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Mechanical properties and microstructure of 2.5D (shallow straight-joint) quartz fibers-reinforced silica composites by silicasol-infiltration-sintering

Yong Liu [a], Jianxun Zhu [b,*], Zhaofeng Chen [a], Yun Jiang [b], Chengdong Li [a], Binbin Li [a], Long Lin [a], Tianru Guan [a], Zhaohai Chen [a]

[a] *College of Material Science and Technology, Nanjing University of Aeronautics and Astronautics, 29#, Jiangjun Road, Nanjing 211106, PR China*
[b] *Sinoma Science & Technology Co. Ltd., Nanjing 210012, PR China*

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Abstract

2.5D (shallow straight-joint) quartz fibers-reinforced silica composites were successfully prepared by silicasol-infiltration-sintering method. The composites were sintered at 450 °C and the density of the composite was up to 1.70 g/cm^3 after 11 infiltration-sintering cycles. The characteristics of 2.5D (shallow straight-joint) structure were determined. Flexural strength and shear strength of the composites were investigated along the warp and weft directions. The undulation of warp yarns could reduce the mechanical properties of the composites along warp direction. The flexural and shear stress-deflection curves exhibited mostly nonlinear behavior. The failure was not catastrophic. Some fibers pull-out indicated that the composite exhibited a certain degree of toughness. Microstructural observations revealed that the interface strength between the fiber and the matrix was weak for the relatively low sintering temperature of 450 °C.

Keywords: C. Mechanical properties; B. Microstructure; B. Composites; 2.5D

1. Introduction

Ceramic materials have been assumed as an important type of structural materials because of the new design and development methodologies. By adopting fiber reinforcements, the fracture toughness of monolithic ceramics can be improved by several folds [1–4]. Due to high melting point, high thermal shock resistance, excellent thermal as well as electrical insulating properties, low dielectric constant and low loss tangents, amorphous silica can be suitable for select technological applications, such as missile radomes, heat-resistant materials for aerospacecraft, crucibles and so on [5]. However, the mechanical properties of silica material in the monolithic form are far from acceptable levels. By comparison with several structural ceramic materials, silica has low strength and extremely low fracture toughness [4–7]. One of the means to improve the mechanical properties of ceramic material is to incorporate either two- (2D-) or three dimensional (3D-) networks of continuous fibers to form a new type of structural materials which called as "continuous fiber-reinforced ceramic matrix composites (CFCCs)" [8–10]. 2D composites have been investigated extensively. However, the widespread applications of 2D composites in many structural components have been limited by poor delamination resistance [11–14]. Although 3D braided composites have improved delamination resistance, damage tolerance and can be used to fabricate complex net or near-net shaped components, they are still inapplicable for manufacturing components with one closed end (e.g., a nose cap)[15,16]. 2.5D composites were studied in literatures [15,17–21]. The characteristics of 2.5D weave technique make the fabric preform particularly suitable for conforming to the mould surface of revolving components and allow net or near-net shaping [15]. The 2.5D composites show better delamination resistance and higher interlaminar fracture toughness than traditional laminated composites when subjected to interlaminar stress concentrations. However, limited attention has been focused towards 2.5D quartz fibers-reinforced silica composites (Q_f/S).

There are a number of 2.5D structures such as 2.5D shallow bend-joint, shallow straight-joint, deep straight-joint, and so on. In order to utilize these new composites most efficiently,

* Corresponding author. Tel.: +86 25 52112909; fax: +86 25 52112626.
E-mail address: jianxun_zhu@163.com (J. Zhu).

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thorough understanding of their mechanical properties is essential. In this paper, the 2.5D shallow straight-joint preform was used as the fiber reinforcement. 2.5D (shallow straight-joint) Q_f/S composites were prepared by silicasol-infiltration-sintering (SIS) method. The structure characteristics of 2.5D (shallow straight-joint) preform were studied. The flexural behavior of the 2.5D (shallow straight-joint) composites and its relation to the microstructures were investigated. Also, the differences in flexural behavior between warp and weft directions were compared.

