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Effects of sintering temperature on mechanical properties of 3D mullite fiber (ALF FB3) reinforced mullite composites

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Abstract

A new method to weaken the interfacial bonding and increase the strength of 3D mullite fiber reinforced mullite matrix (Mu_f/Mu) composites is proposed and tested in this paper. Firstly, Mu_f/Mu composites were fabricated through sol–gel process with varied sintering temperature. Then, the effects of sintering temperature on mechanical properties of the composites were tested. As sintering temperature was raised from 1000 °C to 1300 °C, the three-point flexural strength of the composites firstly decreased from 66.17 MPa to 41.83 MPa, and then increased to 63.17 MPa. In order to explain the relationship between composite strength and sintering temperature, morphology and structure of the mullite fibers and mullite matrix after the same heat-treatment as in the fabrication conditions of the composites were also investigated. Finally, it is concluded that this strength variation results from the combined effects of matrix densification, interfacial bonding and fiber degradation under different sintering temperatures.

Keywords: A. Sol–gel process; C. Mechanical properties; Sintering temperature; Mu_f/Mu composites

1. Introduction

Continuous oxide fiber-reinforced oxide ceramic matrix composites have attracted significant scientific and technological interest in high temperature applications for their excellent properties such as high fracture toughness, damage tolerance, thermal shock and oxidation resistance [1,2]. As one of the most promising oxide ceramic, mullite ($3Al_2O_3 \cdot 2SiO_2$) ceramic has good chemical and high thermal stability, low thermal expansion coefficient (4.5×10^{-6} C^{-1}) and conductivity (0.06 W cm^{-1} K^{-1}), low dielectric constant ($\varepsilon \approx 7$), and high creep resistance. However, its application is largely limited by the low fracture toughness (about 2.2 MPa $m^{-1/2}$) [3–5]. One way to overcome this limitation is incorporating high-strength continuous ceramic fibers into mullite matrix, and then the activated debonding, delamination, crack deflection, fiber bridging and fiber pull-out mechanisms would lead to a non-linear stress–strain response and high fracture energy [6].

Nevertheless, Mu_f/Mu composites usually have low strength due to the strong interfacial bonding originated from the reaction between fibers and matrix, and the degradation of mullite fibers at high temperatures during the fabrication process [7,8]. Two common ways to weaken the interfacial bonding and thus increase the strength of Mu_f/Mu composites are: (1) introducing interphases or fiber-coating with low toughness, e.g. BN [9], fugitive carbon [10], monazite [11], or porous ZrO_2 [12], by chemical dip-coating or CVD method; (2) adopting porous matrix [13]. Fiber-coating is generally more complex and costly, and it is much more difficult for near-net-shape manufacturing, while the implementation of porous matrix is simpler, more convenient, efficient and economical.

In this paper, we propose a new method to weaken the interfacial bonding–a new diphasic mullite precursor was introduced, and the densification degree of mullite matrix was controlled by varying sintering temperature. The effects of

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Table 1
Properties of ALF mullite fibers.

Type	Diameter (μm)	Density (g/cm^3)	Tensile strength (MPa)	Strength retention (1300 °C, %)	Chemical content (wt%)	Phase content	Highest using temperature (°C)
FB3	10	3.0	1.75	~50	Al_2O_3: 72 SiO_2: 28 B_2O_3:2.0	γ-Al_2O_3, amorphous SiO_2	1400

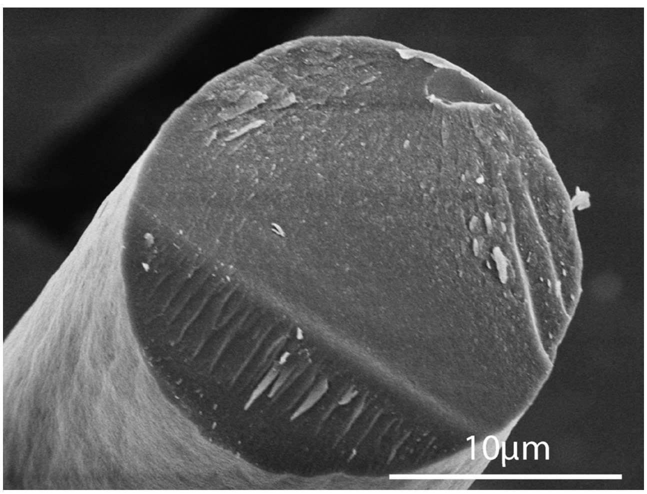

Fig. 1. Fracture surface of the mullite fiber.

