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High performance 3D textile Hi-Nicalon SiC/SiC composites by chemical vapor infiltration

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Abstract

Three dimensional textile Hi-Nicalon silicon carbide fiber reinforced silicon carbide composites with high toughness and reliability were fabricated by chemical vapor infiltration. The mechanical properties of the composite materials were investigated under bending, shear, and impact loading. The density of the composites was 2.5 g cm^{-3} after the three dimension silicon carbide perform has been infiltrated for 30 h. The values of flexural strength were 860 MPa at room temperature and 1010 MPa at 1300°C in vacuum. Above the infiltration temperature, the failure behavior of the composites became brittle because of the strong interfacial bonding and the mis-match of thermal expansion coefficients between fiber and matrix. The obtained value of shear strength was 67.5 MPa. The composites exhibited excellent impact resistance and the value of dynamic fracture toughness is 36.0 kJ m^{-2} was measured with Charpy impact tests.

Keywords: C. Mechanical properties; 3D SiC/SiC composites; Chemical vapor infiltration

1. Introduction

Continuous fiber reinforced ceramic matrix composites (CFCCs) show superior performance when superalloy at elevated temperatures and higher toughness when compared with monolithic ceramics [1–4]. For these reasons, CFCCs have the most potential to be used in advanced aero-engines, space, and fusion power reactors [1–4]. Among these CFCCs, silicon carbide fiber reinforced silicon carbide composites (SiC/SiC) are promising and have received considerable attention. Many investigations have been conducted on one dimension (1D) and two-dimension (2D) woven SiC/SiC composite materials [5–12]. Recently, attention has been focused on three-dimension woven or braided ceramic matrix composite materials in order to meet mechanical and thermal properties requirements under most complex loads [13–18].

The present research involved 3D textile Hi-Nicalon SiC/SiC composites prepared by chemical vapor infiltration and examined the mechanical properties over the temperature range from room temperature to 1300°C. The aims of the current contribution are to develop an understanding of the architecture on the mechanical properties and the damage behavior of the 3D Hi-Nicalon SiC/SiC composites and to expand the experimental knowledge for the three dimensional textile composite materials.

2. Materials and experimental procedures

2.1. Fabrication of the composites

Hi-Nicalon SiC fiber was employed and each yarn contained 500 filaments. The three dimensional (3D) fabric perform was braided by four-step processing and supplied by Nanjing Institute of Glass Fiber in China. The structure of the preform is illustrated in Fig. 1. The fiber volume fraction was 40%. In the present experiment, chemical vapor infiltration was employed to deposit a pyrolytic carbon 1ayer and silicon carbide, which has been described previously in detail [14,15]. A thin carbon layer as the interfacial layer was deposited on the surface of the Hi-Nicalon SiC fiber with butane

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(C_3H_6) prior to densification. Methytrichosilane (MTS, CH_3SiCl_3) was used for deposition of SiC and carried by bubbling hydrogen (H2). Typical conditions used for the densification of silicon carbide matrix are 1100°C, a hydrogen to MTS mol ratio of 10, and a pressure of 2–3 kPa. Argon(Ar) was employed as a diluent gas to slow down the chemical reaction rate of deposition.

2.2. Mechanical properties measurement

Mechanical properties of the composite materials were characterized under flexural shear, and impact loading. Flexural strength was measured with a three-point-bending method at temperatures ranging from room temperature up to 1300°C in vacuum. Shear strength was measured by the short beam bending method with a span of 15 mm. Fracture toughness was determined with single edged-notched beam method. The impact tests were performed with an instrumented Charpy equipment for the test. The sample size was $3.0\times20\times70$ mm, and the impact velocity of 3 m s^{-1} was imposed for the test.

2.3. Microstructure observation and surface analysis

The density of the samples was determined by the water displacement method. The microstructure of the fracture surface was observed by a scanning electron microscope.

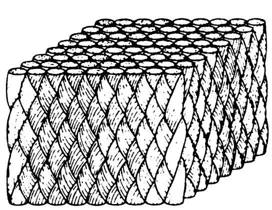

Fig. 1. Structure of a three dimension preform.