2. Experimental details

2.1. Composite preparation

The 2.5D (shallow straight-joint) woven preforms were provided by Nanjing Institute of Glass Fiber. The fiber volume fraction was 47.5%. The 2.5D Q_f/S composites were prepared by SIS method. Fig. 1 shows the schematic diagram of the SIS method. The 2.5D preforms were vacuum impregnated using colloidal silica solution precursor (35 vol% SiO_2) for 0.5 h, then the pressure of the container was increased to 10 atm and maintained for 1 h. The interconnected network of capillaries in the preforms facilitated silica solution impregnation, thus provided uniform matrix for the CFCC. The infiltration process was repeated 10 times. After each infiltration, the $CFCC_S$ were dried at 80 °C for 1 h and 110 °C for 1 h, respectively. During this drying process, water content of the matrix gel solution was gradually removed. Then the dried preforms were heated in an oven at 450 °C for 2 h in order to remove the coupling agent and bound water. Compared with the sintering temperature of silica composites from other papers [7,22,23], 450 °C was a relatively low sintering temperature.

2.2. Mechanical properties measurement

The presence of the peculiar structure of 2.5D composites provided two types of test specimens as the length of the composites had been taken parallel to whether the warp or the weft direction of the preform. The as-fabricated composites were cut parallel to the warp and weft directions, and then the cross-sections were ground and polished. Mechanical properties of the composites were characterized under flexural loading and shear loading for both warp direction and weft direction. Flexural testing and shear testing were performed on a pc-controlled electronic universal testing machine (Model CMT5105, SANS Corp., China). Flexural strength was measured using the three-point-bending method. The nominal flexural specimen dimensions were 3.5 mm × 5 mm in cross section and 40 mm in length. The bending support span size was 30 mm and the crosshead speed was 0.03 mm/min. Shear strength was measured using the Iosipescu shear testing method, meanwhile, the composite panels were cut into V-Notched beam specimens. Fig. 2 shows the geometry and dimensions of shear specimens. Fig. 3 shows the test fixture of Iosipescu shear testing method.

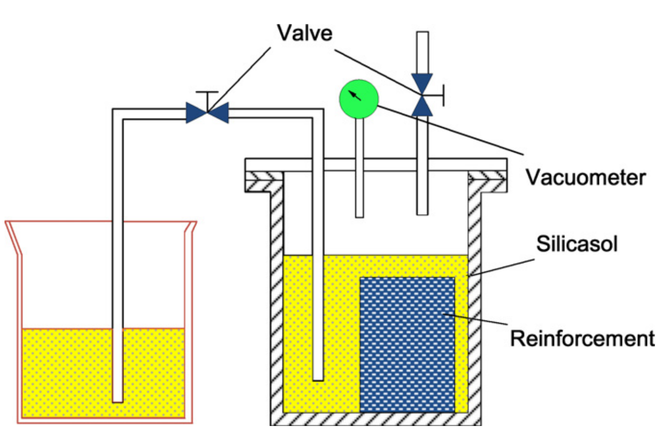

Fig. 1. Schematic diagram of the SIS method.

2.3. Microstructure observation and surface analysis

The composite density was measured using the Archimedes principle. Microstructure of the fracture surface was observed by scanning electron microscopy (SEM, FEI CO., Quanta200

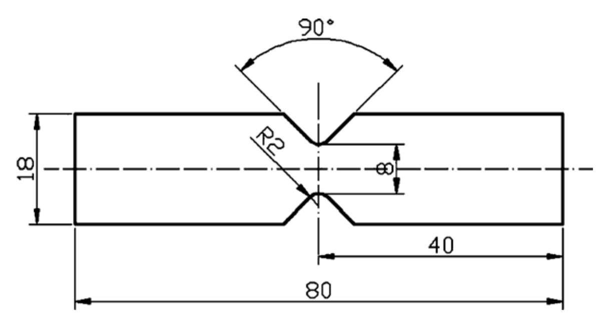

Fig. 2. Geometry and dimensions of shear specimen (dimensions in millimeters).

