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Influence of Heat Treatment in Sintering Process on Characteristics of Al_2o_3-Zro_2 Ceramics Systems

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Abstract: Al_2O_3-ZrO_2 ceramics containing 5-50 mol% ZrO_2 were prepared by solid state reaction method. The bulk density, average grain size and microhardness of Al_2O_3-ZrO_2 ceramics system as function of ZrO_2 content were investigated. The dense Al_2O_3-ZrO_2 ceramics were successfully by means of carefully control processing parameters that include sintering temperature and heating/cooling rates. These ceramics with higher ZrO_2 content was obtained high bulk densities and small grain sizes. However, the high hardness values were exhibited from Al_2O_3-ZrO_2 ceramics with dopants ZrO_2 between 1-10mol% and its tend to decrease with concentration of ZrO_2. It can be found that the fast heating/cooling rates are controlled grain growth and obtained high hardness in materials. The results gave good correlation between stoichiometry characteristics, heating/cooling rates in sintering and microstructure of fabricated dense and mechanical properties.

Key words: Al_2O_3-ZrO_2 ceramics, microhardness, heating/cooling rates, sintering

INTRODUCTION

The most widely used ceramic materials are alumina, Al_2O_3 and zirconia, ZrO_2, because of their excellent bio-compatibility. The main advantage of Al_2O_3 is its high hardness and wear resistance, while ZrO_2 exhibits higher strength and fracture toughness, besides its lower Young' s modulus[1,2,3,4,5]. Tetragonal zirconia in alumina matrix is known as Zirconia Toughened Alumina (ZTA). ZTA is a high purity combination of the low cost of alumina and high strength of zirconia. Moreover, its is a ceramic-ceramic composite with good mechanical properties as shown by Aruna and Rajam[6]. Then ZTA ceramics are attractive materials due to the combination of both ZrO_2 and Al_2O_3 properties. The stoichiometry of ZTA is known to be an important factor for ensuring phase composition, mechanical properties and microstructure characteristics. To obtain stoichiometric ZTA, different preparative method have been introduced, such as hydrothermal[7], mixed oxide[8], and gel casting[9]. All these techniques are aim to improve properties of Al_2O_3-ZrO_2 ceramics. Recently, some researchers have focused their attention on optimal addition ZrO_2 into Al_2O_3 to enhance good mechanical property[10]. Moreover, they have found that the sinterability of the ceramics matrix is reduced when a large amount of second phase is added[11]. Therefore, in the present work focus on Al_2O_3-ZrO_2 ceramics systems difference stoichiometry which are prepared using solid state reaction of mixed oxide route. The effect of heating/cooling rates in sintering conditions on densification, grains size and hardness are investigated in this connection.

EXPERIMENTAL PROCEDURE

The Al_2O_3-ZrO_2 ceramics with (1-x) Al_2O_3-xZrO_2 where x = 0.5, 0.15, 0.25, 0.35, 0.45 and 0.50 were prepared from Al_2O_3 and ZrO_2 as precursors and isopropyl alcohol as solvent. All the six different batches were then ball milled with ZrO_2 media under isopropyl alcohol for 24 h. After ball-milling for 24 h, drying in electric furnaces, the resulting powders were calcined for 2 h at 1100°C with 5°C min^{-1} heating/cooling rate. Powders were uniaxial pressed at 3 MPa to form pellets. Sintering temperature was done at 1600°C for 2 h with heating/cooling rates from 1-10°C min^{-1}. The bulk densities of sintered sample were calculated using Archimedes's method. Microstructural analysis was examined by using Scanning Electron Microscopy (SEM) and Energy-Dispersive X-ray

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spectrometry (EDX) (JEOL JSM840A) on a polished surface of sintered samples. The average grain sizes of the ceramics were determined using the linear intercept method as suggested by Lee and Rainforth[12]. Hardness of bulk ceramics were measured using a microscan OD Vickers (Model MAT 24 Brooks).