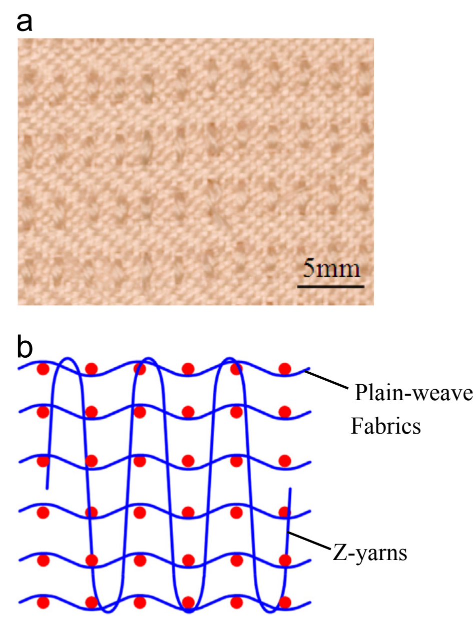

Fig. 2. Photograph (a) and structure (b) of 3D-sewed mullite fiber preform.

sintering temperature on mechanical properties of the composites were tested and discussed in this paper.

2. Experimental procedure

2.1. Materials processing

The reinforcements used to prepare Mu_f/Mu composites were twill-woven mullite fiber fabrics (ALF 3025 T-FB3, from Nitivy ALF Company, Japan), and the properties of the ALF mullite fibers are listed in Table 1. Fracture surface of the mullite fiber is shown in Fig. 1, which demonstrates that the mullite fiber has a circular cross-section, with a diameter of approximately 10 μm. The photograph and structure of the 3D-sewed mullite fiber preform with a fiber volume fraction of about 41.5% are shown in Fig. 2.

Diphasic mullite sol, the precursor of the mullite matrix, was prepared by blending a silica sol and an alumina sol (both from Snowchemical S&T Co., LTD, China) in proportion to their contents in the chemical composition of $3Al_2O_3 \cdot 2SiO_2$ (3/2-mullite) through mechanical stirring lasting for 0.5–1 h. Properties of the silica and alumina sol are listed in Table 2. The density and viscosity of the mullite sol are 1.15 g/cm^3 and 6 mpa s, respectively. The ceramic yield is about 24.1 wt% at 1300 °C and pH is 5.0.

As shown in Fig. 3, the composites were fabricated via sol–gel process, which included vacuum infiltration, gelation and

Table 2
Properties of silica and alumina sol.

Type	SiO_2 (wt%)	Al_2O_3 (wt%)	pH	Density (g/cm^3)	Ceramic yield (1300 °C, wt%)
Silica sol	24.90	–	4.43	1.12	25.50
Alumina sol	–	20.60	4.00	1.15	17.80

sintering processes. Firstly, the 3D-sewed preforms were vacuum impregnated with mullite sol for 6 h, and then the preforms were gelated at 80 °C for 10 h. Finally, the dried preforms were heated at a rate of 10 °C/min and sintered at 1000 °C, 1200 °C and 1300 °C for 1 h in Ar atmosphere. This sol–gel process was repeated 12 times to densify the composites. The sintering behavior of the mullite matrix was investigated by cold-compacting dried gel powders under the pressure of 100 MPa into Φ40 × 6 mm cylinder samples, which was then sintered at the composites fabrication temperature for 1 h. The mullite fibers having been heat-treated at the composite fabrication temperature for 1 h were also investigated.