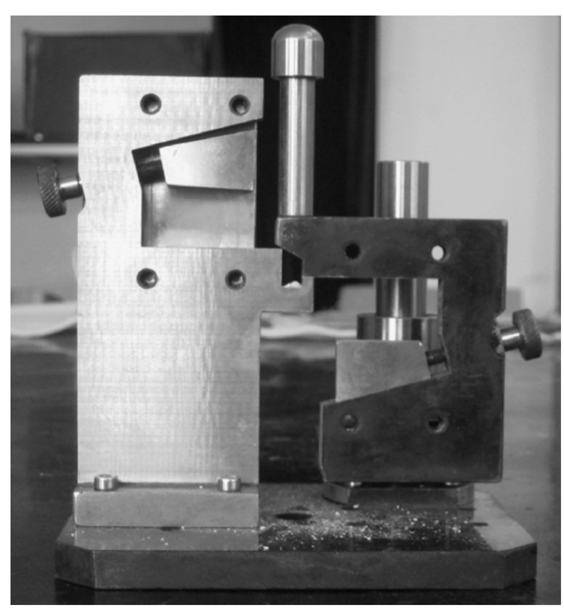

Fig. 3. Test fixture of iosipescu shear testing method.

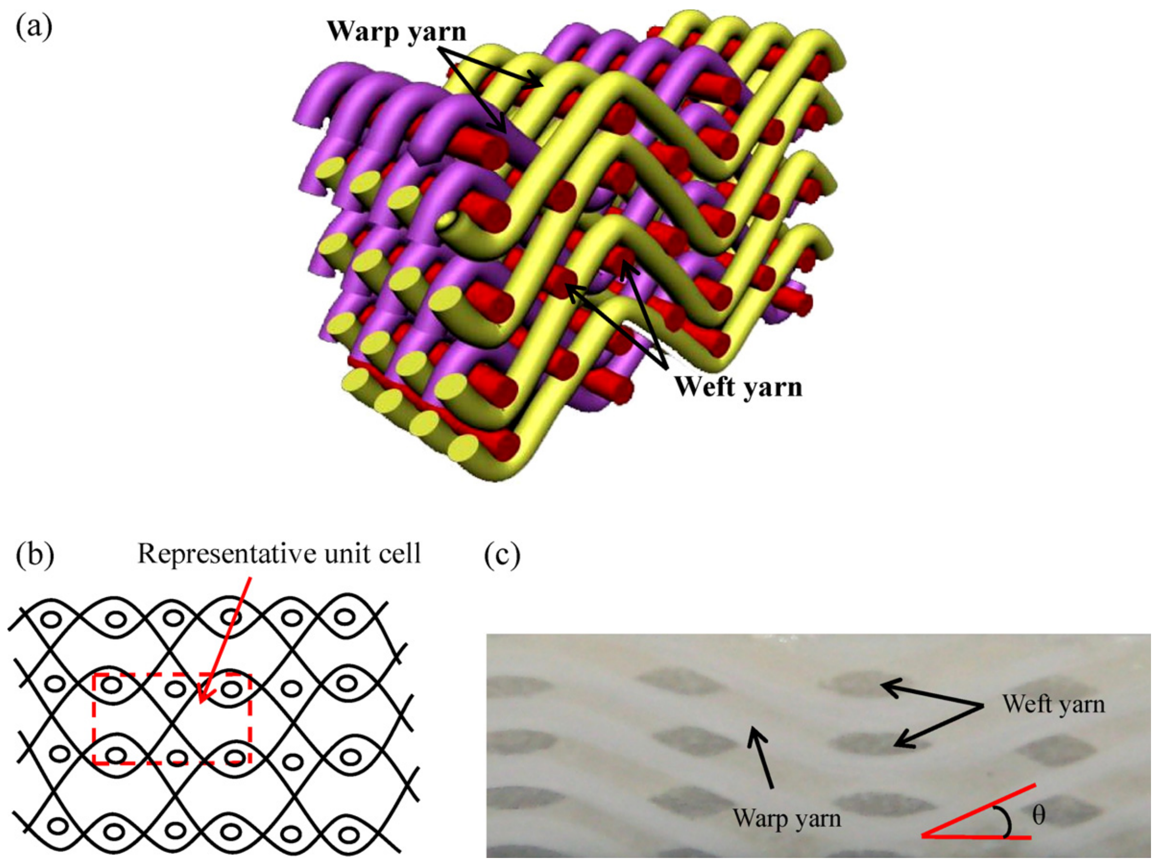

Fig. 4. Structure of 2.5D (shallow straight-joint) preform and specimen. (a) Schematic of fiber preform, (b) Plane graph of fiber preform and (c) longitudinal section of the specimen.

and JSM-6360LV). Before observing, the samples were coated with gold (thicknesses of 10 nm) on the surface because the Q_f/S composites were non-conducting samples.

