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Nicalon-Fiber/SiC Matrix Composites*

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Abstract

The effect of fiber-coating thickness on retained in-situ fiber strength and the resulting mechanical properties of composites was investigated. Flexure tests in a four-point-bend mode were used to evaluate ultimate strength and work-of-fracture of the composites with various fiber-coating thicknesses. Retained in-situ fiber strength in the fractured composites was evaluated by fractographic techniques. Retained in-situ fiber strength, ultimate strength, and work-of-fracture of the composites increased with increasing fiber-coating thickness, reaching a peak value at a coating thickness of $\approx 0.3 \mu\text{m}$. Further increases in coating thickness did not improve the mechanical properties of either the fibers or the composites. A direct correlation between retained in-situ fiber strength and mechanical properties of the composites suggests that retained in-situ fiber strength has a significant influence on the mechanical properties of composites.

Introduction

Continuous-fiber-reinforced ceramic matrix composites are becoming increasingly important 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. In order to produce composites with high toughness, it is essential to obtain optimal fiber/matrix interfacial characteristics and high fiber strength.⁴ Optimal fiber/matrix bond strength will lead to debonding and subsequent fiber sliding during fracture of composites. This results in substantial dissipation of elastic energy and hence improved fracture toughness. Interfacial characteristics can be tailored by appropriately coating the fiber surfaces.

Strength of reinforcing fibers 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. As observed in a recent study,⁵ the amount of fiber pullout, which contributes to the toughening of a composite, is strongly influenced by the mean strength of the reinforcing fibers. Also, the ultimate load-bearing capacity of the composite is determined by fiber strength characteristics.⁶ Because fiber strength is controlled primarily by critical flaws introduced during composite processing and service, application of fiber coatings can significantly decrease fiber damage and increase retained strength.

It is therefore clear that fiber coatings play an important role in establishing interfacial characteristics and fiber strength which are important parameters for the design and development of fiber-reinforced ceramic matrix composites with superior mechanical properties. In the present study, we have evaluated the effects of fiber coating on flaw generation, retained fiber strength, and resulting mechanical properties of chemical-vapor-infiltrated Nicalon fiber-reinforced SiC matrix composites.

Experimental Procedure

Seven sets of Nicalon-fiber-reinforced SiC matrix composites with approximately 42 vol.% fiber reinforcements were fabricated at Oak Ridge National Laboratory by densifying multiple layers of Nicalon mats stacked in a graphite die. Each set has a different carbon coating thickness ranging from 0 to 1.25 μm . Table 1 lists the specimen identification numbers and respective fiber-coating thicknesses. Chemical vapor infiltration (CVI), under forced conditions of thermal and pressure gradients, was used to densify the preforms with SiC. The resulting composites were nearly 90% dense. Details of composite specimen fabrication are described elsewhere.^{7,8}

The composites were fractured in a four-point-bend mode on the universal testing system[†]. Inner and outer loading spans were 15 and 30 mm, respectively. Typical dimensions of the flexure bar specimens were 3 x 4 x 32 mm. Flexure tests were conducted at a loading rate of 1.27 mm/min under ambient conditions.

Figure 1 shows a typical load-displacement plot obtained during flexural testing of composites with coated fibers. The first matrix cracking stress was determined from the load at which the first deviation from the linear variation in the load vs. displacement plots was observed. Ultimate stress was determined from the peak load value. In addition, composite work-of-fracture (WOF) was estimated from the area under the load-specimen displacement plots normalized on the basis of unit cross-sectional area of the fractured composites. True specimen displacement was obtained by subtracting system displacement from total displacement. System displacement was determined by measuring system compliance with a stiff alumina piece.

[†]Model 4505, Instron Corp., Canton, MA.

Table 1. Fiber Coating Thicknesses of CVI SiC/SiC Composites

Sample ID	Coating Thickness (μm)
571	0.0
526	0.03
522	0.06
538	0.13
524	0.27
549	0.61
521	1.25

Fractured composite specimens were examined on a scanning electron microscope[§] (SEM) to locate the failure origin and establish the associated characteristic fracture surface morphology of the fibers to estimate in-situ fiber strength.

Results and Discussion

Figure 2 shows ultimate strength as a function of fiber-coating thickness. The observed variations for the two cases are similar. Ultimate strength increases with coating thickness, reaching a peak value of 380 MPa at $\approx 0.2\text{-}0.6\ \mu\text{m}$ coating. Further increases in coating thickness do not significantly improve ultimate strength.

