

Preparation and characterization of Al₂O₃–25 mol% ZrO₂ composites

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Abstract

Al₂O₃–ZrO₂ (AZ_x), with 25 mol% ZrO₂ content, was prepared using the co-precipitation method. Synthesized powders were characterized by thermal reaction using a differential thermal analysis technique (TG–DTA) and were investigated by phase formation using X-ray diffraction. It indicated that the reaction occurred at 850 °C; cubic (c)-ZrO₂ phase and Al₂O₃ were obtained. By increasing temperature to 1100 °C, tetragonal (t)-ZrO₂ phase was detected. The Al₂O₃–25 mol% ZrO₂ was sintered for 2 h in the temperature range of between 1300 and 1600 °C. The majority phases of ceramics were m-ZrO₂ and α-Al₂O₃, although a t-ZrO₂ phase also appeared as a minor phase and decreased with higher temperature. Moreover, morphology and particle size evolution have been determined via the SEM technique. SEM showed that the particles of powder are agglomerated and basically irregular in shape. An SEM micrograph of ceramics exhibits uniform microstructure without abnormal grain growth. © 2011 Elsevier Ltd and Techna Group S.r.l. All rights reserved.

Keywords: A. Grain growth; A. Sintering; D. Al₂O₃; D. ZrO₂

1. Introduction

Al₂O₃ ceramics is one of the most widely used engineering ceramic materials due to its high elastic modulus, high wear resistance and chemical corrosion resistance, high temperature stability and the retention of strength at high temperatures [1]. It is well known that the mechanical properties of alumina ceramics can be considerably increased by the incorporation of fine zirconia particles [2]. Therefore, the alumina–zirconia system (AZ_x with 15–30 mol% ZrO₂) is interested to study [3,4]. As a structural oxide ceramic, zirconia has received much attention due to its high toughness while alumina is known as a hard, low-cost ceramics with high thermal conductivity [5,6]. The toughening mechanisms associated with zirconia toughened alumina are mainly based on the stress-induced transformation tetragonal-monoclinic martensitic transformation toughening and micro crack toughening. The extent of a stress-induced transformation toughening depends on the dispersion of tetragonal zirconia in an alumina matrix, its volume fraction and transformability. On the other hand, a uniform distribution of ZrO₂ ceramic matrix is an important

factor for optimization of micro crack nucleation-induced toughening [7,8]. The phase composition structure is the key to control material properties.

In this work, Al₂O₃–25 mol% ZrO₂ samples were prepared by a co-precipitation route and the influence of heat treatment on phase compositions and microstructure were investigated.

2. Experimental

The Al₂O₃–ZrO₂ powders with 25 mol% ZrO₂, respectively were synthesized using aluminum chloride (AlCl₃·6H₂O, 99.9% pure), zirconium oxychloride (ZrOCl₂·8H₂O, 99.9% pure) and ammonia solution (NH₄OH, 28%) as raw materials. Aluminum chloride and zirconium oxychloride were mixed and dissolved in distilled water. The solution was thoroughly stirred at room temperature, and then precipitated by adding ammonia to get Zr(OH)₄ gel. The precipitate was washed repeatedly using distilled water to remove all the chloride ions (as tasted by AgNO₃ solution), and then dried in air at 100 °C. The samples from the precipitate were investigated by thermal analysis using TA instruments (SDT Q600, USA) and after, they were calcined at different temperatures between 700 and 1100 °C. Then, various sintering conditions were employed by varying the sintering temperatures from 1300 to 1600 °C for 2 h. The crystallized phase of the Al₂O₃–ZrO₂ solid solution was

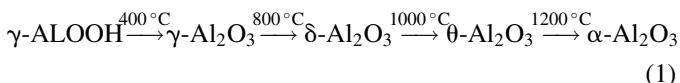
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measured by X-ray diffraction (XRD) using CuK α radiation (Philips PW 1729 diffractometer, Netherlands). Microstructural evolution of the powders and ceramics were observed using the scanning electron microscopy: SEM (JEOL, JSM 840A, Japan).

3. Results and discussion

Fig. 1 shows the TG and DTA curves of the Al₂O₃–ZrO₂ (AZ_x) mixed powders, with $x = 25$ mol% ZrO₂ content. It can be seen that the TG curve demonstrates three distinct weight losses below 600 °C. The first weight loss occurs below 230 °C, the second between 240 °C and 290 °C and the third above 300 °C. In the temperature range of room temperature to ~ 150 °C, the sample shows an endothermic peak in the DTA curve at ~ 100 °C, which relates to the first weight loss. This DTA peak is attributed to the elimination of water. The second weight loss between 240 °C and 290 °C were observed, which corresponded to endothermic peak at 282.50 °C on DTA curve. Previous works reported that the crystallization of t-ZrO₂ is obtained at 280 °C [7]. The third weight loss was found above 300 °C, and related to a small exothermic peak at 398 °C in the DTA curve. This result indicates the boiling point of the derivative product and corresponds to the transformation of Al(OH)₃ and γ -AlOOH into γ -alumina, consistent with other works [8]. The phase transformation from γ -AlOOH to α -Al₂O₃ follows the following path as reported by a number of workers [9].



