

Properties and microstructure of Nextel 720/SiC composites

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Abstract

Continuous Nextel 720 fibers reinforced SiC composites were fabricated by CVI at 1100 °C for 200 h using SiCH₃Cl₃ as precursor. The mechanical properties were measured by three-point bending. The microstructures of the interface and fracture surface were characterized by SEM. The values of the Nextel 720/SiC for mean strength and Yong's modulus were 316 MPa and 211 GPa, respectively. The composites had a high failure strain of 0.25%. The multi-layer PyC interface and the crevices between the fiber and the matrix resulted in a metal-like fracture behavior.

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1. Introduction

Fiber reinforced ceramic matrix composites have been widely investigated as high-temperature structural materials because of their low density, metal-like fracture model, high resistance to erosion and corrosion [1]. C/C composite and SiC ceramic matrix composites (C/SiC, SiC/SiC etc.) are the leading candidate materials for high-temperature application for their excellent mechanical properties, such as high-temperature strength and low-creep rate. However, C fiber is susceptible to long-term degradation after high-temperature exposure to oxidizing environment [2]. SiC/SiC composites are an attempt to overcome the deficiencies of C/SiC by replacing the carbon fiber reinforcement with SiC fibers. To date SiC/SiC composites are limited to operating temperatures of about 1200 °C because of degradation of the SiC fibers. Although High-Nicalon SiC fiber has excellent properties at high-temperature, it is very expensive. This has motivated the search for continuous oxide fibers reinforced SiC ceramic matrix composites, which can combine the other outstanding composite properties with long-term oxidation stability [3].

More recently, the commercially available Nextel series of fibers produced by 3 M has received much attention. These fibers are composed of Al₂O₃ with varying amounts of SiO₂ for grain size control and the promotion of mullite crystallization for improved creep resistance and stress-rupture [4]. Nextel 720 fiber has both a secondary phase and elongated grains incorporated into its microstructure. The (~55 vol.%) mullite is present primarily as needles surrounding the Al₂O₃ grains (~45 vol.%). Thus, the creep resistance of the Nextel 720 is better than other oxide fibers [5]. SiC has excellent oxidation-resistance for the creation of dense SiO₂ film on the SiC surface during oxidation. CVI (Chemical vapor infiltration) is a near-net shape and flexible process, which can be applied to preforms of complex and different shapes. CVI SiC has been developed to fabricate 3D C/SiC composites at 1100–1300 °C. The main objective of this study is to fabricate Nextel 720/SiC composites using CVI, and investigate the microstructure and mechanical properties.

2. Experimental

3D preforms were formed from Nextel 720 fiber produced by the 3 M Corporation (St. Paul, MN). Each

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tow nominally contains 750 fibers. The strength of the Nextel 720 fiber is about 30% less than the Nextel 610 fiber for single filament properties, but it was selected because of its greater creep resistance.

As detailed below, 3D preform was infiltrated with $C_2H_2=C_2H_2$ precursor by CVI to fabricate a PyC interfacial layer. The preform coated with PyC was densified by CVI at 1100 °C for 200 h using $SiCH_3Cl_3$ as precursor. Argon was used as carrier gases. Hydrogen was used as reactive gas. All gas streams were controlled by mass flowmeter. The fibers volume fraction was 50%, equally distributed in the three rectangular directions.

Microstructure was examined by scanning electron microscopy (SEM, JEOL JXA-840). Flexure strength, flexure modulus, and fracture work of the composites at room temperature were determined by three-point bending test on an Instron Universal Tester at a displacement rate of 0.2 mm/min and 40 mm span according to British Standard Methods of Testing (BS 2782: Part 10: Method 1005, EN631977). The rectangular test specimens cut from CVI board composite samples were ground and polished to 50 mm in length, 4.0 mm in width and 3.0 mm in thickness. The load–deflection curve for the composites was recorded to fracture. The failure mechanism was identified by visual examination after the test and by SEM examination of the fracture surface. The fracture work was calculated from the area under the load–deflection curve.

3. Results and discussion

Fig. 1 shows a typical load–deflection curve for as-received Nextel 720/SiC composites. The values for mean strength and Yong's modulus are 316 MPa and 211 GPa, respectively. The fracture work is 320 MPa, higher than Nextel 720/mullite composites [6].