RESULTS AND DISCUSSION

The densification data of sintered samples are shown in Table 1. Table 1 contains the information of densities of the ceramics. In general, the bulk density was found to slightly increase with heating/cooling rate but the densities were obviously tend to increase with x contents, which could be due to ZrO_2 concentrations.

It is observed that a density of between 3.97 and 4.84 g cm^{-3}. SEM micrographs of selection ZTA ceramics are shown in Fig. 1. In general, similar microstructural characteristics were observed in these samples, i.e., uniformly sized grains with a high degree of grain close-packing. By applying the linear intercept method as suggested by to these SEM images, grain sizes were estimated for these samples as given in Fig. 2.

It is obviously seen that heating/cooling rates are the important parameters for the development of ceramic microstructures. In that the average grain size decreases with increasing heating/cooling rate. It may be assumed that, the short heating in sintering process were inhibited growth of grain then in slow heating/cooling rates were obtained a large grains. Further increase in ZrO_2 contents lead to decrease in average grain size. This results indicates that ZrO_2 is to reduce the grain growth in Al_2O_3-ZrO_2 ceramics system and to improve the homogeneity of microstructure and being consistent with literatures[13,14].

Figure 3 shows the effect of heating/cooling rates on the hardness of Al_2O_3-ZrO_2 ceramics system. It was found that the nearly increase in hardness with fast heating/cooling rates. The maximum hardness of 0.68 MPa is obtained at heating/coolig rate of 10°C min^{-1} and showed the indenter impression and radial cracks in Fig. 4. In addition, it is observed that the high concentration of ZrO_2 were reduced the hardness of Al_2O_3-ZrO_2 ceramics. Thus, the optimal addition of ZrO_2 is an important parameter for development of

Table 1: Density of the (1-x) Al_2O_3-xZrO_2 ceramics from sintered various heating/cooling rates

Heating/ cooling rates °C/min	Densities (g cm^{-3}) x = 0.05	x = 0.15	x = 0.25	x = 0.35	x = 0.45	x = 0.50
1	3.97	4.13	4.27	4.45	4.65	4.84
3	4.15	4.16	4.31	4.46	4.58	4.71
5	4.01	4.18	4.25	4.49	4.62	4.82
7	4.02	4.14	4.31	4.48	4.64	4.72
10	4.24	4.21	4.32	4.45	4.68	4.75

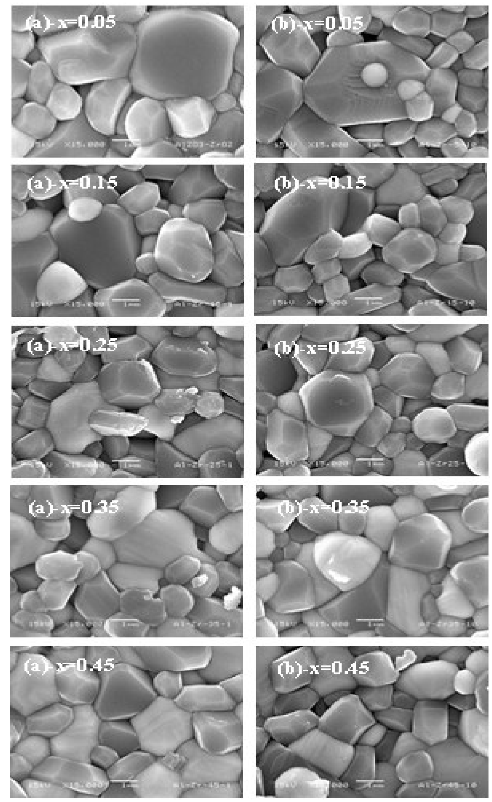

Fig. 1: The SEM images of as-received surfaces of (1-x) Al_2O_3-xZrO_2 ceramics sintered at 1600°C for 2 h with heating/cooling rate of (a) 1°C/min (b) 10°C/min