The relationship between the TG curve and the DTA curve between 300 and 700 °C (Fig. 1) reveals a loss in the weight of raw material (TG), and a corresponding loss in the reaction of zirconium hydroxyl and aluminum hydroxyl (DTA). The DTA curve displays a sharp exothermic peak at 855 °C, which is the formation of a crystal line phase of Al₂O₃–ZrO₂ solid solution. Similar change in the crystallization temperature of Al₂O₃–ZrO₂ system were also observed by the same research group in

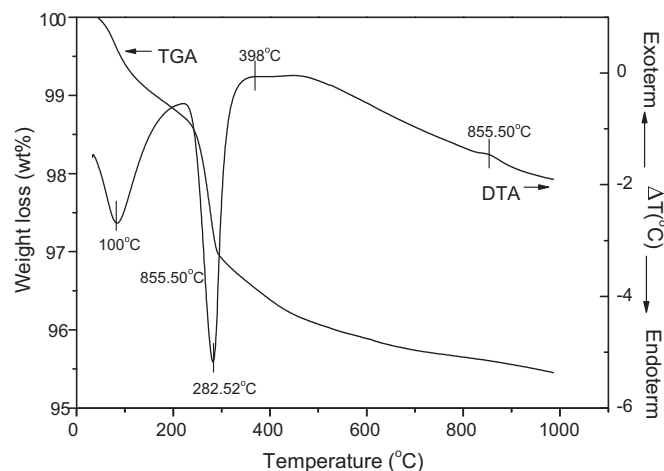


Fig. 1. A TG–DTA curve for the mixture of Al₂O₃–25 mol% ZrO₂ powders.

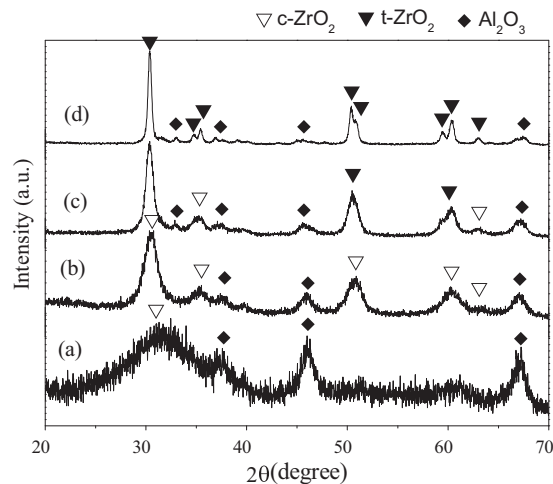


Fig. 2. XRD patterns of Al₂O₃–25 mol% ZrO₂ powders at various calcinations temperatures for 2 h: (a) 700 °C, (b) 850 °C, (c) 900 °C, and (d) 1100 °C.

earlier works [3,4,10,11]. These curve data were used to select the ranges of temperatures (700–1100 °C) for XRD investigation. To study the effect of heat treatment temperatures on Al₂O₃–25 mol% ZrO₂, the precursor powders were calcined between 700 and 1100 °C for 2 h. All calcined powders were examined by XRD in order to investigate the phase development. As shown in Fig. 2(a), phase formation of Al₂O₃–25 mol% ZrO₂ calcined at 700 °C. X-ray peaks of θ -Al₂O₃, δ -Al₂O₃, and very broad peaks of c-ZrO₂ phase are present, indicating incomplete reaction. So the sample was calcined at 850 °C and the resulting XRD patterns are shown in Fig. 2(b). Due to the broad peak observed at 855 °C (Fig. 1) and the clear intensity peaks of the c-ZrO₂, θ -Al₂O₃ and δ -Al₂O₃ phases (Fig. 2(b)) it was concluded that 850 °C would complete the Al₂O₃–ZrO₂ phase. XRD pattern in Fig. 2(b) exhibits very broad peaks confirming a small crystallize size. As the temperature increase to 900 °C, the phases present are θ -Al₂O₃, c-ZrO₂ and t-ZrO₂. At this temperature, t-ZrO₂ were appeared. Agreement with previous reported that alumina could stabilize the cubic zirconia (c-ZrO₂) and the tetragonal zirconia (t-ZrO₂) if the Al₂O₃–ZrO₂ powders were prepared by the aqueous co-precipitation route, starting with mixture of zirconium oxychloride and aluminum chloride [1]. In general, the c-ZrO₂ \rightarrow t-ZrO₂ phase transformation took place at about 900 °C confirming their phase and being consistent with other work [11]. When heating further to higher temperature (1100 °C), zirconia phases are composed mainly of t-ZrO₂. The presence of tetragonal phases of ZrO₂ is detected by the presence of high intensity peak at $2\theta = 30^\circ$ and splitting of peaks at around $2\theta = 50^\circ$. It is indicated the c-ZrO₂ phase transformed to complete t-ZrO₂ phase. From the little detection of θ -Al₂O₃ and δ -Al₂O₃ peaks, it was suspected that some Al₂O₃ phase still existed in an amorphous state even at 1100 °C and incompletely transformed to α -Al₂O₃. Fig. 3 shows the morphological evolution of all samples as a function of calcination conditions. For all the samples, the micrographs revealed foamy agglomerated particles. Powders calcined at 700 °C (Fig. 3(a)) shows more agglomeration state and irregular in

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