3. Results and discussion

3.1. Flexural loading

The density of the composites was 2.5 g cm^{-3} after the three dimensional silicon carbide preform was chemical vapor infiltrated for 30 h. Fig. 2a showed the typical failure behavior of 3D Hi-Nicalon SiC/SiC textile composites at room temperature. The mechanical behavior was initially linear elastic. Then, a non1inear region was observed, reflecting matrix damage which induced significantly compliance, and residual displacement. Finally, the fiber failed, initiating at the maxim load, causing the unstable fracture of the composites. As the temperature was increased from room temperature to 1300°C, the flexural strength of the 3D Hi-Nicalon SiC/SiC composites was slightly increased but not decreased. The average values of the flexural strength were 920 MPa at room temperature and 1010 MPa at l300°C in vacuum. The failure behavior of the composites changed with increase of the temperature. At room temperature the stress drop was very gradual after the maximum stress point. However, the failure behavior became brittle and the composites exhibited steep stress drops after the maximum stress point at high temperatures (Fig. 2b).

The variation of failure behavior of the composites was attributed to the interfacial bonding between fiber and matrix. Fig. 3 showed the typical microstructure of the Hi-Nicalon SiC/SiC composite materials. The pyrolysis carbon interfacial layer was very uniform and the thickness was 300 nm. It was this layer that ensured the proper interfacial bonding between fiber and matrix as well as the load transfer from the silicon carbide matrix to the Hi-Nicalon SiC fiber. Moreover, it has been reported that the thermal expansion coefficients of the silicon carbide matrix and the Hi-Nicalon fiber were 2.2×10^{-6} K^{-1} and 5.3×10^{-6} K^{-1}, respectively [19–21]. After the composites were cooled down from the infiltration temperature to room temperature, a tensile stress was generated cross the interfacial layer. As a result, it was easy for the

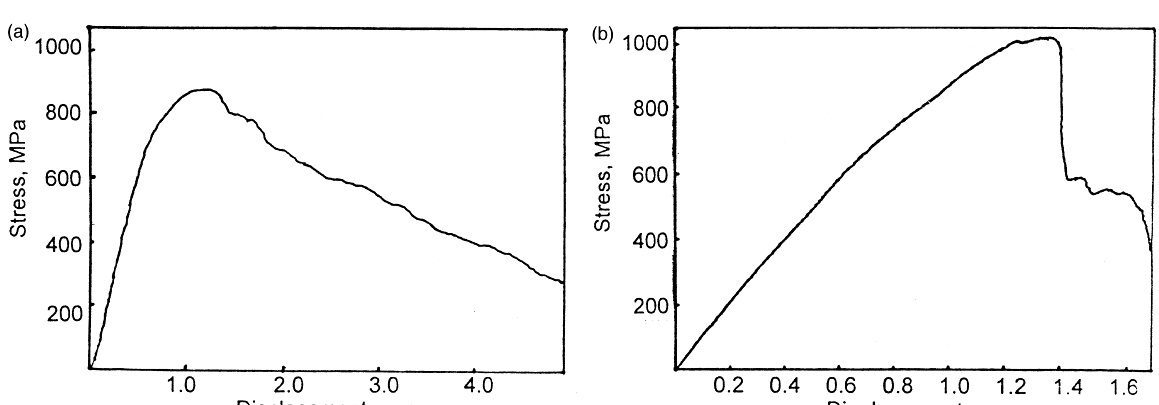

Fig. 2. Stress–deflection curve under flexural loading.

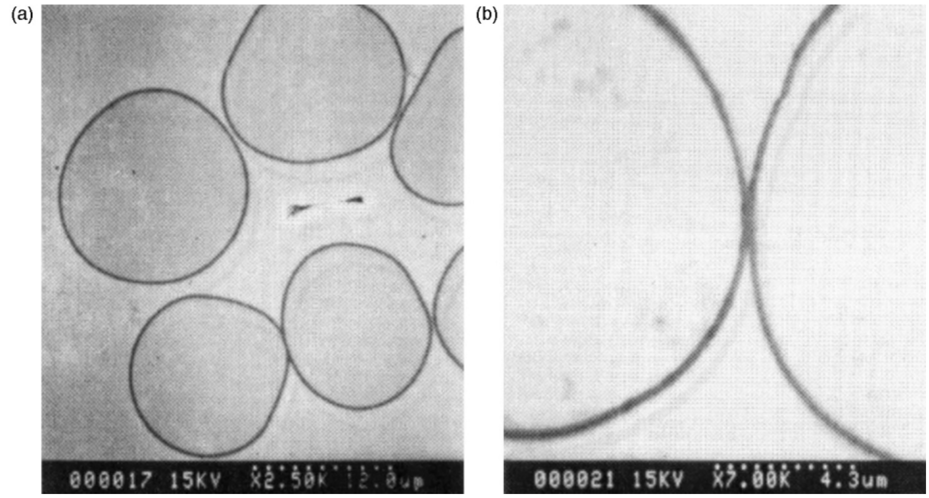

Fig. 3. Microstructure of 3D Hi-Nicalon SiC/SiC composites.

