

# Synthesis of spherical (30 nm) and rod-like (200 nm) zirconia co-reinforced mullite nanocomposites

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## Abstract

The objective of this research is to study microstructure and phase evolution of zirconia–mullite (ZM) nanocomposites which were synthesized by the direct transformation from amorphous precursor monoliths. The monolithic precursors were heat treated at 950–1250 °C to obtain ZM composites. Scanning electron microscopy (SEM), energy-dispersive spectroscopy (EDS), transmission electron microscopy (TEM) and high resolution TEM (HRTEM) were employed to investigate the microstructure of the composites. A unique nano- and submicron-zirconia co-reinforced mullite composite microstructure was obtained after a controlled crystallization. The nano tetragonal zirconia (t-ZrO<sub>2</sub>) grains (< 50 nm) were spherical and embedded in the mullite lattice. The unconfined zirconia grains showed a preferred growth direction along <1 0 0> direction of t-ZrO<sub>2</sub>, and formed submicron grains (< 650 nm) with bar-like shape. The precursor derived ZM composites also demonstrated enhanced mechanical properties comparing with the conventional powder processed ZM composites.

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## 1. Introduction

Over the past decades, mullite has been extensively investigated due to its outstanding engineering properties, such as high melting point, good chemical and thermal stability, excellent high-temperature strength, high creep resistance and high corrosion stability [1–6]. However, its low toughness and susceptibility to flaw have greatly restricted its industrial applications as advanced structural ceramic. In order to reduce its inherent brittleness by improving toughness, numerous researches have been carried out to synthesize mullite-based composites by introducing other ceramics components, such as zirconia, silicon carbide and alumina etc. [4,6–12]. Among these composites, mullite–zirconia composite has attracted the

widest attention because of its excellent fracture toughness, high hardness and strength at room temperature.

Several toughening mechanisms related to martensitic transformation (from tetragonal (t-ZrO<sub>2</sub>) to monoclinic (m-ZrO<sub>2</sub>)) are the origins why zirconia is included into various ceramics matrices as a toughening agent [13,14]. Various oxides, such as CeO<sub>2</sub>, Y<sub>2</sub>O<sub>3</sub>, MgO and CaO etc., were studied to preserve tetragonal zirconia to room temperature by forming zirconia solid solution to enhance the toughening efficiency of ZrO<sub>2</sub> [15–18]. Moreover, secondary oxides could also improve the strength of ceramics through depressing m-ZrO<sub>2</sub> formation *via* the refining of ZrO<sub>2</sub> grains [19,20]. Apart from doping additives, ceramic manufacturing routes also played a key role on the properties control of ZM composites [6,21–24]. For instance, regarding to the densification techniques, recently developed spark plasma and microwave sintering could provide high energy within seconds. Those rapid sintering process can achieve the purpose of inhibiting the abnormal

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grain growth and enhancing the mechanical properties by promoting the reaction rate [25,26].

It is well accepted that the mechanical properties of a material largely depend on its microstructures. Optimized microstructures, such as fine grains, low porosity, low abnormality etc. are usually required for ceramics to obtain enhanced strength and toughness. Many studies have shown that introducing nano sized phases to form nanocomposite materials could be an effective way to significantly enhance the mechanical properties of ceramics [27,28]. This is due to the toughening mechanisms existing in the smaller scale of second phase particles. Conventional powder-processed ZM ceramics, restricted by composition homogeneity of starting powder, are very difficult to obtain nano-sized matrix grains and/or second phase particles (zirconia) [11,18,29,30]. The normally generated micro- and sub-micro grains are not the supreme microstructure for acquiring ceramics with excellent mechanical properties (e.g., high strength and good fracture toughness). In contrast, the homogenous pre-ceramic precursor derived composite ceramic could be easily produced with nano-crystalline microstructures by controlling certain parameters [31–34].

