MECHANICAL PROPERTIES AND CHARACTERIZATION OF A NOVEL COUPLED 2.5D BRAIDED SiO$_2$/SiO$_2$ COMPOSITE

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Abstract
Coupled 2.5D SiO$_2$/SiO$_2$ composites were successfully fabricated by silica sol-infiltration-sintering method. The fiber volume fraction was 48.4%. The composites were sintered at 450°C. The density of the composite was 1.74 g/cm$^3$. Tensile strength, Flexural strength and shear strength of the composites were investigated. The tensile, flexural and shear stress-displacement curves exhibited mostly nonlinear behavior. The failure was not catastrophic. Toughing effect of the coupled 2.5D quartz preform was obvious.

1 Introduction
Amorphous silica is an ideal material to make radome and windows of antenna due to its high melting point, high thermal shock resistance, and excellent thermal as well as dielectric properties[1-4]. Unfortunately, due to the low strength and extremely low fracture toughness of the silica in the monolithic form, the use of silica as a structure material is limited. Silica reinforced by silica fiber reinforcements (SiO$_2$/SiO$_2$) can be used to increase the fracture toughness and reliability of monolithic silica. Previous work mainly concentrated on incorporating short and/or two-dimensional (2D) silica fiber into the silica matrix. It was shown that the work of fracture and the strength properties were enhanced. However, there was uneven distribution of fiber density in the short silica fibers reinforced silica composites and poor delamination resistance in the 2D silica composites[5, 6]. 2.5D composites were studied in literatures[7-11]. 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[12]. There are a number of 2.5D structures such as 2.5D shallow bend-joint, shallow straight-joint, deep straight-joint, and so on. However, most of the studies only paid close attention to unitary 2.5D structure, coupled 2.5D braided silica composites are rarely reported in literatures.

In this paper, a novel coupled 2.5D preform (combines 2.5D shallow straight-joint with 2.5D shallow bend-joint) was used as the fiber reinforcement. The SiO$_2$/SiO$_2$ composites were prepared by silica sol-infiltration-sintering (SIS) method. The mechanical properties and the failure behavior of the 2.5D SiO$_2$/SiO$_2$ composites were investigated.
2 Materials and testing methods

2.1 2.5D preform
The coupled 2.5D preform was provided by Nanjing Institute of Glass Fiber, Nanjing, China. The geometrical parameters of the coupled 2.5D preform are listed in Table 1. Figure 1 shows the schematic diagram and structure of the coupled 2.5D preform. The coupled 2.5D preform was made of 2.5D shallow straight-joint and 2.5D shallow bend-joint (alternating four weft yarns).

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<tbody>
<tr>
<td>coupled 2.5D</td>
<td>195 X 2</td>
<td>10</td>
<td>4</td>
<td>48.4%</td>
<td>alternating four weft yarns (shallow bend joint)</td>
</tr>
</tbody>
</table>

Table 1. Geometrical parameters of the coupled 2.5D quartz fiber preform.

![Figure 1](image)

Figure 1. Schematic of the coupled 2.5D braided quartz preform: (a) coupled 2.5D quartz preform; (b) coupled 2.5D structure.

2.2 Composites preparation procedures
The coupled 2.5D preform was vacuum impregnated using colloidal silica solution precursor (35vol% SiO2) for 0.5h. Afterwards, the pressure of the container was increased to 10 atm and maintained for 1h. The interconnected network of capillaries in the preform facilitated silica solution impregnation thus provided uniform matrix for the composites. The infiltration process was repeated 10 times. After each infiltration, the composites were dried at 80°C for 1h and at 110°C for 1h, respectively. During this drying process, water content of the matrix gel solution was gradually removed. Then the dried preform was sintered in an oven at 450°C for 2h in order to remove the coupling agent and bound water. Compared with the sintering temperature of silica composites from other papers, 450°C was a relatively lower sintering temperature [13-15].

2.3 Testing methods
Mechanical properties of the composite were characterized under tensile loading, flexural loading and shear loading. The mechanical tests were carried out in a pc-controlled electronic
universal testing machine (Model CMT5105, SANS Corp., China). Tensile test specimens with dimensions of 3.5 mm × 23mm × 94 mm were cut from the fabricated composite plates and tapered aluminum tabs were glued at both ends to provide a gauge length of 48 mm. Tensile tests were performed at a constant cross-head speed of 0.3 mm/min. The flexural strengths were measured using three-point bending method on 3.5mm X 4mm X 40mm beams at a cross-head speed of 0.3 mm/min and a span of 40 mm. Shear strength was measured using the Iosipescu shear testing method. The samples were 3.5mm X 18mm X 80mm with two 45° notched (5mm depth) beams.

The composite density was measured using the water displacement method. Fracture surfaces of failed specimens were characterized using scanning electron microscopy (SEM, FEI CO., Quanta200 and JSM-6360LV). The specimens for SEM were gold coated.