3. Results and discussion

3.1. Preform structure

Fig. 4 shows the structure of 2.5D (shallow straight-joint) preform and the longitudinal section of the specimen. 2.5D (shallow straight-joint) preform was a unique kind of multilayer fabric. The first set of tows that ran in the weaving direction was called warp yarns, while the second set of tows that ran transverse to the weaving direction was called weft yarns. The preform repeated itself on a certain number of warp and weft yarns. The repeat was a complete representative unit cell of the preform (see Fig. 4(b)). 2.5D composite at representative unit cell level was homogeneous and orthotropic. The preform was composed of layers of straight weft yarns and a set of sinusoidal warp yarns, with the adjacent layers of weft yarns interlaced together by warp yarns. The warp yarns were placed at an angle θ, called the undulation angle (see Fig. 4(c)). The structural characteristics of 2.5D (shallow straight-joint) preform (see Fig. 4) were: (1) a warp yarn was interlaced with each two weft yarns along the thickness direction; (2) a warp yarn was interlaced with every three weft yarns along the weaving direction; (3) the preform was a symmetrical object. The warp density and the weft density of the as-received 2.5D preform were 10 picks/cm and 4 picks/cm, respectively. The 2.5D process could produce near-net-shape preforms for components having complicated geometry and thus reduces the production time and associated costs. Especially, this kind of structure could be used to prepare dome-shaped components.

3.2. Weight-cycle curve of the preparation

Eleven infiltration-sintering cycles were used for densification. The density of the composite was up to 1.70 g/cm^3. As shown in Fig. 5, the weight of the composite could not be

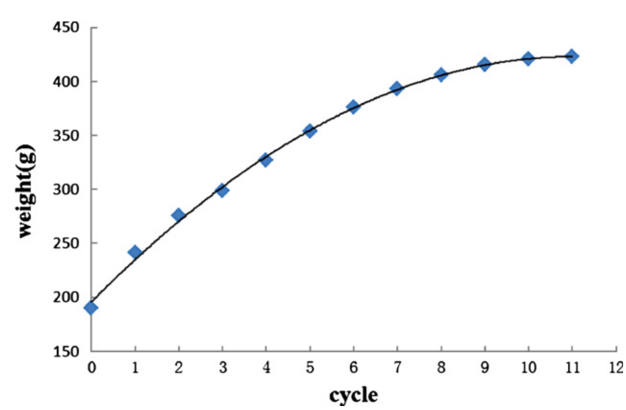

Fig. 5. Weight-cycle curve of 2.5D (shallow straight-joint) composites.

further increased after 9 infiltration-sintering cycles. The infiltration efficiency was debased. There are two reasons for that. First, the number and size of pores decreased with the increase of the cycle number. Second, infiltration paths of the silicasol had being jammed, the silica sol particle could not infiltrate into the composites fully [23].

3.3. Flexural loading and microstructure analysis

Fig. 6 shows the stress-deflection curves tested by three-point bending, which were different from that of monolithic ceramics. The curve (a) and the curve (b) show the test results of weft direction specimen and warp direction specimen, respectively. Both of the curves exhibited mostly nonlinear behavior. The stress-deflection curves could be divided into three stages (See curve (a), omitted in curve (b)). At the stage I, the mechanical behavior of the composites was linear elastic.

