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**EFFECTS OF HIGH-TEMPERATURE ENVIRONMENTS ON FLAW
GENERATION AND FRACTURE BEHAVIOR OF SiC/SiC COMPOSITES***

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EFFECTS OF HIGH-TEMPERATURE ENVIRONMENTS ON FLAW GENERATION AND FRACTURE BEHAVIOR OF SiC/SiC COMPOSITES

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ABSTRACT

Flaw generation and fracture behavior of Nicalon-fiber-reinforced SiC matrix composites are influenced by high temperatures. Therefore, we evaluated the effects of temperature by measuring the strength of these composites at several temperatures ranging from 800 to 1300°C, and by characterizing their microstructure. While composite strength increased from ≈ 400 MPa at room temperature to ≈ 532 MPa at 800°C, it decreased to ≈ 270 MPa at 1300°C. Such degradation of composite strength at high temperature is believed to be partly due to fiber degradation. In-situ fiber strength of composites tested at room and elevated temperatures was estimated by fractographic techniques. The in-situ strength of fibers in composites was significantly lower than that of as-received fibers. This decrease is believed to be related to flaws that develop during processing and to exposure to elevated-temperature service environments.

INTRODUCTION

Continuous-fiber ceramic composites (CFCCs) are candidate materials for structural applications at elevated temperatures because of their improved flaw tolerance, large work of fracture, and noncatastrophic mode of failure.^{1,2} Fiber strength is an important parameter that controls fracture

behavior of CFCCs. High strength and weak fiber/matrix interfaces are requisites for "tough" ceramic composites.² Strength and toughness of composites are greatly influenced by the strength of reinforcing fibers.³ High fiber strength is critical because, once a matrix crack is initiated and extended, load is transferred from the matrix to the fibers in the wake of the crack. Weak fibers fracture and lead to catastrophic failure of the composite, whereas strong fibers accommodate the stresses. Therefore, it is clear that fiber strength and strength retention in elevated-temperature service environments are important parameters to be considered when designing and developing CFCCs.

The fiber reinforcements of composites are susceptible to thermal degradation and mechanical damage during fabrication and in elevated-temperature service environments. Therefore, it is important to evaluate the effects of high-temperature environments on flaw generation and on the resulting mechanical properties of reinforcing fibers and composites. In this paper, we report results of an investigation into the effects of elevated-temperature environments on the mechanical properties of Nicalon/SiC composites and on strength distribution in Nicalon fibers.

EXPERIMENTAL PROCEDURE

Carbon-coated Nicalon-fiber-reinforced SiC matrix composites fabricated by chemical vapor infiltration, were obtained from Oak Ridge National Laboratory. As-received composites (2 x 3 x 45 mm) were $\approx 90\%$ dense and had a fiber cloth layup sequence of $0^\circ/30^\circ/60^\circ$. Details of fabrication are described in Ref. 4. Composite specimens were fractured at room temperature and various elevated temperatures, ranging from 800 to

1300°C. For room-temperature testing, 2.9 x 4.2 x 25.4-mm bar specimens, were fractured in a four-point-bend mode with loading and support spans of 9.53 and 19.05 mm, respectively. Crosshead speed was maintained at 1.27 mm/min. For testing at elevated temperature, specimens were first exposed to the test temperature in air for 15 min. Subsequently, the specimens were tested in vacuum by fracturing in a four-point-bend mode at a crosshead speed of 1 mm/min. The loading and support spans were 12.7 and 25.4 mm, respectively. All specimens were loaded perpendicular to the layers of fiber mats. The fractured composites were evaluated by scanning electron microscopy to evaluate fiber pullout length and critical flaws.

Single-fiber strength distribution of as-fabricated Nicalon fibers was obtained by the fiber bundle test.⁵ Weibull parameters for strength distribution were estimated from load-vs-strain plots of a fiber bundle loaded in uniaxial tension.⁶ Tests were conducted on a universal testing* system that uses fiber tows with gauge lengths ranging from 27 to 100 mm under ambient conditions and at a crosshead speed of 0.5 mm/min.

RESULTS AND DISCUSSION

Figure 1 shows strength variation of composites as a function of temperature. The ultimate strength first increased from ~390 MPa at room temperature to ~470 MPa at 800°C, then decreased rapidly to ~290 MPa at 1300°C. The strength degradation is believed to be partly due to fiber degradation, which was evaluated by estimating the size of critical flaws for fibers in composites tested at various temperatures.

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

Fig. 1. Variation of strength of Nicalon/SiC composites with temperature.

The single-fiber strength distribution of carbon-coated ceramic-grade Nicalon fiber tows was evaluated by bundle tests.⁵ Nicalon fiber tows of various lengths were carefully extracted from Nicalon fiber mats.[†] Typically, each tow consisted of 500 individual fibers with nominal diameters of 10–15 μm . In-situ strength distribution of fractured fibers in Nicalon/SiC composites, as-fabricated and after exposure to 1300°C, was assessed by measuring fracture mirror radii.⁷ The measured values of in-situ strength were described by the Weibull strength distribution function, as shown below in Eq. 1.

$$F(\sigma) = 1 - \exp \left[-\frac{L}{L_0} \left(\frac{\sigma}{\sigma_0} \right)^m \right] \quad (1)$$

where $F(\sigma)$ is the cumulative failure probability at an applied load σ ; L_0 is the fiber gauge length at which Weibull parameters are estimated; L is the

[†]Distributed by Dow Corning Corp., Midland, MI.

standard gauge length, taken to be 10 mm; σ_0 is the scale parameter signifying a characteristic strength of the distribution; and m is the Weibull modulus that characterizes the flaw distribution in the material. Scanning electron microscopy of the fractured composites (tested at room temperature and 1300°C) was used to determine fiber pullout length distribution (as shown in Fig. 2), from which the gauge length of fractured fibers was determined. Fiber gauge length was taken to be approximately equal to fiber pullout length and was 320 μm in room-temperature tests and $\approx 200 \mu\text{m}$ in high-temperature tests. Using these values for fiber length, we estimated the values of σ_0 to be 1.20 GPa and 0.84 GPa for room- and high-temperature-tested composites, respectively.

Figure 3 shows the in-situ strength distribution of Nicalon fibers in composites tested at room temperature and at 1300°C. For comparison, strength distribution of as-fabricated fibers, measured by the bundle test, is also shown in the figure. It was observed that the Weibull modulus of these strength distributions was similar (5.2 for fibers tested at 1300°C, 6.0 for fibers tested at room temperature, and 7.1 for as-received fibers). However, the strength of the fibers decreased by $\approx 50\%$ after incorporation into composites. This strength decreased further by $\approx 30\%$ at 1300°C. The strength reduction during composite processing is believed to be caused either by increased severity of preexisting flaws or by introduction of new flaws from mechanical and thermal damage. Further strength reduction at 1300°C is attributed to microstructural and stoichiometric changes in the fibers at elevated temperatures. Similar degradation has been reported for ceramic-grade Nicalon fibers exposed to 100 and 1200°C for 12 h in a wet-air

Fig. 2. Fiber pullout length distribution of Nicalon fibers in Nicalon/SiC matrix composite tested at (a) room-temperature and (b) 1300°C.

Fig. 3. Weibull strength distribution of Nicalon fibers as-fabricated, after processing, and after exposure of composites at 1300°C for 15 min.

atmosphere.⁸ Okamura et al.⁹ have shown that formation of an SiO₂ film can also contribute to reduction of fiber tensile strength and Young's modulus.

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