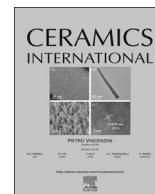




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Low-temperature sintering of coarse alumina powder compact with sufficient mechanical strength

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ABSTRACT

Coarse alumina powder compacts doped with various amounts of titania and copper oxide were pressurelessly sintered from 900 °C to 1600 °C. Their phase assemblages and microstructural evolution, as well as their properties, were investigated by X-ray diffraction (XRD), scanning electron microscopy (SEM), differential scanning calorimetry/thermogravimetric (DSC/TG) analysis, and three-point bending and wetting test. The role of TiO₂ and CuO during the sintering is discussed in detail. The experimental results show that the liquid phase from the copper oxide appeared at approximately 1200 °C, so the solid-state reaction between alumina and titania took place at a lower temperature. Such solid state-reaction sintering had a strong impact on the grain growth and greatly promoted the densification of the alumina compact. In addition, the liquid phase inhibited the abnormal grain growth and microcracking. As a result, the coarse alumina powder compacts doped with 5 wt% TiO₂–CuO were fully densified and exhibited sufficient flexural strength (342 ± 21 MPa) when sintered at a temperature of 1450 °C for 2 h.

1. Introduction

Alumina (α-Al₂O₃) is one of the most widely used ceramic materials owing to its excellent properties such as refractoriness, chemical stability, and wear and deformation resistance [1]. However, it is still a challenge to develop a simple and reliable process to fabricate low-cost, high-performance Al₂O₃ compact for commercial use. Because of strong Al–O ionic bonding, previous pressureless sintering of Al₂O₃ compact using coarse powders has required high sintering temperatures of as much as 1700–1800 °C [2], which consumes a lot of energy. Although high-density Al₂O₃ compact can be achieved using nanopowders at 1450 °C [3], such nanopowders are expensive and the synthesis process is complex.

Generally, in order to decrease the sintering temperature of Al₂O₃ compact, solid state-reaction sintering is widely used [4–6]. For example, the formation of aluminum titanate through a solid-state reaction of Al₂O₃ and titania (TiO₂) was introduced [7]. The appearance of aluminum titanate increases the vacancy concentration and accordingly accelerates the rate of mass transfer [8,9]. More importantly, as the secondary phase, aluminum titanate decreases the Young's modulus and thermal expansion coefficient of the Al₂O₃ matrix, improving the thermal shock resistance of the resulting Al₂O₃ compact [10]. Thus, TiO₂ is an appropriate and frequently used

additive for low-temperature sintering of alumina compact [11–15]. However, because of the particular characteristics of the Al₂O₃–TiO₂ reaction process [16], the microstructures of such ceramics usually exhibit abnormal grain growth and extensive microcracking [17], which reduces the flexural strength of the ceramics and limits their application.

In the present work, the combination of TiO₂ and copper oxide (CuO) is proposed as a sintering additive for Al₂O₃ compact because a low-temperature eutectic has been known to exist at 1096 °C in the Al₂O₃–CuO system [18]. In addition, co-doping of Ti⁴⁺ and Cu²⁺ is known to promote the solubility of both Ti⁴⁺ and Cu²⁺ as a result of mutual charge compensation [19]. This may have some positive impact on the densification of Al₂O₃ compact, lowering its sintering temperature. Furthermore, the appearance of a liquid phase during sintering may alleviate the abnormal grain growth and extensive microcracking. Therefore, in this study, to determine the optimal TiO₂–CuO content in the Al₂O₃ compact and reveal the role of TiO₂ and CuO in the sintering, coarse Al₂O₃ powders doped with different amounts of TiO₂ and CuO were pressurelessly sintered at different temperatures. The phase assemblages and microstructural evolution of the samples, as well as their mechanical properties, were systematically investigated and are discussed in detail.