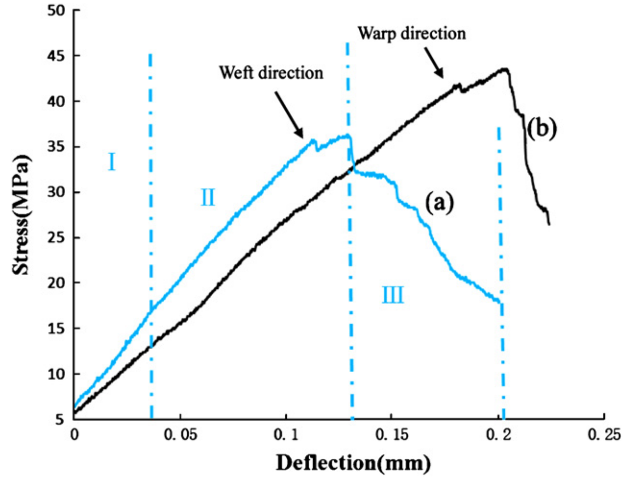

Fig. 6. The flexural stress-deflection curves for 2.5D composites along warp direction and weft direction.

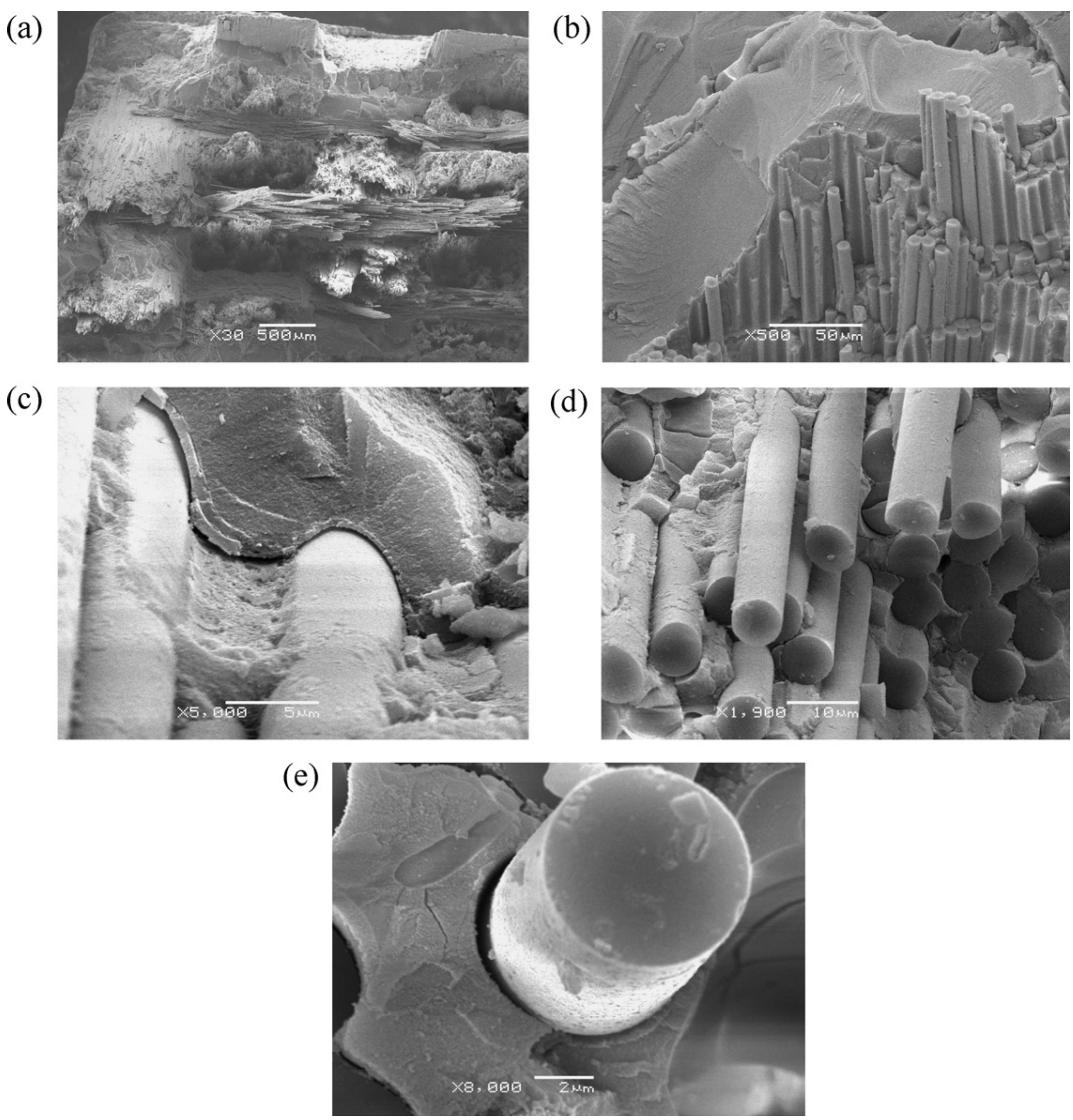

Fig. 7. Fracture surface of 2.5D quartz$_f$/silica composites after flexural loading. (a) Matrix SEM, (b) fiber fracture, (c) interfacial debonding, (d) fiber fracture and (e) fiber pull-out.

The matrix cracks and other voids induced during manufacturing did not grow and contribute to the initial matrix properties. At the stage II, a nonlinear region appeared. The matrix continuously cracked, meanwhile the fiber–matrix interface debonded. The matrix failed when the maximum load arrived. At the stage III, some fibers did not fail within the matrix crack plane but within the intact matrix, the composite was carried by a reduced load, as then the fibers were pulled out of the matrix. The stage III was attributed to the fiber pull-out and bridging. This phenomenon could be more clarified from the SEM micrograph as shown in Fig. 7(e). The average values of the flexural strength for warp direction and weft direction were 48.4 MPa and 32.8 MPa, respectively.

The anisotropy of the composites was closely related to the differences in yarn density and yarn path between warp and weft yarns. The composites had 2.5 times as many yarns in the warp direction as compared to the weft direction. However, the flexural strength in the warp direction was only 1.48 times that for the weft direction. Such a decrease in strength could be foreseen because the undulation of warp yarns could significantly reduce the in-plane mechanical properties [15]. Obviously, the reduction in the mechanical properties was influenced to a large extent by the waviness of the warp yarns. The greater the undulation angle θ, the weaker the in-plane mechanical properties of the composites. For the 2.5D preform, by adjusting the weft density, enables the undulation degree of warp yarns change. A lower weft density can make a lower undulation degree of warp yarns and vice versa. However, a lower weft density will reduce the mechanical properties in the weft direction. Consequently, the mechanical properties of the composites can be tailored for specific applications by varying the weave parameters such as weaving tension, weft density and warp density [15].