During the three-point bending test, most of the composites initially showed an elastic response with deflection increasing linearly with increase in load, followed by an extended un-elastic regime. It can be seen that the failure was not catastrophic. The curve indicated a linear elastic behavior up to the proportional limit of 250 MPa. It

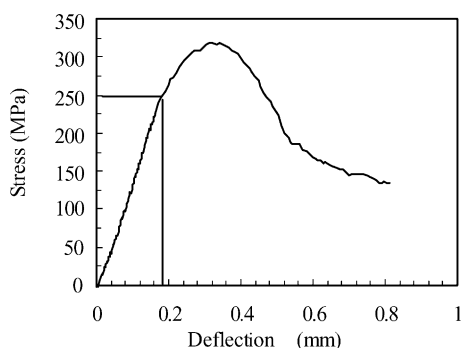


Fig. 1. Typical load vs. deflection plots tested by three-point bending.

was observed that the specimen did not fall apart after the load was removed.

Generally after the maximum value of load was reached, the degree of the subsequent extension was strongly dependent on the nature of the fiber/matrix interface. The Nextel 720/SiC composite exhibited a good composite behavior, which was, it failed gracefully with extensive fiber pull-out. In general, the microstructure is consistent with the mechanical properties. The improved toughness is obtained from a microstructure where the fibers can absorb energy of stress concentrations by bridging crack surfaces and dissipating energy as the matrix disintegrates during fiber pull-out.

The nature of the fiber/matrix interface can be seen by examining the fracture surface of the composite specimens after the three-point test. Fig. 2 shows SEM micrograph of PyC interface for the as-received Nextel 720/SiC composites. A sharply defined interface was observed between the fiber and the matrix. The SEM micrograph indicates the thickness of PyC interface to be about 200 nm and the PyC interface to be multi-layer. Some crevices occurred along the interface, which would further weaken the interfacial bonding. The crevices may be the result of a relative displacement between fibers and matrix on cooling from the high processing temperature to room temperature because of the thermal expansion mismatch. The thermal expansion coefficient of Nextel 720 fibers is $6.0 \times 10^{-6} K^{-1}$ and that of the CVD SiC obtained by chemical vapor deposition is $3.3\text{--}5.8 \times 10^{-6} K^{-1}$.

Fig. 3 shows a SEM micrograph of the fracture surface of the Nextel 720/SiC composite after three-point test. Extensive fiber pull-out was observed in the fracture surface of the composite. The influence of the PyC structure on fiber pull-out should also be considered. From Fig. 2, some crevices may be detected along the interfacial region between the Nextel fiber and the SiC matrix. The crevices could contribute to the weak interfacial bonding, resulting in extensive fibers pull-out on failure with a substantial failure strain of 0.25%.

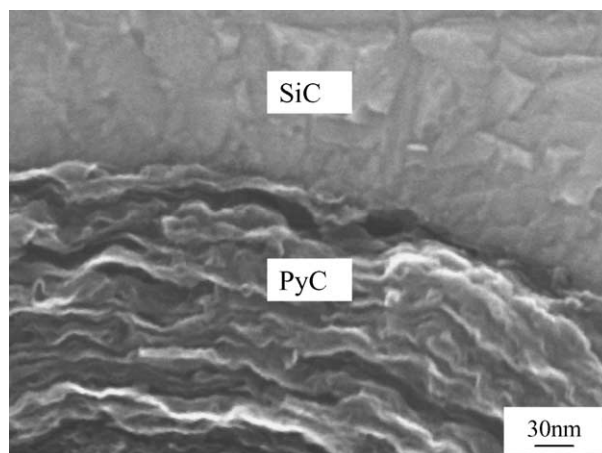


Fig. 2. SEM micrograph of PyC interface.

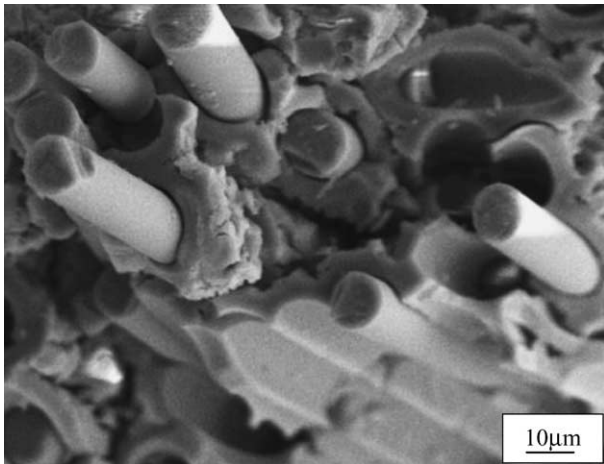


Fig. 3. SEM micrograph of fracture surface of composite after three-point bending test.

4. Conclusions

Continuous Nextel 720 fibers reinforced SiC composites were successfully fabricated at 1100 °C by CVI using SiCH_3Cl_3 as precursor. The values for mean strength and Young's modulus were 316 MPa and 211 GPa, respectively. The composites possessed a high failure strain of 0.25%. The multi-layer PyC interface and the crevices between the

fiber and the matrix resulted in a metal-like fracture behavior.

Acknowledgments

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