed composite mechanical properties and fiber lay-up sequences to establish the effect of fiber orientation on mechanical properties at room and elevated temperatures.

EXPERIMENTAL PROCEDURE

Material

Nicalon (SiC)-fiber–reinforced SiC matrix composites, fabricated by Ceramic Composites, Inc. (Millersville, MD), were chosen for this study because of their potential for high–temperature structural use in advanced heat engines and hot-gas filters. The composites were fabricated by densifying multiple layers of 2-D plain weave Nicalon mats with a chemical vapor infiltration process. Composites with three different lay-up sequences (0°/20°/60°, 0°/40°/60°, and 0°/45°) were tested. The fibers were coated with carbon to a thickness of 0.4 μm in all sets of composites. Fiber content in the final composite was ≈40 vol.%. The composites were fabricated into billets from which rectangular bar specimens were cut for evaluation of mechanical properties. Densities of composites with fiber lay-up sequences of 0°/20°/60°, 0°/40°/60°, and 0°/45°, measured by the fluid displacement method, were 2.40, 2.25, and 2.35 g/cm^3, respectively.

Mechanical Testing

Flexure testing in a four–point–bend mode were used to evaluate mechanical properties of the composites at room and elevated temperatures. This method was chosen because of its relatively low cost and ease of use. For room–temperature tests, flexural bars (2.9 x 4.2 x 25.4 mm) were tested with loading and support spans of 9.5 and 19.0 mm, respectively. All tests were conducted at a crosshead speed of 1.27 mm/min at ambient conditions on a universal testing machine. High-temperature flexure tests were conducted at 1000, 1200, and 1300°C. The composite bars for high-temperature tests had an additional SiC surface coating (≈100 μm thick) to prevent oxidation of the exposed carbon coating on the fiber surfaces. SiC fixtures with loading and support spans of 12.7 and 25.4 mm, respectively, were used, and crosshead speed was 1 mm/min. All specimens were loaded perpendicular to the mat layers. At least three specimens were tested under each set of test conditions. Fractured composite specimens were examined on a scanning electron microscope (SEM) to locate and identify critical flaws in the fibers. In addition, pullout lengths of fractured fibers were measured to establish fiber/matrix interfacial strength characteristics [3].

The first matrix cracking stress was determined from the load at which first deviation from the linear variation in the load–vs.–displacement plots was observed. Nominal ultimate stress was determined from the peak load value. Composite work-of-fracture (WOF) was estimated from the total area under the load-specimen displacement plots normalized on the basis of unit cross-sectional area of the fractured composites.

ANALYTICAL BACKGROUND

Mechanical response of continuous fiber-reinforced ceramic matrix composites with increasing stress levels is dependent on in-situ fiber strength and its distribution. Based on the weakest-link-principle, i.e., failure occurs at the most severe flaw, strength distribution of fibers can be represented by the Weibull distribution function as follows:

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

where $F(\sigma)$ is the cumulative failure probability at an applied stress σ, σ_o is the scale parameter signifying a characteristic fiber strength at a fiber gauge length, L_o, and m is referred to as the Weibull modulus that characterizes flaw distribution in the material. Thus, by using the Weibull distribution function as given by Eq. 1, we can estimate Weibull scale parameter at some standard gauge length, L, with the following expression:

$$\sigma = \sigma_o \left(\frac{L_o}{L} \right)^{\frac{1}{m}} \tag{2}$$

The in-situ fiber strength distribution parameters, σ_C and m, can be evaluated by measuring mirror sizes on fractured fibers. Figure 1 shows typical flaw morphology and associated fracture features such as mirrors (smooth regions) and hackles (regions of multiple fracture planes) on a Nicalon fiber in a composite tested at room temperature. For brittle materials such as glasses and ceramics, it is possible to correlate sizes of fracture features to fracture stress with empirical relationships. For example, fracture stress, σ_f, of the fibers can be obtained from mirror-size measurements with the following relationship [6]:

$$\sigma_f = \frac{3.5K_f}{\sqrt{r_m}}, \tag{3}$$

where r_m is the mirror radius and K_f is the fracture toughness of the fiber. This semiempirical relationship is applicable for mirror sizes much smaller than the fiber diameter.