3 Results and Discussion

The density of the composite was up to 1.74 g/cm³ after 10 SIS cycles. Table 2 summarized the mechanical properties of different types of 2.5D preform reinforced SiO₂ /SiO₂ composites[7, 16]. The mechanical properties of the coupled 2.5D braided SiO₂ /SiO₂ composite were between that of 2.5D shallow straight-joint and 2.5D shallow bend-joint SiO₂ /SiO₂ composite. It was demonstrated that 2.5D composites have good designability. The 2.5D braiding technology can make kinds of products with different properties. According to the need and/or material selection criteria of an engineering project, a combination of more than two types of 2.5D structures may be required.

<table>
<thead>
<tr>
<th>Types</th>
<th>Density[g/cm³]</th>
<th>Tensile strength [MPa]</th>
<th>Flexural strength [MPa]</th>
<th>Shear strength [MPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.5D shallow bend-joint</td>
<td>1.77</td>
<td>22.7</td>
<td>50.3</td>
<td>22.4</td>
</tr>
<tr>
<td>2.5D shallow straight-joint</td>
<td>1.70</td>
<td>24.5</td>
<td>48.4</td>
<td>18.0</td>
</tr>
<tr>
<td>coupled 2.5D</td>
<td>1.74</td>
<td>23.2</td>
<td>49.9</td>
<td>21.5</td>
</tr>
</tbody>
</table>

Table 2. Mechanical properties of 2.5D composites.

Table 3 summarizes some flexural strength test data of different types of preform reinforced SiO₂ /SiO₂ composites[6, 17, 18], and can be inferred that the SiO₂ /SiO₂ composites with the coupled 2.5D braided preform had higher flexural strength than those with 3D woven preforms. However, compared to 3D braided SiO₂ /SiO₂ composites, the coupled 2.5D braided had the lower flexural strength. The difference of fiber volume fraction between these four types of composites was very small; however, there were large differences in flexural properties. The fiber placement in the preform strongly affects the mechanical properties. The coupled 2.5D SiO₂ /SiO₂ composites exhibited well comprehensive performances.

Fig.2 shows the stress-displacement behavior of the composites under tensile loading, flexural loading and shear loading. The curves exhibited highly nonlinear behavior and it was clearly showed that coupled 2.5D SiO₂ /SiO₂ composites exhibited graceful failure under loading. Toughening effect of the coupled 2.5D quartz preform was obvious. Although both constituents (quartz fibers and silica matrix) of SiO₂ /SiO₂ composites were brittle, the
composites displayed quasi-ductile deformation. The fracture surface morphology can well reflect the fracture characteristics of the composites.

<table>
<thead>
<tr>
<th>Types</th>
<th>Density [g/cm$^3$]</th>
<th>Fiber volume fraction</th>
<th>Flexural strength [MPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>3D orthogonal woven</td>
<td>1.71</td>
<td>50.2%</td>
<td>29.4</td>
</tr>
<tr>
<td>3D sinking woven</td>
<td>1.74</td>
<td>49.8%</td>
<td>47.9</td>
</tr>
<tr>
<td>3D braided coupled 2.5D</td>
<td>1.71</td>
<td>46.8%</td>
<td>64.0</td>
</tr>
<tr>
<td></td>
<td>1.74</td>
<td>48.4%</td>
<td>49.9</td>
</tr>
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</table>

Table 3. Flexural test data of SiO$_2$/SiO$_2$ composites

![Mechanical testing curves of the coupled 2.5D braided SiO$_2$/SiO$_2$ composites](image)

**Figure 2.** Mechanical testing curves of the coupled 2.5D braided SiO$_2$/SiO$_2$ composites: (a) tensile testing curve; (b) flexural testing curve; (c) shear testing curve.

Fig.3 shows the SEM micrographs of the fractured surface of the coupled 2.5D SiO$_2$/SiO$_2$ composites after flexural loading. The properties of fiber reinforced composites were markedly influenced by the interfacial properties and the fabric structures. If the interface was
weak enough for the matrix crack to be deflected along the interface, the fibers remain intact and the composite could be tough. If the interface was too strong, the matrix crack penetrates into the fibers and the composite was brittle like a monolithic ceramic. 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[19]. Because the composites were prepared at a relatively low temperature(450°C), incomplete sintering happened between the matrix and the fiber. The interface strength was relatively weak and hence debonding at the fiber/matrix interface was easily achieved under low mechanical load. The fibre-matrix debonding led to fiber-matrix sliding with friction, and then the quartz fibers pulled-out of the silica matrix. The frictional sliding contributed to energy absorbing mechanisms of the coupled 2.5D SiO₂f/SiO₂ composites and led to a non-catastrophic gradual failure of the SiO₂f/SiO₂ composites.

![Figure 3. The fractured surface of the coupled 2.5D SiO₂f/SiO₂ composites after flexural loading.](image)

4 Conclusions
Coupled 2.5D SiO₂f/SiO₂ composites were successfully fabricated by silica sol-infiltration-sintering method. 2.5D composites have good designability. According to the need and/or material selection criteria of an engineering project, a combination of more than two types of 2.5D structures may be required. The average values of tensile strength, flexure strength and the shear strength of the composites were 23.2 MPa, 49.9 MPa and 21.5 MPa at room temperature, respectively. Due to a relatively low preparation temperature of 450°C, the fiber matrix bond strength was relatively weak. The as-fabricated coupled 2.5D SiO₂f/SiO₂ composites exhibited non-catastrophic gradual failure under loading.

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References