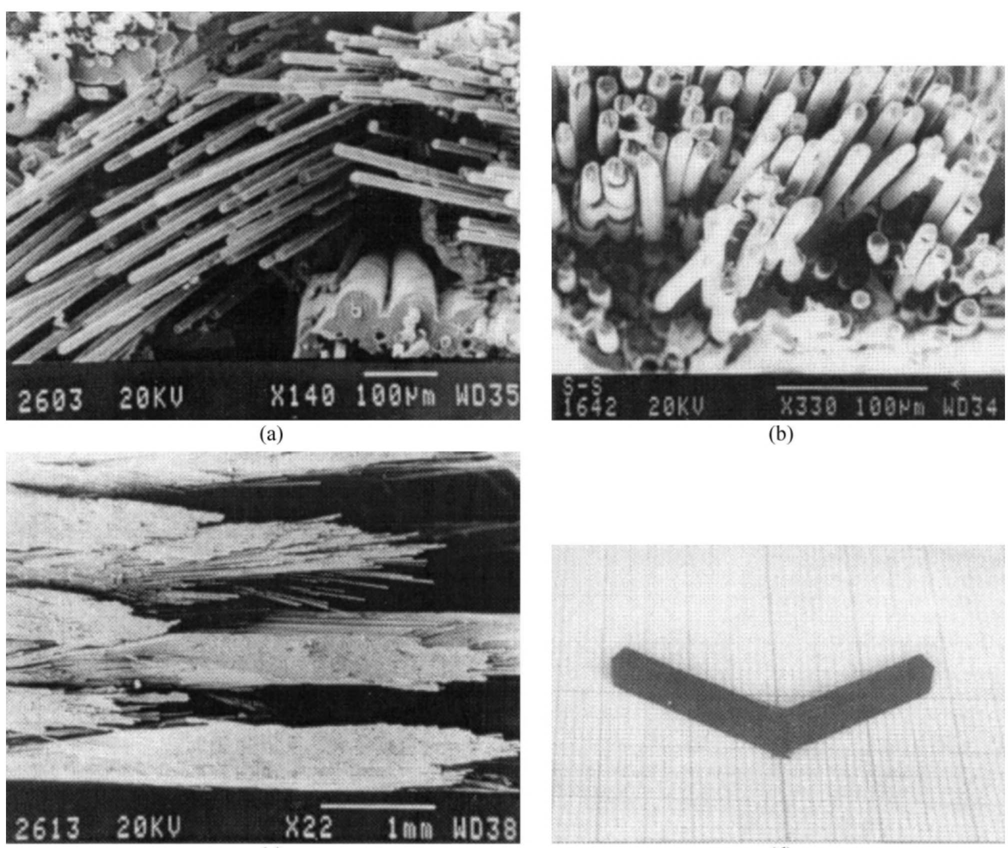

Fig. 4. Fracture surface of 3D Hi-Hicalon SiC/SiC composites.

fiber to be pulled out from the silicon carbide matrix. If the temperature was increased above the silicon carbide infiltration temperature (1100°C), a compressive stress was created across the interfacial layer. Hence, the fiber was very difficult to pull out. The pull-out length at l300°C was much shorter than at room temperature, as shown in Fig. 4a,b. For the three dimensional texti1e CFCCs, the bundle/bundle interfacial bonding is usually considered as a kind of weak interfacial bonding because of the pores residual in the composites caused by the ‘‘bottom neck effect’’ during the chemical vapor infiltration process. Accordingly, fiber bundle pullout was a]ways observed at both room temperature and high temperature (Fig. 4c,d).

3.2. Failure behavior under shear loading

The shear strength of the 3D Hi-Nicalon SiC/SiC composites was measured by the short bending beam method, and the span was 15 mm. Shear strength was calculated by the following equation:

$$\tau = \frac{3p}{4bh} \tag{1}$$

where p is the maximum fracture load (N), b and h are the width and height of the sample respectively.