Based on the fiber fragmentation theory [5], the resulting value of scale parameter, σ_C, from fracture mirror evaluations is at a gauge length, L_C, that is controlled by fiber/matrix interfacial shear strength and fiber strength. An average value for the gauge length for in-situ fractured fibers can be written as

$$L_c = r\frac{\sigma_c}{\tau}, \tag{4}$$

where r is the fiber radius and t is the fiber/matrix interfacial shear strength.

The fiber/matrix interfacial shear strength can be determined from average fiber pullout length measurement, h, as [3,5]

$$\tau = \frac{\lambda(m) r \sigma_c}{4h}, \tag{5}$$

where $\lambda(m)$ is a nondimensional function and is dependent on fiber fracture statistics.

Fig. 1. Fracture Surface Morphology of SiC (Nicalon) Fiber.

In-situ fiber strength distribution parameters (σ_c and m) can be correlated to the ultimate strength, σ_{UTS}, of the composite as follows [5]:

$$\sigma_{UTS} = f_l \sigma_c \left(\frac{2}{m+2} \right)^{1/m+1} \left(\frac{m+1}{m+2} \right) , \qquad (6)$$

where f_l is the fiber volume fraction parallel to the loading direction.

RESULTS AND DISCUSSION

Figure 2 shows typical load-displacement behavior obtained from flexure tests conducted on $SiC_{(f)}/SiC$ composites with 0°/40°/60° fiber lay-up sequence at both room and elevated temperatures. Similar variations in load displacement were observed for the 0°/20°/60° and 0°/45° composites. Gradual failure was observed in all tests. However, the area under the curve increased somewhat in tests at elevated temperatures. It is recognized here that because of the generation of matrix crack(s) and the shift in the neutral axis, use of the simple beam theory to assess ultimate

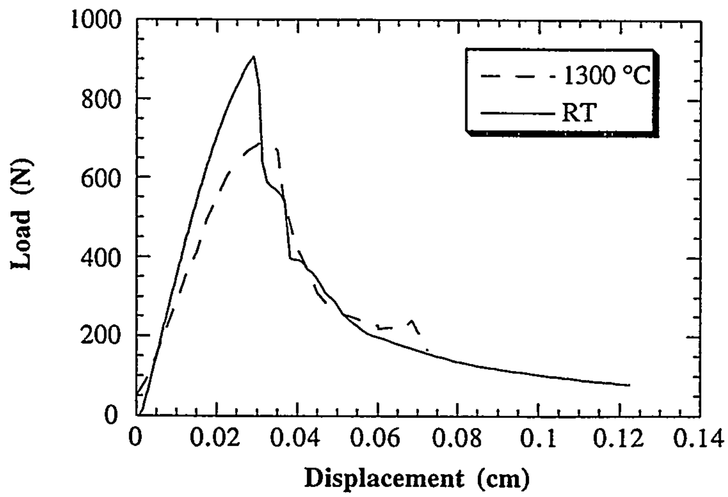

Fig. 2. Typical Load-Displacement Observed for 0°/40°/60° Composites Tested at Room Temperature (RT) and 1300°C.

stresses gives a semiquantitative estimate of ultimate strength. However, the purpose of estimating these values is to compare the relative load-bearing properties of the composites under specific fiber orientations and test conditions.

Table I shows the room-temperature mechanical properties of the SiC(f)/SiC composites with the three fiber orientations. Mechanical properties of the composites with 0°/20°/60° and 0°/40°/60° were similar in magnitudes, whereas the composites with a fiber sequence of 0°/45° had relatively lower values. The decrease in mechanical properties for composites with the 0°/45° sequence is believed to have two causes: first, composites with a fiber lay-up sequence of 0°/45° had a lower density (2.25 g/cm^3) than composites with other fiber lay-up sequences; the second cause could be related to the smaller fiber fraction in the loading direction for 0°/45° composites relative to that of composites with other fiber lay-up sequences. This is discussed in more detail later.