In this manuscript, we report a simple and feasible method to fabricate novel nano/sub-micro ZM composite ceramic through a direct transformation of bulk amorphous precursor. The precursors could be easily mechanically processed into required shapes and which can be maintained in the following heat treatments. The mechanical properties, phase development and microstructure evolution of the ZM composites crystallized at different conditions were investigated in this work. Furthermore, based on the observations, a crystallization mechanism was proposed to elucidate the generation of the unique microstructure.

## 2. Experimental procedure

### 2.1. Samples preparation

To fabricate ceramic precursor, the commercial available oxide powders were mixed first and homogenized by a planetary ball milling with zirconia balls at 100 rpm for 10 h. The batch powder was consisted of 51% of SiO<sub>2</sub>, 30.5% Al<sub>2</sub>O<sub>3</sub>, 9.5% ZrO<sub>2</sub>, 5.8% MgO and 2.7% CaO (molar fractions). CaO and MgO powders were added to decrease the melting temperature of mixture and as the partial stabilizer for ZrO<sub>2</sub> [19]. Then the mixed powder was dried in an oven at 80 °C and then fused in a corundum crucible in an electrical furnace (Si–Mo rod heater) at the temperature around 1700 °C. To generate amorphous pre-ceramic monoliths, the homogeneous flux was poured into a designed steel mold and quenched in liquid nitrogen. The as-made precursor was transparent and maintained in the shape of the mold. No cracks were formed after quenching. Afterward, the amorphous bulks were heated to

950 °C at a rate of 10 °C/min and kept for 2 h, and then heat treated at higher temperature (1100–1250 °C, 5 °C/min) for two more hours to finish crystallization. The samples were named as ZM-X, where ZM means zirconia–mullite ceramics, and X stands for the crystallization temperature. For example, the sample ZM-1150 is zirconia–mullite ceramic pre-treated at 950 °C and crystallized at 1150 °C.

### 2.2. Characterization

XRD profiles were collected by a X-ray diffraction using Japanese D/MAX 2500VB diffractionmeter equipped with Ni-filtered Cu-K<sub>α</sub> radiation at a scan rate of 2°/min and a step size of 0.02°. The volume fractions of tetragonal zirconia ( $V_t$ ) were calculated by the following Eqs (1)–(3) [35].

$$V_t = 1 - V_m \quad (1)$$

$$V_m = \frac{PX_m}{1 + (P-1)X_m} \quad (2)$$

$$X_m = \frac{I_{m(\bar{1}11)} + I_{m(111)}}{I_{m(\bar{1}11)} + I_{m(111)} + I_{t(101)}} \quad (3)$$

$V_m$  is the volume fraction of m-ZrO<sub>2</sub>.  $X_m$  is the integrated intensity ratio and  $P=1.340$ .  $I_m$  and  $I_t$  are the peak intensities of m-ZrO<sub>2</sub> and t-ZrO<sub>2</sub>, respectively.

Fracture toughness was determined for all the samples using a Vicker's hardness tester at a load of 9.8 N (Micro-Duromat 4000). The Eq. (4) was used to calculate the fracture toughness [36].

$$K_{IC} = 0.0319 \frac{P}{a l^{1/2}} \quad (4)$$

$P$  is the indenter load (N).  $a$  is the indentation half-diagonal length (m) and  $l$  is the average length of the generated cracks (m).

Flexural strength was obtained using a three-point bending on 3 mm thickness, 4 mm width and 35 mm length, with a 30 mm span. The crosshead of the test is 0.5 mm per minutes (CSS-44100 tensile machine). The quenched glass precursors were mechanically processed into 3 × 4 × 35 mm direct after quenching and then crystallized for the bending test. The secondary electron and back scattering electron (BSE) images were acquired using a Siri-on200 microscope. Platinum was coated on the samples before the observation. TEM images and the EDS patterns were taken utilizing a Tecnai G220 S-TWIN instrument operated at 200 kV. The HRTEM images were obtained using a JEOL 3010 microscopy operated at 300 kV. A Gatan 691 precision ion polishing system was employed to prepare TEM samples.

## 3. Results

### 3.1. Phases and mechanical properties

The quenched ceramic precursor was amorphous, suggested by its XRD curve in Fig. 1. The precursor bulks

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