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Directionality effect on mechanical properties of 3D *n*-directional braided $(SiO_2)_f/SiO_2$ composites prepared by silica sol-infiltration-sintering method

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Abstract

Three-dimensional (3D) and *n*-directional (*n* = 4, 5, 6 and 7) woven $(SiO_2)_f/SiO_2$ composites were prepared by silica sol-infiltration-sintering (SIS) method. All the woven reinforced structures were of the same fiber content, and the prepared samples were cut in the same manner parallel to the respective braiding direction. The effect of *n*-direction on mechanical properties has been studied by conducting tensile, flexural and shear tests. The results showed that flexural and shear strengths of the samples increased gently with increasing *n*-direction from 4 to 7. However, with increasing *n*-direction, tensile strength initially increased steeply for 4–5 *n*-directions, followed by a gradual decrease for 6–7 *n*-directions. 3D 5-directional sample had the maximum tensile strength while 3D 7-directional sample had the maximum flexural strength and shear strength.

Keywords: C. Mechanical properties; Silica composites; *n*-Direction; Silica sol-infiltration-sintering (SIS)

1. Introduction

Continuous fiber reinforced ceramic matrix composites (CFCMCs), such as C_f/SiC and SiC_f/SiC systems, are considered as potential materials to be used in advanced aero-engines, space and fusion power reactors, due to their higher surface strength at elevated temperatures and higher fracture toughness compared with metals and other monolithic ceramics [1–4]. Silica has high melting point, high thermal shock resistance and excellent ablation resistance as well as low thermal conductivity, and has received considerable attraction in the field of radar materials [5]. However, low flexural strength and low fracture toughness have restrained the use of monolithic silica for many applications. In order to improve the mechanical properties of SiO_2, several efforts [6–12] have been made to reinforce SiO_2 matrix by adding shorter silica fibers, longer silica fibers, 2.5D silica braided and conventional 3D silica braided reinforcements. The mechanical properties of $(SiO_2)_f/SiO_2$ composites are closely related to the woven structure of the reinforcements and the preparation method [13–15]. However, there are hardly any reports on mechanical properties versus *n*-direction of 3D woven $(SiO_2)_f/SiO_2$ composites.

In this paper, 3D *n*-directional (*n* = 4, 5, 6 and 7) woven $(SiO_2)_f/SiO_2$ composites were prepared using SIS method, which includes sol–gel processing, vacuum infiltration and sintering processing. SIS method is a straightforward and convenient process to achieve uniform distribution of materials that can be formed into complex shapes. The mechanical properties of the composites were investigated by tensile, flexural and shear tests. The aim of this paper is to understand the effect of woven structure on the mechanical properties of $(SiO_2)_f/SiO_2$ composites.

2. Materials and methods

The 3D *n*-directional (*n* = 4, 5, 6 and 7) woven silica reinforcements were provided by Nanjing Institute of Glass Fiber. The specifications of the yarns were: B type silica fiber (190Tex) and the fiber content was 47 ± 1% for all the 3D *n*-directional woven silica reinforcements. Fig. 1 is the representative unit structure of 3D *n*-directional woven structure. Fig. 1(a) is a 3D 4-directional braided unit, which is composed of four directional yarns which have the same