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2. Experimental

2.1. Preparation of starting powders

The starting materials were of analytical grade: Al_2O_3 powders (Chinasun Specialty Products Co., LTD., Jiangsu, China, average particle size $64\ \mu\text{m}$), TiO_2 powders (Tianjin Fuchen Chemical Reagents Factory, Tianjin, China, average particle size $2\ \mu\text{m}$), and CuO powders (Tianjin Fuchen Chemical Reagents Factory, Tianjin, China, average particle size $1\ \mu\text{m}$). Mixtures of TiO_2 and CuO were prepared at a weight ratio of 4:1 (this ratio was based on preliminary experiments). The Al_2O_3 powders, TiO_2 - CuO powders, and polyvinyl alcohol (PVA, 3 wt%) were mixed with ethanol (the weight ratio of ethanol/powders=4). The TiO_2 - CuO contents were 1–8 wt%, respectively. The mixtures were milled in a planetary ball mill (QM-ISP4-CL, Instrument Plant of Nanjing University, China) with Al_2O_3 balls (the weight ratio of ball/powders=7). To prevent the Al_2O_3 powders from being excessively crushed, the ball mill was operated at 150 rpm for 1 h. After milling, the slurry was dried at $60\ ^\circ\text{C}$ for 12 h in air. The resulting powders were then placed in an agate mortar and ground. Finally, the powders were sieved through a 60-mesh sieve. The morphology of the milled Al_2O_3 powders was observed by scanning electron microscopy (SEM, QUANTA 200FEG, FEI Company, USA), and the particle-size distribution of the obtained Al_2O_3 powders was measured with a laser scattering particle-size analyzer (BT-9300S, Bettersize, China), as shown in Fig. 1. The average size of the obtained Al_2O_3 powders was $23\ \mu\text{m}$.

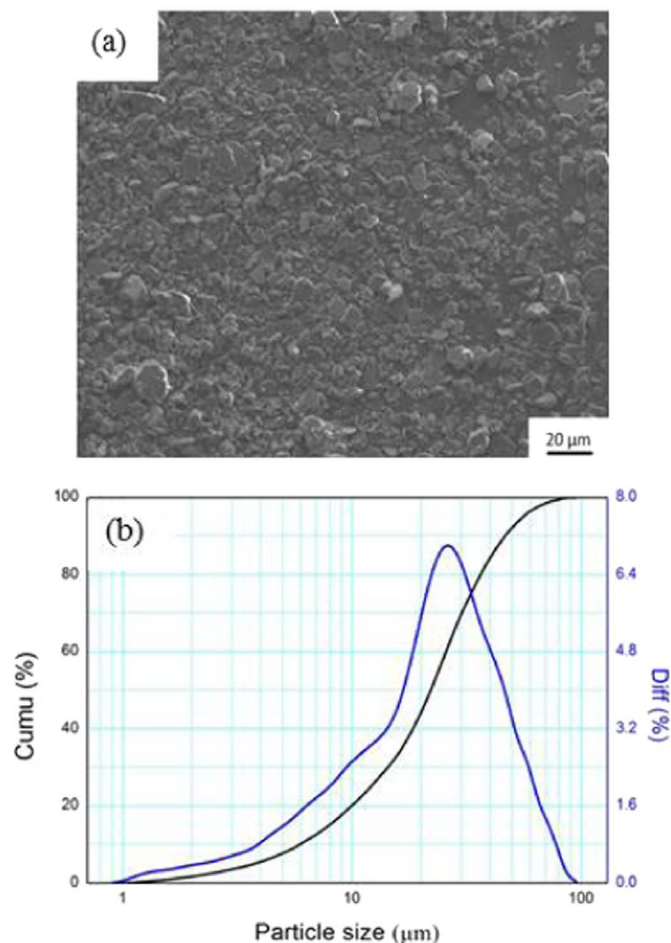


Fig. 1. SEM micrograph and particle size distribution of the milled alumina powders.