Figure 3 shows the variation of ultimate strength for the three sets of composites (0.4 µm coating thickness) as a function of temperature. At 1000°C, the strength of 0°/40°/60° composites was similar to its room-temperature value. No 0°/45 specimens were available for tests at 1000°C. Beyond 1000°C, the ultimate strengths of both sets of composites increased dramatically over their

Table I. Room-Temperature Mechanical Properties of SiC(f)/SiC Composites with Various Fiber Architechtures

Fiber Architecture	First Matrix Cracking Stress (MPa)	Ultimate Stress (MPa)	Work-of-Fracture (kJ/m^2)
0°/20°/60°	115 ± 25	287 ± 8	15.7 ± 4
0°/40°/60°	116 ± 28	312 ± 28	14.4 ± 4
0°/45°	86 ± 23	153 ± 41	9.8 ± 2

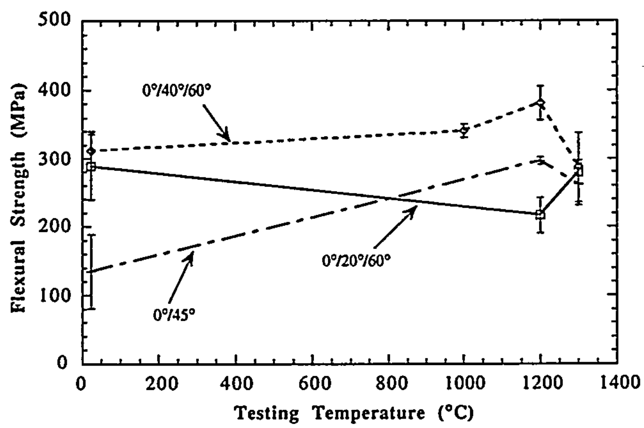

Fig. 3. Variation of Ultimate Strength with Test Temperature for SiC(f)/SiC Composites of Different Fiber Lay-up Sequences.

room-temperature values, probably because of matrix-softening effects at elevated temperatures. Such behavior is well documented in monolithic ceramics and CFCCs [7]. In general, at elevated temperatures, the 0°/40°/60° composites had higher strengths than those of the 0°/45° composites. However, at 1300°C, strengths of both sets of composites dropped to ≈270 MPa. This decrease in strength above 1200°C is believed to be due to the degradation in strength of the reinforcing fibers [8]. In the 0°/20°/60° composites, strength remained relatively unchanged up to 1300°C, except for a slight decrease at 1200°C.

Observed WOF variation with test temperature of two sets of composites (0°/45° and 0°/40°/60°) was similar and is shown in Fig. 4. With increasing test temperature, WOF reached its peak at 1200°C because of matrix-softening effects, but dropped rapidly above 1300°C. This drop is related to physical changes in the in-situ Nicalon fibers in composites tested at elevated temperatures; formation of silica at the fiber surface is a distinct possibility at elevated temperatures and can lead to degradation of fiber/matrix interfacial properties. This oxidation can minimize the effective fiber pullout during fracture of the composites, thus accounting for low WOF values. However, for the 0°/20°/60° composites, the WOF remained relatively unchanged up to 1300°C, except for a slight decrease at 1200°C. This behavior is consistent with observed strength behavior. The difference in mechanical behavior of the 0°/20°/60° composites from that of the 0°/40°/60° and 0°/45° composites is probably due to specimen-related variations.

To estabish the strength variation of the composites as a function of temperature, in-situ fiber strength measurements were made on composites with the three lay-up sequences. In-situ fiber strength measurements were made by fractography on the composites samples fractured at room temperature and at 1300°C. In most fibers, fractures originated at surface flaws, as shown in Fig. 1. Using mirror size measurements and a value of 1 MPa$\sqrt{m}$ as the Nicalon fiber fracture toughness [3], we estimated fiber strengths from Eq. 3. These strength values were then used to construct linearized Weibull plots [9,10], from which the scale parameters and Weibull moduli for the composites were determined. Results are tabulated in Table II.

Equation 6 was used to determine the ultimate strengths of the three sets of composites tested at room temperature and 1300°C and are listed in Table III. As a first approximation, the fraction of fibers along the loading direction (i.e., 0°) are accounted for in the calculations. Therefore, values of f_l for the 0°/20°/60°, 0°/40°/60°, and 0°/45° composites are 0.07, 0.07, and 0.1, respectively. Based on these values and the Weibull parameters, room-temperature predicted ultimate strengths for the 0°/20°/60°, 0°/40°/60° and 0°/45° composites are 88, 117, and 165 MPa. The predicted strength for the 0°/45° composites agrees well with the observed room-temperature strength of ≈153 MPa, while for the 0°/20°/60° and 0°/40°/60° composites, there is a large discrepancy. It is possible that in the 0°/20°/60° and 0°/40°/60° composites, fibers in the lay-ups oriented at 20° or 40° and 30° (in 60° oriented mats) are contributing to the mechanical response of the