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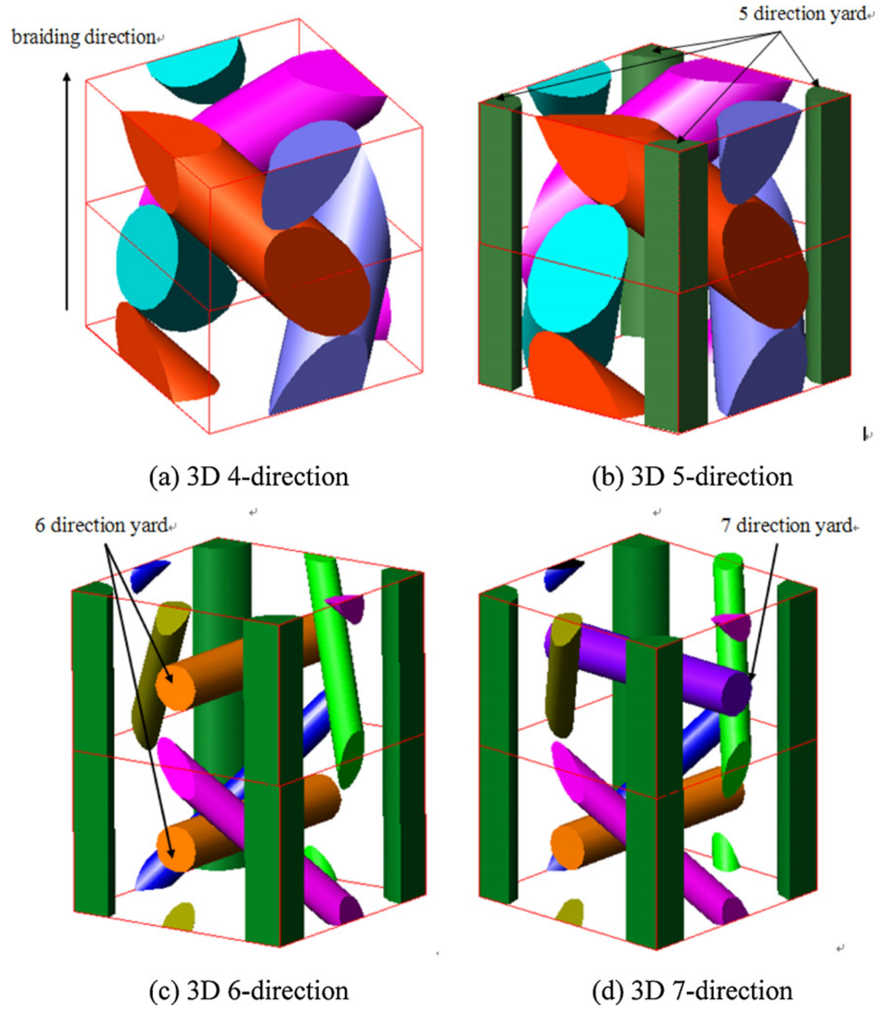


Fig. 1. Representative units of 3D *n*-directional woven structure: (a) 3D 4-direction, (b) 3D 5-direction, (c) 3D 6-direction and (d) 3D 7-direction.

angle between the braiding directions. Fig. 1(b) is a 3D 5-directional braided unit, which adds one more uniaxially reinforced yarn along the braiding direction compared with that of 3D 4-directional braided unit. Fig. 1(c) is a 3D 6-directional braided unit, which adds one more uniaxially reinforced yarn vertically along the braiding direction compared with that of 3D 5-directional braided unit. Fig. 1(d) is a 3D 7-directional braided unit, which adds one more uniaxially reinforced yard vertically along the braiding direction compared with that of 3D 6-directional braided unit.

The $(SiO_2)_f/SiO_2$ composites were fabricated by silica sol-infiltration-sintering method, as shown in Fig. 2. To start with, the 3D, *n*-directional braided silica reinforcements were placed in a closed container and the container was evacuated to 0.1 Pa. Then, highly pure silica sol of average particle size 10 nm and pH 9 (supplied by Ningbo Company, China), was sucked into the container at a volume ratio of 35%; during this process, the pressure in the container was maintained at 0.1 Pa. Afterwards, the container was pressurized to 10 atm and maintained at that high pressure for 1 h. The composites were then dried at 80 °C for 1 h and subsequently at 110 °C for 1 h to gradually reduce the water content of the gel solution, followed by sintering at 450 °C for 2 h to remove the coupling agent and bounded water. The process was repeated for 10 cycles to obtain the final $(SiO_2)_f/SiO_2$ composite.

Specimens for tensile and flexural tests were cut parallel to the respective braiding directions and then ground into bars of 40 mm in length, 5 mm in width and 3.5 mm in thickness, as shown in Fig. 3(a) and (b). Tensile and three-point bend tests were conducted with an Iosipescu Universal tester (SANS CMT5105 electronic universal testing machine) at a displacement rate of 0.1 mm/min and 30 mm span according to British Standard Methods of Testing. The holder was reinforced by aluminum plates. During the tensile test, the applied force was aligned parallel to the braiding direction of the composite, where as for the flexural and shear tests the applied force was aligned perpendicular to the braiding direction of the composite. The load versus deflection data were recorded until fracture. The fracture strength was computed from the load deflection data. The shear strength was also measured by the Iosipescu shear testing method, and the loading rate was 0.3 mm/min, as shown in Fig. 3(c). Microstructure and fracture mechanism were examined by scanning electron microscopy (JEOL JSM-6360). The specimens for SEM were gold coated.

