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Influence of yttria additives on structural, microstructural and mechanical properties of alumina–zirconia composites prepared by two-stage sintering

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Abstract

The effect of the addition of 2–8 wt% Y_2O_3 on the structural evolution, microstructural development and mechanical properties of $0.50Al_2O_3$–$0.50ZrO_2$ ceramics was determined. The present study has been performed to understand and control the densification behaviour with optimal Y_2O_3 addition. The phase composition of the samples was characterized by the X-ray diffraction (XRD) technique. Refinement of the lattice parameters using Rietveld analysis revealed the quantitative phases of the alumina–zirconia–yttria composites.

Keywords: B. Composite; Alumina; Zirconia; XRD

1. Introduction

Alumina–zirconia (Al_2O_3–ZrO_2) composites have excellent strength, toughness, heat-shock and anti-wear properties as well as oxidation resistance. These properties make alumina–zirconia ceramics suitable for a variety of high demand applications including dental screws, cutting blades, electrosurgical insulators, valve seals, body armor, pump components, oxygen sensors, dies and prosthesis components such as hip joints [1,2]. Therefore, the alumina–zirconia system (AZx with 15–50 mol% ZrO_2) is interesting to investigate [3–5]. Zirconia grains embedded in an alumina matrix enhanced the flexural strength, fracture toughness and fatigue resistance. The toughening mechanisms identified in zirconia–reinforced alumina ceramic are attributed to the stress induced phase transformation of the metastable tetragonal grains towards the monoclinic symmetry ahead of a propagating crack, which leads to an increase in the work of the fracture [6]. Common zirconia is therefore doped with Y_2O_3, CaO, Sc_2O_3 and other oxides to stabilize its tetragonal or cubic phase. Owing to a transformation strengthening mechanism the polycrystalline tetragonal zirconia (t-ZrO_2) reveals the highest fracture toughness among advanced ceramics and has been widely used in structural applications [7]. In addition, the metastable tetragonal zirconia without dopants can also be prepared in the form of fine particles that are smaller than the critical size [8–10]. However, to improve the mechanical properties of structural ceramics, it is desirable to achieve microstructures with nearly full density and fine grains with a homogeneous distribution. One of the ways of eliminating grain growth in the final stage of sintering is a two-stage sintering process reported by Chen and Wang [11]. This was originally successfully applied to the densification of a nanometer-sized yittia powder without the final stage of grain growth.

Consequently, one approach to make a $0.50Al_2O_3$–$0.50ZrO_2$ composite powder is synthesis using the solid state reaction process. $0.50Al_2O_3$–$0.50ZrO_2$ composite ceramics with different Y_2O_3 amounts added were prepared using two stages sintering to optimize the ceramics' properties. The present work investigates the influence of dopants known to influence densification, phase formation, phase analysis, microstructure and mechanical properties.

2. Experimental

$0.50Al_2O_3$–$0.50ZrO_2$ ceramics with 2, 4, 6 and 8 wt% Y_2O_3 adding were prepared from Al_2O_3, ZrO_2 and Y_2O_3 (commercial grades) as precursors and isopropyl alcohol as a solvent. Sample powders were mixed and ball milled with ZrO_2 media under

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isopropyl alcohol for 24 h. After ball milling for 24 h and drying in an electronic furnace, the resulting powders were calcined at 1200 °C for 2 h. The green bodies were prepared by uniaxial pressing of the powders at 3 MPa to form disks. The sintering experiments were carried out in an electrical furnace (Nabertherm, Germany). For the two-stage sintering regime the temperature T_1 was obtained by heating the samples at 10 °C/min up to 1550 °C with a hold time of 3 min, and they were cooled down to T_2 at 1300 °C with a hold time of 10 h. Finally, the specimens were cooled to room temperature at 10 °C/min. The bulk densities of the sintered samples were calculated using the Archimedes' method. The crystallized phase of the ceramic samples was measured by X-ray diffraction (XRD) using Cu-K_α radiation (Philips PW 1729 diffractometer, Netherlands). The crystalline size of the ceramics was determined from the X-ray line broadening using the Scherrer equation; $D = 0.9\lambda/B \cos\theta$ [12]. Phase analysis was calculated using the Rietveld method. Microstructural evolution of the ceramics was observed using scanning electron microscopy (SEM, JEOL, JSM 840A, Japan). The microhardness of the bulk ceramics was measured using a microscan from Vickers and Knoop (FM-700e type D, Future Tech., Japan).