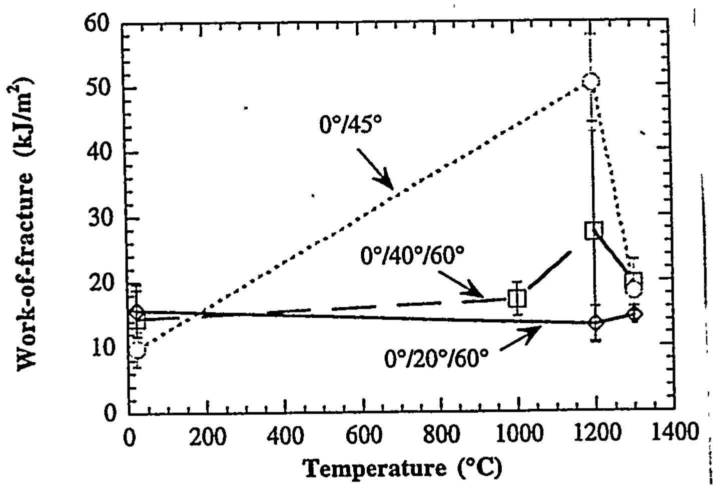

Fig. 4. Variation of Work-of-Fracture with Test Temperature for SiC(f)/SiC Composites of Different Fiber Lay-up Sequences.

Table II. Weibull Parameters of SiC(f)/SiC Composites Tested at Room-Temperature and 1300°C

Fiber Orientation	Test Temperature (°C)	Scale Parameter (GPa)	Weibull Modulus
0°/20°/60°	RT	1.71	6.8
	1300	2.20	4.6
0°/40°/60°	RT	2.38	4.9
	1300	2.53	6.5
0°/45°	RT	2.42	4.9
	1300	2.42	5.4

composite. If the contribution of these fibers is included in the model, the predicted strength for the 0°/20°/60° and 0°/40°/60° is 234 and 289 MPa; this is in accordance with their observed strengths of ≈300 MPa. It seems that fibers oriented off-axis by more than 45° do not contribute significantly to the ultimate strength of the composites. However, if they are oriented <40° from the loading direction, they do influence composite strength. Moreover, the contribution of the off-axis fibers on the composite strength is expected to change gradually with the off-axis angle.

Table III. Comparison of Predicted and Observed Ultimate Strengths of SiC(f)/SiC Composites with Varying Fiber Orientations

Fiber Orientation	Fiber Fraction (fl)	Test Temperature (°C)	Predicted Ultimate Strength (MPa)	Observed Ultimate Strength (MPa)
0°/20°/60°	0.19	RT	234	288
		1300	282	280
0°/40°/60°	0.18	RT	289	312
		1300	322	285
0°/45°	0.1	RT	168	153
		1300	171	262

Comparison of the predicted and observed strength of the composites tested at 1300°C also shows good agreement for the 0°/20°/60° and 0°/40°/60° composites. However, for the 0°/45° composites there is a large difference the reason for which is not clear. It is quite possible that in addition to the fiber fraction available to sustain the applied loads, there may also be a change in the failure mechanism as the fiber lay-up sequence changes in the composites.

CONCLUSIONS

1. Mechanical response (ultimate strength) of SiC(f)/SiC composites is dependent on fiber lay-up sequence.

2. In-situ fiber strength characteristics, in conjunction with an analytical model, provide a good prediction of the ultimate strength of composites with different fiber architectures.

3. Comparison of the predicted and observed ultimate strengths for composites with 0°/20°/60°, 0°/40°/60°, and 0°/45° fiber lay-up sequences suggests that fibers off-axis by more than 40° from the loading direction contribute only minimally to ultimate strengths of composites.

4. At elevated temperatures, mechanical properties of both sets of composites increase due to matrix-softening effects. However, at 1300°C, both ultimate

strength and work-of-fracture degrade, probably because of degradation in the Nicalon fibers and also because of increased fiber/matrix interfacial shear strengths due to oxidation effects.

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