Work-of-fracture (WOF) as a function of coating thickness is shown in Fig. 3. The WOF increases rapidly with fiber-coating thickness, reaching a peak of 1.92×10^4

Figure 1. Typical Load-Displacement Plot for Composites with Coated Fibers.

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

Figure 2. Measured Variation of Ultimate Strength as a Function of Fiber Coating Thickness.

N-m/m² at a thickness of approximately 0.13-0.27 μm. Values did not change significantly with further increases in coating thickness.

SEM investigation on the fracture surfaces of composites reinforced with fibers with and without carbon coatings revealed differences in their fracture surface morphology, as shown in Fig. 4. The fracture surface shows brittle failure without any fiber pullout in composites with no fiber coating (Fig. 4a). On the other hand, substantial fiber pullout was seen in composites with coated fibers (Fig. 4b). These observations are consistent with load-displacement behavior for the two types of composites. In the coated fiber-reinforced composites, there was gradual decrease in load after the peak load was reached, as shown in Fig. 1. On the other hand, in the uncoated fiber-reinforced composites, there was an instantaneous drop at the peak load.

SEM was also used for fractographic evaluation to estimate in-situ fiber strengths. 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, tested in the four-point-bend mode, is shown in Fig. 5. 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 3. Measured Variation of Work-of-Fracture as a Function of Fiber-Coating Thickness.

It is well known that for glasses and ceramics, such fracture surface features as mirror radii can be correlated to tensile strength through the empirical relationship:^{9,10}

$$\sigma_f \sqrt{r_m} = A_m \quad (1)$$

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}, following the work of Thouless et al.⁵ Strengths of more than 30 Nicalon fibers for each set of composite specimens were determined by measuring their fracture mirror radii and using Eq. 1.

The strength distribution for fibers in the composites can be described using the Weibull function given by:

$$F(\sigma) = 1 - \exp \left[- \left(\frac{\sigma}{\sigma_0} \right)^m \right] \quad (2)$$

In Eq. 2, F is the fracture probability at a given stress σ , m is the Weibull modulus that characterizes the flaw size distribution in fibers, and σ_0 is the scale parameter signifying a characteristic strength value of the fibers.

Figure 4 Typical Fracture Surface of Composites Reinforced With
(a) Uncoated Fibers and (b) Coated Fibers

Figure 5. Micrograph Showing Surface Morphology of Fractured Fiber in Composite.

Figure 6 shows in-situ fiber strength as a function of coating thickness. The measured scale parameters depend on gage length, which is approximately equal to the fiber pullout lengths. An evaluation of fiber pullout lengths indicated no significant difference as a function of fiber coating thickness. Therefore, no additional modification to account for variation in pullout length was needed. It can be clearly seen in Fig. 6 that fiber strength initially increases with coating thickness and reaches a peak value at a coating thickness of $\sim 0.13\text{--}0.6\ \mu\text{m}$. Further increases in coating thickness have no significant effects on fiber strength. It is believed that the initial increase in strength is due to fiber protection by coating, which minimizes fiber surface damage during processing and fabrication. Further increases in coating thickness do not increase the effectiveness of coating in protecting fiber damage. This result has very important implications for determining optimal fiber-coating parameters and composite processing.

A comparison of Figs. 3 and 4 with Fig. 5 shows a direct correlation between in situ-fiber strength and ultimate strength and WOF of the composites with various coating thicknesses. This suggests that in-situ fiber strength has a significant influence on the ultimate strength and WOF of the composites. In addition, fiber coating may also be partly involved in improved fiber/matrix interfacial characteristics, leading to the observed increase in both ultimate strength and WOF.

Conclusions

1. Processing-induced damage of Nicalon fibers was minimized by coating the fibers with carbon. This resulted in a corresponding increase in retained in-situ strength of the fibers during composite processing.
2. Mechanical properties (ultimate strength and work of fracture) of the composites increased with increasing fiber-coating thickness up to a thickness of $\sim 0.3 \mu\text{m}$. Further increases in coating thickness had no significant effects on mechanical properties, indicating an optimal coating thickness for processing.
3. Dependence of in-situ fiber strength, ultimate strength, and work of fracture on fiber-coating thickness suggests a direct correlation between retained in-situ fiber strength and resulting mechanical properties.
4. Increased ultimate strength and work of fracture with increasing fiber-coating thickness may also be partly due to improved fiber/matrix interfacial characteristics.

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Figure 6. Variation of In-Situ Fiber Strength with Fiber Coating Thickness.

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