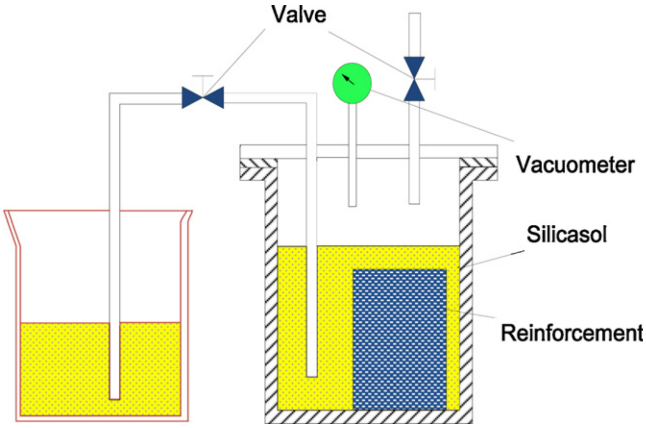


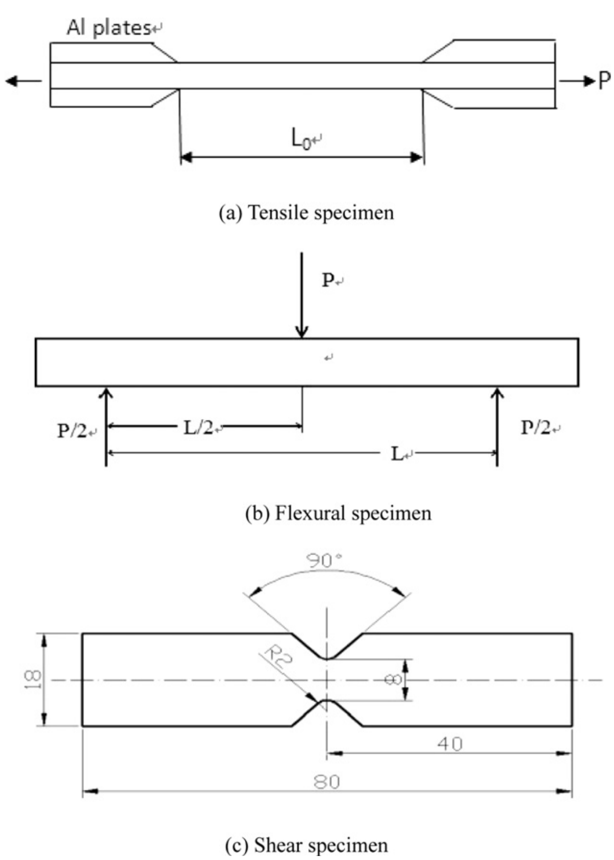
Fig. 2. Schematic diagram of silica sol-infiltration method.

3. Results and discussion

The specimen density was determined using the Archimedes principle according to ASTM specification D792-00 [16]; that is

$$\rho = \frac{m_a}{m_a + m_p - m_b} \rho_w \quad (1)$$

where ρ is the density of the composite material, m_a is the weight of the specimen when in air, m_p is the weight of the partly immersed wire holding the specimen, m_b is the weight of the specimen when immersed fully in distilled water, along with the partly immersed wire holding the specimen, and ρ_w is the density of the distilled water at testing temperature.



(a) Tensile specimen

(b) Flexural specimen

(c) Shear specimen

Fig. 3. Specimen size for mechanical tests: (a) tensile specimen, (b) flexural specimen and (c) shear specimen.

Table 1
Density of 3D *n*-directional braided samples.

	3D *n*-direction			
	4	5	6	7
Density (g/cm^3)	1.71	1.73	1.75	1.76

The density of 3D *n*-directional braided samples is shown in Table 1. The density of 3D *n*-directional braided samples was found to be in the range of 1.71–1.76 g/cm^3, which can be understood because the SIS process was the same and was repeated for 10 cycles to obtain the final composite. The density was slightly enlarged with the increase of *n*-direction from 4 to 7, which may be caused by the differences in woven structures.

Table 2 is the mechanical properties of 3D *n*-directional woven $(SiO_2)_f/SiO_2$ composites. It can be seen that the mechanical properties are closely related to the woven structure of reinforcements. The following can be deduced:

(1) The tensile strength initially increased steeply from 30.8 MPa to 38.1 MPa for 4–5 *n*-directions and then dropped gradually from 33.7 MPa to 29.5 MPa for 6–7 *n*-directions. 3D 5-directional sample and 3D 7-directional sample had the maximum and minimum tensile strengths respectively.
(2) The flexural strength monotone increased gently from 64.0 MPa to 107.0 MPa with increasing *n*-direction. 3D 7-directional sample had the maximum flexural strength of 107.0 MPa.
(3) The shear strength monotone also increased gently from 22.0 MPa to 30.2 MPa with increasing *n*-direction. Again, 3D 7-directional sample had the maximum shear strength of 30.2 MPa.