The fracture surface morphology can well reflect the fracture characteristics of the composites. Fig. 7 shows the SEM micrographs of fracture surface of the 2.5D (shallow straight-joint) Q_f/S composites after flexural loading. It was found that the yarns were fractured at various regions and the fracture surfaces of the yarns were very ragged. The fibers exhibited multi-step fracture and without extensive pull-out (see Fig. 7(a)). Fig. 7(b) shows the edge of the specimen. The quartz preform was surrounded by dense silica matrix. It indicated that the preparation method was successful. The tough mechanical response of CFCCs relied upon their interfacial mechanical behavior. The interface characteristics resulted from the interaction between the matrix and the fiber during process. It was generally accepted that interfacial debonding at the fiber–matrix interface was the precondition for crack energy dissipating mechanisms, such as crack deflection, crack bridging and fiber pull-out. Thus, the interface strength must be weak enough to facilitate the crack energy dissipating mechanisms. Because the composites were prepared at low temperature, the interaction between the matrix and the fiber was weak. The interface strength was weak and hence debonding at the fiber–matrix interface was easily achieved under low mechanical load (see Fig. 7(c)). Fig. 7(d) and (e) shows that some fibers were pulled out of the matrix, and it can be observed from Fig. 7(d) that the surface of quartz fiber was smooth and fiber degradation was not happened. Fiber pull-out was a toughening process operating in the CFCCs during failure. So the composite exhibited a certain degree of toughness. In summary, quartz fiber would be pull out of the matrix easily at a low preparation temperature thus leaded to enhanced fracture toughness.

