Electronic supplementary information for:

# Hierarchical structural silica-fiber-woven/mullite-whiskers material prepared by surface etching and gas phase reaction

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## Part 1. Preparation and test methods of the hierarchical structural silica-fiber-woven/mullite-whiskers material

### 1.1 Preparation

Silica-fiber-woven (with thickness of about 1mm) was economically purchased. $AlF_3$ (analytically pure) and $SiO_2$ (analytically pure) powders were mixed through high-energy ball-milling by planetary ball milling machine[$(AlF_3/SiO_2)_{mole}$=3/1] . The silica-fiber-woven was placed in an airtight alumina crucible, and the mixed $AlF_3$-$SiO_2$ powders were placed surround the silica-fiber-woven in the crucible {[(mixed $AlF_3$-$SiO_2$ powders)/silica-fiber-woven]$_{mass}$=1/1}. The heat-treating temperature was 800-1000°C, holding for 2h.

### 2. Testing methods

Scanning electronic microscope (SEM, Nanosem430 from FEI) with energy dispersive spectrometer attached on it was applied for observing the microstructure and detecting the chemical composition of the samples. Transmission electron microscope (TEM, Tecnai G2 F20 from FEI) was used for detecting the morphology and crystallinity of the mullite whiskers that grew on the silica fibers. Volume density of the material was calculated by Archimedes method. Thermal conductivity of the material was tested by hot-line thermal conductor meter (XIATECH TC3000). Tensile strength of the material was tested by electronic universal testing machine (CSS-44001).

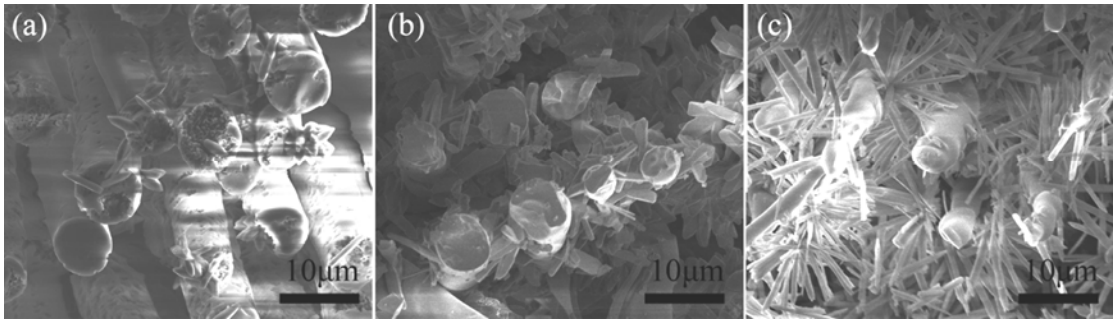


**Fig. S1** SEM images of the cross-section of the samples that was formed at 800°C (a), 900°C (b) and 1000°C (c)

**Part 2. Silica-fiber-woven's complete etching in the gas phase reaction**

In the experiment, excess raw material powders ($AlF_3$+$SiO_2$) were placed around the silica-fiber-woven in an airtight alumina crucible, and the heating temperature was 1000°C. **Fig. S2** showed the SEM image of the completely etched sample that formed at 1000°C. The sample was very weak. It could be broke off very easily by hand. The microstructure of the sample was composed of cumulate mullite whiskers.. This result proved the etching of the silica-fiber-woven during the heating process.

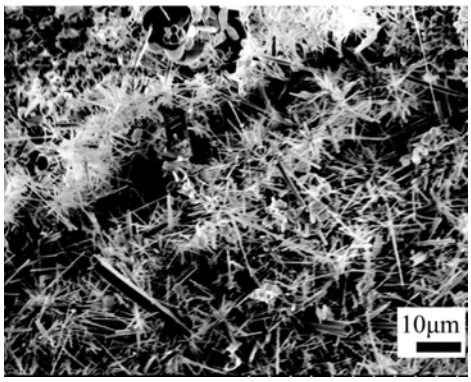


**Fig. S2** SEM image of the completely etched silica-fiber-woven that formed at 1000°C.

**Part 3. Supplementary experiment on changing the mass ratio of the starting material**

In the supplementary experiment, the samples' mass ratio of (raw material)/silica-fiber-woven was 0.5/1 and 1.5/1. The heating temperature was 1000°C. **Fig. S3** showed the SEM images of the samples with different mass ratio of the starting material. It could be observed in **Fig. S3** that, as the mass ratio of (raw material)/silica-fiber-woven increased, the mullite whiskers gradually became larger. **Table S1** showed the volume density, thermal conductivity and tensile strength of the samples with different mass ratio of the starting material. As the mass ratio of (raw material)/silica-fiber-woven increased, the volume density/thermal conductivity/tensile strength decreased.

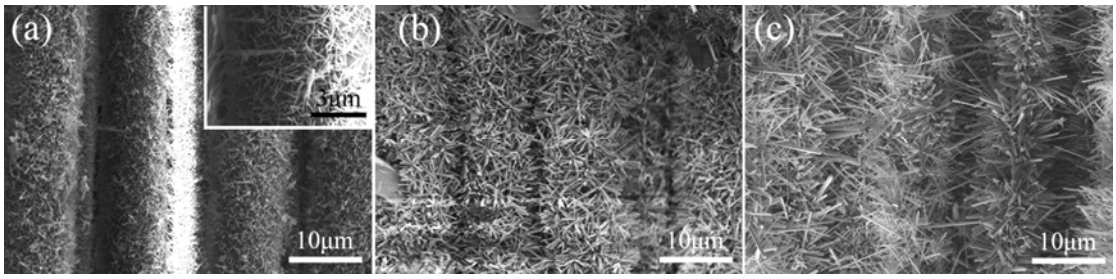


**Fig. S3** SEM images of the samples with different mass ratio of starting material, (a) $[(\text{raw material})/\text{silica-fiber-woven}]_{mass}$=0.5/1 (sample in the supplementary experiment), (b) $[(\text{raw material})/\text{silica-fiber-woven}]_{mass}$=1/1 (sample which was prepared in the manuscript), (c) $[(\text{raw material})/\text{silica-fiber-woven}]_{mass}$=1.5/1 (sample in the supplementary experiment).

**Table S1** Volume density, thermal conductivity and tensile strength of the samples with different mass ratio of the starting material.

| [(raw material)/silica-fiber-woven]$_{mass}$ of the sample | Volume density (g/cm$^3$) | Thermal conductivity (W/m⊙k) | Tensile strength (MPa) |
|---|---|---|---|
| 0.5/1 | 0.641 | 0.1254 | 0.502 |
| 1/1 | 0.572 | 0.1233 | 0.441 |
| 1.5/1 | 0.538 | 0.1229 | 0.237 |