The tensile strength of composites is closely dependent on the fiber content which is aligned in the direction of an applied force. Although the total volume fraction of fiber of all the composites was almost the same, the fiber content in the direction of the tensile force is different. Normally, the fiber content in one direction will decrease with an increase of *n*-direction, while the total volume fraction of the fiber is fixed. In our samples, the direction of applied force is aligned in the braiding direction as shown in Fig. 1(a)–(d). Consequently, the 5-directional yard will play an important role in the tensile

Table 2
Mechanical properties of 3D *n*-directional woven $(SiO_2)_f/SiO_2$ composites.

3D *n*-directional	Tensile strength (MPa)	Flexural strength (MPa)	Shear strength (MPa)
4	30.8	64.0	22.0
5	38.1	72.7	24.2
6	33.7	94.8	28.6
7	29.5	107.0	30.2

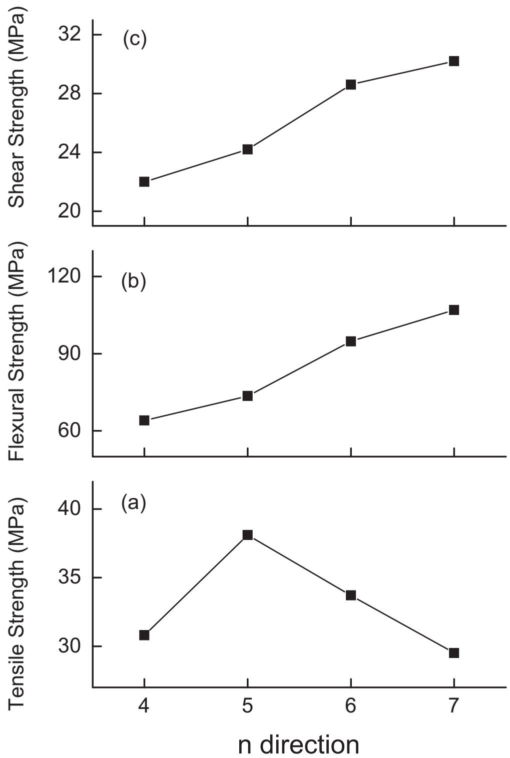


Fig. 4. Mechanical properties versus *n*-direction of 3D woven $(SiO_2)_f/SiO_2$ composites: (a) tensile strength, (b) flexural strength and (c) shear strength.

strength, causing the tensile strength of 3D 5-directional sample to be higher than that of 3D 4-directional sample. With increasing *n*-direction, the fiber content in the braiding direction was reduced, causing the tensile strength of 3D 6-directional sample to be lower than that of 3D 5-directional sample. When the *n*-direction was increased to 7, the fiber content in the braiding direction became the minimum, causing the tensile strength of 3D 7-directional sample to be the minimum, as shown in Fig. 4(a).

The flexural and shear strengths were monotone increasing with increasing *n*-direction, and their graphical representations showed two curves of similar shape, as illustrated in Fig. 4(b) and (c). During the flexural and shear tests, the applied force was aligned perpendicular to the braiding direction of the composite. Micro cracks during flexural and shear tests will break through the whole cross-section of sample, therefore the flexural strength and shear strength closely depends on the whole properties of samples. Even though the whole volume fraction of the fiber remained the same, the structure of braided unit became more complex with increasing *n*-direction, and the yarns became solidly interlaced with each other in space. In addition, density of the composites also increased slightly from 1.61 to 1.66 g/cm^3 with increasing *n*-direction, which also enhanced the whole properties of samples. Thus, the 3D 7-directional sample had the maximum flexural strength and shear strength.

All the samples were prepared by the same process and the volume fraction of the fibers in the reinforcements were fixed. Therefore, the mechanical properties were mainly affected by the woven structure of the reinforcements. The 3D 5-directional sample had the maximum tensile strength because the applied force during the tensile test was aligned parallel to the braiding direction and the 5-direction yard played an important role. 3D 7-directional samples had the maximum flexural and shear strengths because these properties are dependent on the whole properties of samples.

4. Conclusions

In this paper, 3D *n*-directional ($n = 4$, 5, 6 and 7) woven structure $(SiO_2)_f/SiO_2$ composites were prepared by silica sol-infiltration-sintering (SIS) method. It was experimentally deduced that, the mechanical properties were strongly affected by the woven structure of the reinforcements. 3D 5-directional sample had the maximum tensile strength; its 5-direction yard, cross enhanced the sample against the applied force during the tensile test, which was aligned to the braiding direction and the 3D 7-directional samples exhibited the maximum flexural and shear strengths because such properties were dependent on the whole property of the respective samples.

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