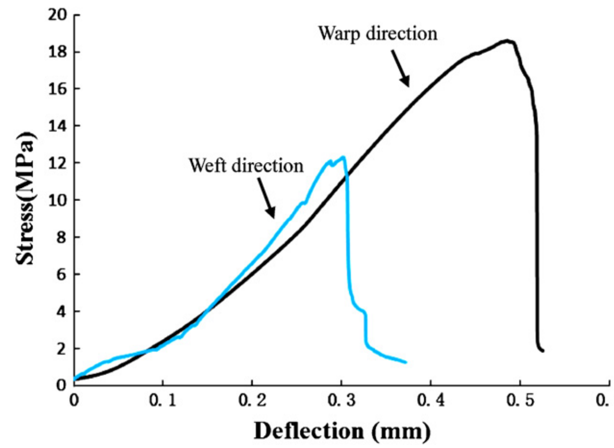

Fig. 8. Stress-deflection curves under shear loading.

3.4. Shear loading

The shear strength of the Q_f/S composites was measured by the Iosipescu shear testing method. The loading rate was 0.3 mm/min. Shear strength was calculated by the following equation:

$$\tau = \frac{P}{h\omega}$$

where P is the maximum fracture load (N), h and ω are the height and the minimum distance between v-notched of the sample, respectively.

Fig. 8 shows the stress-deflection curve of the shear failure behavior of the 2.5D (shallow straight-joint) Q_f/S composites. The shear failure behavior was similar to the behavior of bending failure. The average values of the shear strength for warp direction and weft direction were 18 MPa and 11.2 MPa, respectively. The shear strength in the warp direction was only 1.61 times that for the weft direction due to the undulation of warp yarns. Different from 2D CFCCs and the other laminated composites, interlayer debonding was not observed in the present composites. The results indicated that the 2.5D (shallow straight-joint) Q_f/S composites exhibited good shear resistant.

4. Conclusions

2.5D (shallow straight-joint) quartz$_f$/silica composites were successfully prepared by 11 silicasol-infiltration-sintering cycles. The density of the composites was 1.70 g/cm^3. The average values of the flexural strength for warp direction and weft direction were 48.4 MPa and 32.8 MPa, respectively. The average values of the shear strength for warp direction and weft direction were 18 MPa and 11.2 MPa, respectively. Because of the undulation of warp yarns, the warp strength/weft strength ratio did not match the warp density/weft density ratio.

The flexural stress-deflection curves of 2.5D quartz$_f$/silica composites exhibited mostly nonlinear behavior. The curves could be divided into three stages: the linear elastic stage follow by the nonlinear damage stage and finally the failure stage. Toughening effect of 2.5D quartz preform was obvious. Low temperature preparation could lead to enhanced fracture toughness.