2.2. Sintering of Al_2O_3 compact

Cylindrical green bodies (15 mm in diameter) and rectangular green bodies (dimensions of $5\times 6\times 45\ \text{mm}$) were made from the obtained powders. For each green body, the obtained powders (1.5 g for a cylindrical green body and 3 g for a rectangular green body) were individually packed to form pellets by uniaxial pressing at 40 MPa. The samples were then pressurelessly sintered in a muffle furnace in air. For comparison, three identical cylindrical samples and five identical rectangular samples were put into the muffle furnace each time. The samples were sintered from $900\ ^\circ\text{C}$ to $1600\ ^\circ\text{C}$ for 2 h at a heating rate of $10\ ^\circ\text{C}/\text{min}$ up to $1000\ ^\circ\text{C}$ and at $5\ ^\circ\text{C}/\text{min}$ from $1000\ ^\circ\text{C}$ to $1600\ ^\circ\text{C}$. In addition, pure Al_2O_3 and Al_2O_3 doped with 5 wt% TiO_2 were also sintered from $900\ ^\circ\text{C}$ to $1600\ ^\circ\text{C}$ for 2 h.

2.3. Characterization of sintered Al_2O_3 compact

Thermal analysis was performed using a differential scanning calorimetry/thermogravimetric (DSC/TG) instrument (NETZSCH STA449C/3/MFC/G, Germany). The experiments were carried out in Al_2O_3 crucibles at a heating rate of $10\ ^\circ\text{C}/\text{min}$ in air up to a final temperature of $1350\ ^\circ\text{C}$.

The phase assemblages of the samples heated to different temperature were characterized by X-ray diffraction (XRD) (X'Pert Pro, PANalytical Co., Holland) using graphite monochromatized $\text{Cu K}\alpha$ radiation ($\lambda=0.15418\ \text{nm}$). The tube voltage was 40 kV, the tube current was 40 mA, the step size of the 2θ angle was 0.0330° , the scan step time was 10.160 s, and the 2θ angle scan ranged from 10° to 90° .

The polished samples were thermally etched at $100\ ^\circ\text{C}$ lower than the sintering temperature in air for micrograph observation. A field-emission environmental scanning electron microscope (SEM) (QUANTA 200FEG, FEI Company, USA) equipped with an energy-dispersive X-ray spectrometer (EDS, EDAX Genesis) was employed to observe the morphology of all samples.

The relative density of the sintered samples was measured by the Archimedes method using distilled water as the liquid medium. The sintered rectangular samples were machined into dimensions of $4\times 3\times 36\ \text{mm}$, with one side polished. The flexural strength of the processed rectangular samples was measured with an electronic digital control system (INSTRON-5567, Co. Instron Engineering Corporation, USA) using the three-point bending test at room temperature. The span length was 30 mm and the loading speed was $0.5\ \text{mm}/\text{min}$.

The wetting behavior of CuO on an Al_2O_3 plate was analyzed using the sessile drop method. A polished Al_2O_3 plate ($\Phi 60\times 5\ \text{mm}$) with a relative density of 99.13% was used as the substrate for the wetting test. The CuO powder was first sonicated with ethanol for 10 min. After it was dried, crushed, and sieved, the powder was compacted into spheres 10 mm in diameter. Finally, the CuO sample was put on the Al_2O_3 plate and heated to the melting point of CuO in a muffle furnace (Nabertherm GmbH P330, Germany) at a heating rate of $10\ ^\circ\text{C}/\text{min}$ in air. The wetting behavior was observed through images taken by a camera, whereas the contact angles were measured using drop shape analysis software. After the wetting test, the plate was cut in order to observe its cross section.

3. Results and discussion

3.1. Thermal analysis

The results of the DSC/TG analysis of Al_2O_3 samples doped with 5 wt% TiO_2 - CuO are presented in Fig. 2. The TG curve shows the first mass loss of 4.1 wt% below $250\ ^\circ\text{C}$, with an endothermic peak at $95\ ^\circ\text{C}$ in the DSC curve, which indicates the removal of water. The second mass loss of 2.8 wt% occurs between $250\ ^\circ\text{C}$ and $900\ ^\circ\text{C}$. This mass loss corresponds to the decomposition of PVA. However, there is no endothermic peak in the DSC curve in this temperature range owing

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