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Mechanical properties of aluminosilicate fiber heat-treated from 800 °C to 1400 °C: Effects of phase transition, grain growth and defects

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ABSTRACT

Thermal stability of oxide fiber is a key property for high temperature structural applications of typical oxide fiber reinforced oxide matrix composites. This work contributes to provide insight into the mechanical properties of a typical aluminosilicate (AS) fiber heat-treated from 800 °C to 1400 °C in air, with the emphasis on the effects of phase transition, grain growth and defect formations. The results showed a phase reaction occurring at 1150 °C, which formed an orthorhombic structured mullite phase. The grain size of the newly formed mullite was observed increased significantly at higher temperatures. Both the phase transition and grain size led to more flaw formations in the microstructure, which influenced significantly the mechanical property of the AS fiber. Specifically, the Young's modulus and tensile strength were both degraded remarkably from ≈145 GPa to ≈110 GPa, and from ≈1.03 GPa to ≈0.52 GPa, respectively, as the heat-treatment temperature increased from 1200 °C to 1400 °C. Finally, the formations of more flaws yielded a higher dispersion of tensile strength of AS fiber, due to the volume dependent flaw distributions, as revealed by the Weibull statistical analysis.

1. Introduction

Oxide fiber reinforced oxide matrix (oxide/oxide) composites are excellent candidates for high temperature structural applications (aircraft engines, hypersonic missiles and flight vehicles, etc.), due to their all oxide natures of composite components, which can be thermally stable up to extremely high service temperatures (> 1000 °C) in harsh environments [1–6]. Compared to other ceramic matrix composites (CMCs) composed of non-oxide components, like SiC fiber reinforced SiC matrix (SiC_f/SiC) composites, the oxide/oxide composites exhibit comparably poor mechanical properties, mainly due to the low modulus/strength of oxide matrix, like SiO_2, mullite, etc. [7,8]. High strength aluminosilicate (AS) fiber reinforced oxide matrix (AS_f/oxide) composites [7–12], which have been profusely studied in literatures, is a paradigmatic example. They typically display a bending strength ≈100 MPa [7,9], which is several orders lower than ≈400 MPa for typical SiC_f/SiC composites [13,14].

The mechanical properties of oxide/oxide composites can be optimized by tailoring the processing temperatures, like the sintering temperature for sol-gel fabricated composite [8,15], the pyrolysis temperature for polymer derived composite, etc. [16], to enhance the mechanical property of as-fabricated oxide matrix. However, the effect of processing temperature on the overall mechanical properties of oxide/oxide composite is complicated, because not only the property of oxide matrix, but the properties of oxide fiber and fiber/matrix interface are temperature dependent [7,15], which may arise difficulties to decouple the effect of individual composite components. Specially, for typical AS fiber (like ALF 2880D [17]) composed mainly of crystallized Al_2O_3 and amorphous SiO_2, chemical reactions between the two phases were widely reported at ≈1200 °C, which led to the formation of mullite phase inside the AS fiber [17–21]. This mechanism can be triggered by heat-treatment at high temperatures (≥1100 °C) in air. Accompanied with it, grain growth and defects could also be observed simultaneously. Specially, for the ALF 2880D fiber consisting of ≈2 wt % B_2O_3, the defect formation at high temperatures is presumably due to the evaporation of B_2O_3 phase at high temperature. Nevertheless, these changes in microstructures due to heat-treatment mainly dominate the overall mechanical behavior of typical AS fiber. Measured by uniaxial tensile test on fiber bundles, the tensile strength of ALF 2880D fibers after phase transition was reported only retaining ≈50% of its original strength [17]. Since the toughening mechanisms in CMCs, like fiber bridging, fiber pull-out, etc., are highly dependent on the mean strength and its dispersion of reinforcing fibers, the macro mechanical properties of AS/oxide composite were finally found severely degraded when the

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processing temperature beyond the critical phase transition temperature [7,8].

To this end, this work contributes to provide insight into the temperature dependent mechanical properties of AS fibers heat-treated from 800 °C to 1400 °C in air, with the emphasis on the effects of phase transition, grain sizes and defects formed inside the AS fibers. An ALF 2220S fiber was employed in this work, because it was B_2O_3 free, thus its effect on the microstructure and the mechanical property of the fiber can be eliminated. The phase transition inside AS fiber was ascertained by X-ray diffraction (XRD). The grain size of AS fiber was quantified statistically inside a transmission electron microscopy (TEM). Scanning electron microscopy (SEM) was employed to study the morphological changes after heat-treatment process. All these microstructural features were finally correlated to the mechanical properties of AS fibers, which were measured by filament tensile tests, with the help of Weibull statistical analysis.

2. Experiments

2.1. Description of As-received AS Fiber

The AS fiber employed in this work was ALF 2220S, produced by Nitivy Co. Ltd. in Japan. Table 1 summarizes the basic information of the as-received AS fiber given by the manufacturers. The AS fiber (diameter ≈7.0 μm) was mainly composed of amorphous SiO_2 and γ-$A1_2O_3$ (28 wt% SiO_2, 72 wt% $A1_2O_3$), which was also evidenced by the high-resolution TEM (HR-TEM) analysis, as shown in Fig. 1(a). The average grain size of γ-$A1_2O_3$, ≈15 nm, was very small. Since the Young's modulus of SiO_2 and γ-$A1_2O_3$ were ≈73 GPa and ≈253 GPa, respectively, the AS fiber exhibited an intermediate Young's modulus, averaging ≈190 GPa. The tensile strength of the as-received AS fiber was reported ≈1.80 GPa. As will be shown later, the strength can be degraded by a simple heat-treatment process from 800 °C to 1400 °C in air. The underlying microstructural-based mechanisms are the main objective of this work. The surface of the as-received AS fiber is generally covered by an organic coating, which was removed in priority by high-temperature oxidation at 600 °C for 2 h in air. After heat cleaning, the surface was quite smooth, without observations of any micro-scaled defect in the selected area, as shown in Fig. 1 (b). Heat-treatment at 800 °C, 1000 °C, 1100 °C, 1200 °C, 1300 °C and 1400 °C were finally carried out in air for 4 h to study the temperature dependent mechanical property of the AS fiber.