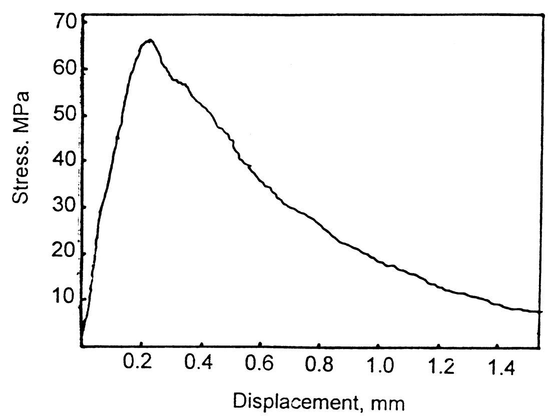

Fig. 5. Stress–deflection curve under shear loading.

The shear failure behavior of the stress–deflection curve was similar to that of bending failure behavior and shown in Fig. 5. The obtained value of shear strength was 67.5 MPa which is much higher than that of 3D C/SiC textile composites. Different from two-dimensional CFCCs and the other laminated composites, inter1ayer debonding was not observed in the present composites. The results indicated that the present 3D Hi-Nicalon SiC/SiC composites exhibited good shear resistant and isotropic properties.

3.3. Failure behavior of notched specimen under flexural loading

In order to determine the fracture toughness of the composites, samples were notched and tested in a three-point-bending sample with a span of 40 mm and a loading rate of 0.05 mm mm^{-1}. The width of the notch was 0.01 mm. The value of fracture toughness (K_{1c}) was calculated by using of the following expression:

$$K_{1c} = \frac{3pl}{2bw^2}\left(1.93 - 3.07\frac{a}{w} + 1.45\left(\frac{a}{w}\right)^2 - 25.07\left(\frac{a}{w}\right)^3 + 25.80\left(\frac{a}{w}\right)^4\right) \tag{2}$$

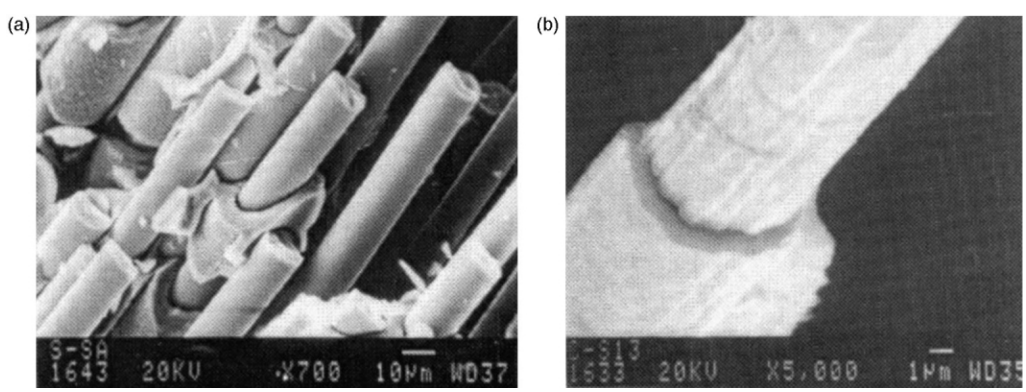

Fig. 6. Microstructure differences between (a) Hi-Nicalon SiC/SiC and (b) C/SiC composites.

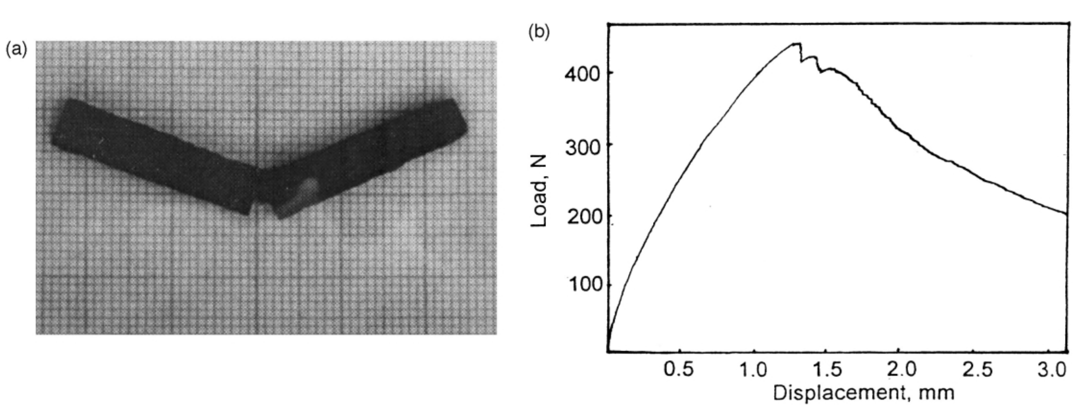

Fig. 7. Failure behavior of the 3D Hi-Nicalon SiC/SiC composites with a notch.