3. Results and discussion

The XRD patterns of $0.50Al_2O_3$–$0.50ZrO_2$ ceramics with 2 to 8 wt% Y_2O_3 added after two-stage sintering at 1550 °C (T_1) for 3 min and 1300 °C (T_2) for 10 h are shown in Fig. 1, which presents the difference in content phases. Apart from the alpha (α)-Al_2O_3 and Y_2O_3, monoclinic (m), tetragonal (t) and monoclinic (m) Zirconia were identified. The main reflections in the pattern form around 2θ=25, 30, 35, 43, 50, 57 and 59° that match the characteristic reflections of Al_2O_3–ZrO_2 well [4,13,14]. The tetragonal phases and cubic phases of ZrO_2 were detected by the presence of a high intensity peak at 2θ=30° and the splitting of peaks around 2θ=35 and 50°. It

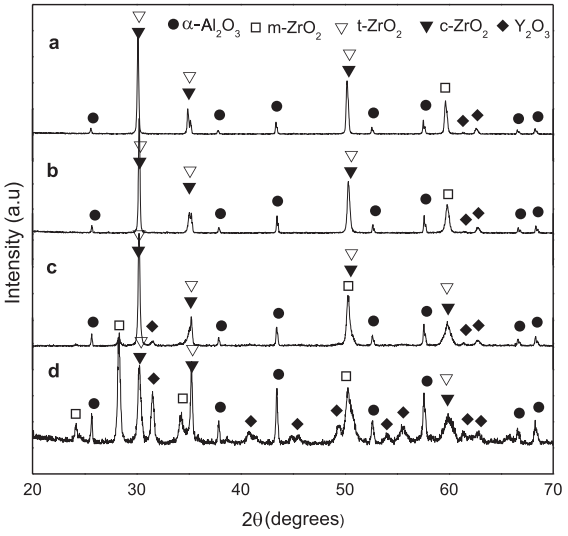

Fig. 1. X-ray diffraction patterns of $0.50Al_2O_3$–$0.50(Y_2O_3)$ ceramics with variation of Y_2O_3 using two stage sintering at 1550 °C (T_1) for 3 min and at 1300 °C (T_2) for 10 h (a) 2 wt% Y_2O_3, (b) 4 wt% Y_2O_3, (c) 6 wt% Y_2O_3, (d) 8 wt% Y_2O_3.

was found that at 2θ=50° the t-ZrO_2 phase and c-ZrO_2 phase transformed to complete the m-ZrO_2 phase with increasing Y_2O_3. The particle size of many zirconia may exceed the critical size and so a transformation t→m takes place. Aruna et al. reported that the stabilization of the tetragonal phase of zirconia at room temperature was due to its small crystalline size as well as constraining effect of the matrix phase of alumina [15]. Densities of the sintered samples were determined using the Archimedes principle. Table 1 contains data about the densities, average grain size from SEM and crystalline size from XRD of the $0.50Al_2O_3$–$0.50ZrO_2$ ceramics with different Y_2O_3 contents. It was observed that the density was between 4.59 and 4.76 g/cm^3. The maximum density was obtained in the samples of $0.50Al_2O_3$–$0.50ZrO_2$ ceramics with 6 wt% Y_2O_3 added. The promotion of densification by the addition of yittia in two-stage sintering is reflected by the lower temperature required to achieve a high final density than when using an un-doped method. In addition, the crystalline size of the alumina and zirconia phases calculated from the XRD patterns is summarized in Table 1. The crystalline sizes of Al_2O_3 are in the range 0.514–0.925 μm and for ZrO_2 they are in the range 0.618–0.870 μm. Fig. 2(a)–(d) shows the SEM/EDX micrographs of the as-received $0.50Al_2O_3$–$0.50ZrO_2$ ceramics with different Y_2O_3 contents, and they indicate typical microstructures. The microstructure of the sintered $0.50Al_2O_3$–$0.50ZrO_2$ with Y_2O_3 additive is presented in Fig. 2, where the white and gray color grains are the ZrO_2 and Al_2O_3 phases, respectively (as shown by arrows). The presence of Al_2O_3 and ZrO_2 grains in specimens was confirmed by EDX analysis. Microstructural characteristics were observed, i.e., uniform sized grains with well-packed and continuous grain structure. Almost no abnormal grain growth appeared. This supported previous work, in which the addition of ZrO_2 prevented abnormal grain growth in alumina ceramics [16]. By applying the linear intercept method [17] to these SEM images, grain sizes were estimated for these samples as given in Table 1. It can be seen that $0.50Al_2O_3$–$0.50ZrO_2$ ceramics with the addition of 2–8 wt%Y_2O_3 showed average grain sizes for Al_2O_3 in the range 0.651–0.892 μm, while the average grain sizes of ZrO_2 were in the range 0.692–0.915 μm. After performing a comparison of the grain sizes in Al_2O_3–ZrO_2 (Y_2O_3) ceramics after conventional sintering [14] and grain sizes in $0.50Al_2O_3$–$0.50ZrO_2$ (Y_2O_3) ceramics after two stage sintering, it was found that the sizes of the grains in $0.50Al_2O_3$–$0.50ZrO_2$ (Y_2O_3) ceramics after two-stage

Table 1
Densities, average grain size and crystalline size of $0.50Al_2O_3$–$0.50ZrO_2$ ceramics with different Y_2O_3 contents.

Contents of Y_2O_3 (wt%)	Density (g/cm^3)	Average grain size Al_2O_{3n} (μm)	Average grain size ZrO_2 (μm)	Crystalline size Al_2O_3 (μm)	Crystalline size ZrO_2 (μm)
2	4.59	0.651	0.720	0.804	0.870
4	4.71	0.783	0.692	0.925	0.773
6	4.76	0.892	0.841	0.514	0.618
8	4.75	0.867	0.915	0.540	0.720