2.2. Characterization Techniques

The surface and cross-sectional morphologies of the as-received and the heat-treated AS fibers were observed inside a SEM (Hitachi FEG S4800, Japan). The phase evolution of AS fiber at increasing temperatures was characterized *out-situ* by XRD (Bruker D8ADVANCE, Germany) using Cu Kα (wavelength λ = 1.54 Å) radiation at incident angle (2θ), 10–80°. Bright-filed TEM (BF-TEM) and high-resolution TEM (HR-TEM) were employed to characterize the grain structure and to quantify statistically the grain size distributions of AS fiber in a Tecnai F20, USA. The TEM specimens were prepared by lifting and thinning a thin lamella from the cross-section of AS fiber inside a focused ion beam (FIB)/SEM system (Helios, FEI 600i Nanolab). Thermogravimetry (TGA) and differential scanning calorimetry (DSC) (SDTQ600, USA) were further conducted in air at a heating rate of 10 °C/min up to 1400 °C, in order to study the thermal stability of the AS fiber in response to extremely high temperatures.

Table 1
Basic information of the as-received AS fiber.

Parameter	Value
Filament diameter	7.0 ± 0.2 μm
Mean grain size	~15 nm
Composition	28 wt% SiO_2, 72 wt% $A1_2O_3$
Phase content	Amorphous SiO_2, γ-$A1_2O_3$
Tensile strength	1.8 ± 0.1 GPa
Elastic modulus	190 ± 1.2 GPa
Surface composition	Organic coating

The mechanical property of AS fiber heat-treated at different temperatures was quantified by filament uniaxial tensile test at room temperature inside an universal strength machine (Testometrix Micro 350, UK). Prior to uniaxial test, the AS fiber filament was glued carefully on a testing paper (gauge length 25 mm) to ensure a loading direction that is strictly along the fiber axis. The tensile test was performed in a quasi-static mode with a loading rate of 5 mm/min. The engineering stress and strain were calculated based on the obtained force and displacement data. The fiber diameter of each specimen was measured carefully inside an optical microscope. At least 25 specimens were tested for each heat treatment condition, in order to quantify statistically the strength distribution as a function of fiber volume based on the well-known Weibull statistical model [22].

3. Results and Discussion

3.1. Crystallized Structure Evolved With Temperature

Fig. 2 shows the TGA and DSC curves of the AS fiber as a function of temperature from 25 °C to 1400 °C. Alike other oxide fibers, the AS fiber was thermal-stable in air as only < 1.4% weight loss was detected up to 1400 °C. Below 200 °C, an endothermic process was observed based on the DSC curve, which led to a decrease in the temperature difference from ≈ −0.5 °C/mg to ≈ −2 °C/mg. This is probably due to the evaporation of H_2O absorbed inside the AS fiber. Interestingly, an exothermic process started at ≈1150 °C, which subsequently resulted in an increase in the temperature difference from ≈ −2.25 °C/mg to ≈ −1.75 °C/mg when the environmental temperature increased from 1150 °C to 1400 °C. This result has been also reported in other oxide fibers, like mullite fiber [17], and implies some microstructural transitions of the AS fiber triggered in this temperature regime.

XRD was employed to ascertain the microstructure of the AS fiber as a function of temperature. As shown in Fig. 3, the as-received AS fiber was less crystallized due to the existence of amorphous SiO_2 inside the fiber, therefore, only five weak and broad crystalline peaks of γ-$A1_2O_3$ were observed in the XRD pattern. They corresponded to the (220), (311), (222), (400) and (440) crystal planes of the γ-Al_2O_3 phase, located at 2θ ≈32.7°, 37.2°, 39.7°, 45.6° and 67.3°, respectively. This microstructural feature was maintained up to a critical temperature (1150 °C), at which an orthorhombic structured mullite phase was formed. Since the composition of mullite phase is generally between $3Al_2O_3{\cdot}2SiO_2$ and $2Al_2O_3{\cdot}3SiO_2$, the formation mechanism could be related to the phase reaction of amorphous SiO_2 and crystallized γ-$A1_2O_3$ [7,23]. Detailed phase reaction is:

$$3Al_2O_3 + 2SiO_2 \rightarrow 3Al_2O_3{\cdot}2SiO_2 \quad (1)$$

This also explains the exothermic process of the AS fiber found at ≈1150 °C based on the DSC curve. Note the phase transition was still incomplete at 1150 °C, because γ-$A1_2O_3$ phase was also found in the AS fiber heat-treated at this temperature. Complete phase transition of AS fiber was achieved after ≥1300 °C heat-treatment.

The intensity of mullite phase in XRD patterns was temperature dependent. As also shown in Fig. 3, when the temperature increased from 1150 °C to 1400 °C, the characteristic peaks of mullite, like (101), (240) and (242) planes at 2θ ≈15.5°, ≈26.3° and ≈40.9°, were much sharper and stronger. This demonstrates a higher crystallinity nature of the mullite phase, which is presumably correlated to the grain growth. Since the structural composition of mullite phase is varied with temperature [24–26], the lattice parameters evolved with temperature were estimated from the obtained XRD patterns [24]. For the