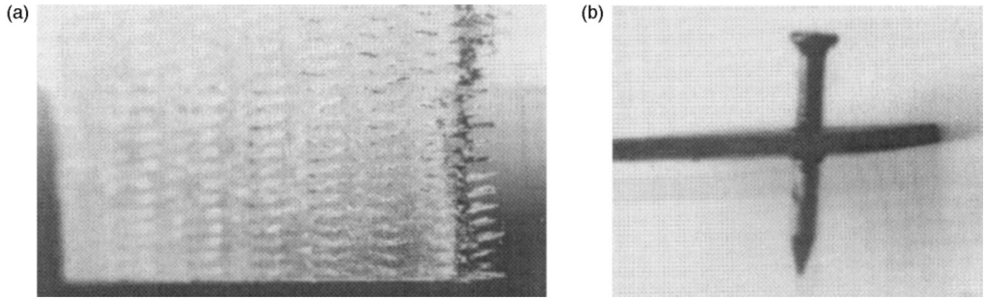

Fig. 8. Impact fracture surface of Hi-Nicalon SiC/SiC composites.

where p is the fracture load, l is the span, and a is the notch depth, b and w are thickness and width of the simp1e respectively.

The fracture toughness calculated from Eq. (2) was 41.5 MPa $m^{1/2}$, which was 10 times that of monolithic ceramic materials (3–5 MPa $m^{1/2}$) and two times that of 3D C/SiC composites (20.3 MPa $m^{1/2}$)[22]. The fracture toughness differences between 3D C/SiC and 3D Hi-Nicalon SiC/SiC composites could be i1lustrated from the microstructure difference of these two kinds of materials. In Fig. 6, it was observed that the surface of Hi-Nicalon SiC fiber was much smoother than that of T300 carbon fiber. Consequently, it was very easy for Hi-Nicalon SiC fiber to be pulled out from the SiC matrix, leading to higher fracture toughness.

Here, the work of fracture was introduced to represent the toughness of the 3D Hi-Nicalon SiC/SiC textile composite materials. The work of fracture was obtained from the characteristic area under the load-displacement curve divided by the cross-section of the specimen. In order to determine the work of fracture effectively, we defined the characteristic area (Ac) which started from the initial point to the 10% drop of the curve (Fig. 7). This gives an average work of fracture as high as 28.1 kJ m^{-2}, which is the nearly three times that of 3D C/SiC composites and six times that of laminated SiC ceramic matrix composites (4625 J m^{-2}), respectively [22,23].

3.4. Impact loading

Instrumented Charpy impact tests on un-notched samples were conducted to determine the energy absorbing capability and dynamic fracture behavior of the composite materials. The dynamic fracture toughness (α_k) was calculated by using the following equation:

$$\alpha_k = \Delta w/bh \quad (3)$$

where w is the absorbing energy of materials during impact processing, b and h are the thickness and width of specimen, respectively.

The value of α_k is 36 kJ m^{-2} for 3D Hi-Nicalon SiC/SiC composite materials, and is lower than that of super-alloy (α_k = 80–160 kJ m^{-2}). The impact fracture surface was brush-1ike in Fig. 8a. It was very interesting to observe that the present composite materials could withstand the hitting impact of a steel nail (Fig. 8b). These results revealed that the 3D textile Hi-Nicalon SiC/SiC composites exhibited the excellent resistance against dynamic impact.

4. Conclusions

High performance three dimensional textile Hi-Nicalon SiC fiber reinforced silicon carbide composites were fabricated by chemical vapor infiltration. The density of the composites was 2.5g cm^{-3} after the three-dimensional carbon preform was infiltrated for 30 h. The values of flexural strength were 860 MPa at room temperature and 1010 MPa at 1300°C in vacuum. Above the infiltration temperature, the failure behavior of the composites became brittle because a compressive stress was generated cross the interfacial 1ayer caused by the mismatch of thermal expansion coefficients between fiber and matrix. The obtained value of the shear strength was 67.5 MPa. The fracture toughness and work of fracture were as high as 41.5 MPa $m^{1/2}$ and 28.1 kJ m^{-2} respectively. The value of dynamic fracture toughness was 36.0 kJ m^{-2}.

Acknowledgements

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