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References

[1] W.J. Clegg, K. Kendall, N. McN. Alford, T.W. Button, J.D. Birchall, A simple way to make tough ceramics, Nature 347 (1990) 455–457.
[2] Paul F. Becher, Microstructural design of toughened ceramics, J. Am. Ceram. Soc. 74 (10) (1991) 2720.
[3] Anthony G. Evans, Perspective on the development of high-toughness ceramics, J. Am. Ceram. Soc. 73 (2) (1990) 187–206.
[4] N. Eswara Prasad, S.V. Sweety Kumari, M. Kamat, G. Vijayakumar, Malakondaiah, Fracture behaviour of 2d-weaved, silica-silica continuous fibre-reinforced, ceramic-matrix composites (cfccs), Eng. Fract. Mech. 71 (18) (2004) 2589–2605.
[5] N. Eswara Prasad, D. Loidl, M. Vijayakumar, K. Kromp, Elastic properties of silica-silica continuous fibre-reinforced, ceramic matrix composites, Scripta Mater. 50 (8) (2004) 1121–1126.
[6] Carlos R. Ortiz-Longo, Kenneth W. White, Elevated-temperature fracture characterization of advanced fibrous ceramic thermal insulators, J. Am. Ceram. Soc. 77 (10) (1994) 2703–2711.
[7] C. Xu, S.W. Wang, X.X. Huang, J.K. Guo, Processing and properties of unidirectional SiO_{2f}/SiO_2 composites, Ceram. Int. 33 (4) (2007) 669–673.
[8] Y.D. Xu, L.F. Cheng, L.T. Zhang, H.F. Yin, X.W. Yin, High toughness, 3d textile, SiC/SiC composites by chemical vapor infiltration, Mater. Sci. Eng. A 318 (1–2) (2001) 183–188.
[9] P. Lipetzky, G.J. Dvorak, N.S. Stoloff, Tensile properties of a SiC_f/SiC/ composite, Mater. Sci. Eng. A 216 (1–2) (1996) 11–19.
[10] Y.D. Xu, L.F. Cheng, L.T. Zhang, H.F. Yin, X.W. Yin, Mechanical properties of 3d fiber reinforced C/SiC composites, Mater. Sci. Eng. A 300 (1–2) (2001) 196–202.
[11] Y.D. Xu, L.F. Cheng, L.T. Zhang, X.W. Yin, H.F. Yin, High performance 3d textile hi-nicalon SiC/SiC composites by chemical vapor infiltration, Ceram. Int. 27 (5) (2001) 565–570.
[12] J. Lamon, B. Thommeret, C. Percevault, Probabilistic-statistical approach to matrix damage and stress–strain behavior of 2-d woven SiC/SiC ceramic matrix composites, J. Eur. Ceram. Soc. 18 (13) (1998) 1797–1808.
[13] Gregory N. Morscher, Stress-dependent matrix cracking in 2d woven sic-fiber reinforced melt-infiltrated sic matrix composites, Compos. Sci. Technol. 64 (9) (2004) 1311–1319.
[14] J. Chevalier, M. Huger, D. Fargeot, C. Gault, Ultrasonic investigation of the time-dependent damage in a 2d SiC/SiC composite under static loading, J. Eur. Ceram. Soc. 18 (13) (1998) 1857–1867.
[15] J.Q. Ma, Y.D. Xu, L.T. Zhang, L.F. Cheng, J.J. Nie, N. Dong, Microstructure characterization and tensile behavior of 2.5d C/SiC composites fabricated by chemical vapor infiltration, Scripta Mater. 54 (11) (2006) 1967–1971.
[16] F. Zhang, Z.G. Liu, Z. Wu, G.Q. Tao, A new scheme and microstructural model for 3d full 5-directional braided composites, Chin. J. Aeronaut. 23 (1) (2010) 61–67.
[17] L. Hahn, F. Ansorge, A. Bruckner-Foit, Damage and failure behaviour of a woven C/SiC material, J. Mater. Sci. 32 (20) (1997) 5467.
[18] A. Dalmaz, D. Ducret, R. El Guerjouma, P. Reynaud, P. Franciosi, D. Rouby, G. Fantozzi, J.C. Baboux, Elastic moduli of a 2.5d C_f/SiC composite: experimental and theoretical estimates, Compos. Sci. Technol. 60 (6) (2000) 913–925.
[19] G. Boitier, J. Vicens, J.L. Chermant, Understanding the creep behavior of a 2.5d C_f-SiC composite. III. From mesoscale to nanoscale microstructural and morphological investigation towards creep mechanism, Mater. Sci. Eng. A. 313 (1–2) (2001) 53–63.
[20] G. Boitier, J.L. Chermant, J. Vicens, Understanding the creep behavior of a 2.5d C_f-SiC composite: II. Experimental specifications and macroscopic mechanical creep responses, Mater. Sci. Eng. A 289 (1–2) (2000) 265–275.
[21] G. Boitier, J. Vicens, J.L. Chermant, Understanding the creep behavior of a 2.5d C_f-SiC composite-i. Morphology and microstructure of the as-received material, Mater. Sci. Eng. A 279 (1–2) (2000) 73–80.
[22] H. Chen, L.M. Zhang, L.M. Hu, G.Y. Jia, W.H. Luo, S. Yu, Z.Q. Cheng, Flexural properties of 3d-SiO_2/SiO_2 composites, Key Eng. Mater. 249 (2003) 163–166.
[23] S.A. Han, K.H. Jiang, J.W. Tang, Studies on preparation and property of 2.5d SiO_{2f}/SiO_2 composites, Adv. Mater. Res. 79–82 (2009) 1767–1770.