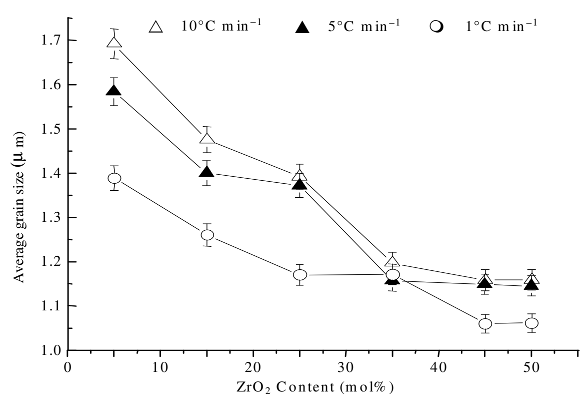

Fig. 2: Average grain sizes of Al_2O_3-ZrO_2 ceramics different ZrO_2 content from various heating/cooling rates sintering temperatures

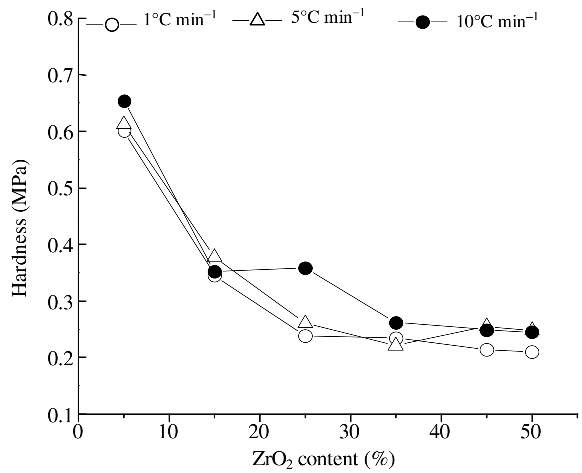

Fig. 3: Vickers hardness of the Al_2O_3-ZrO_2 ceramics with variation of ZrO_2 from sintered at different heating/cooling rates

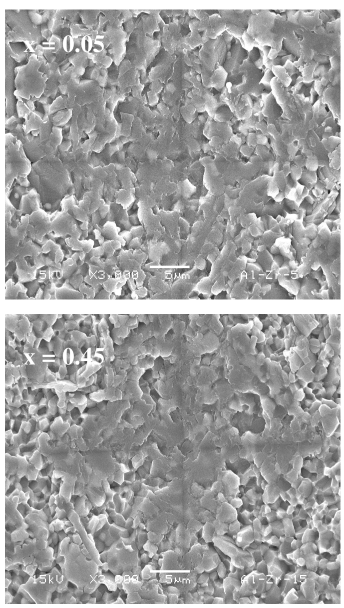

Fig. 4: The indented scar morphologies of Al_2O_3-ZrO_2 ceramics with variation of ZrO_2 from sintered at 1600°C with heating/cooling rates of 10°C min^{-1}

ceramics microstructure and mechanical properties, in agreement with other studies[13]. Corresponding EDX analysis and chemical compositions for some of these Al_2O_3-ZrO_2 ceramics system are shown in Fig. 5 and Table 2. It is seen that the Zr concentration increases with increasing ZrO_2.

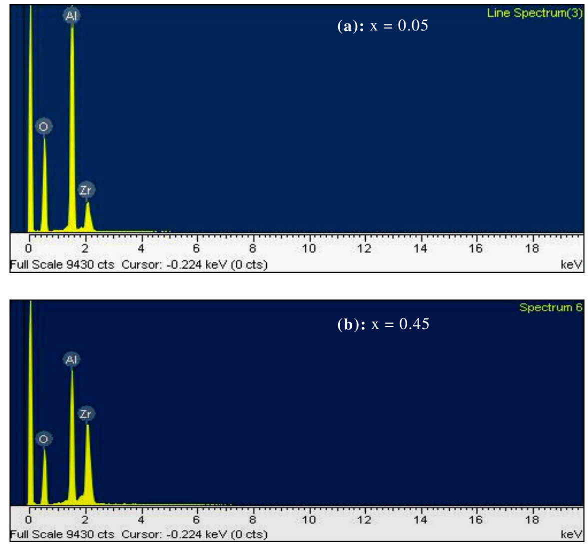

Fig. 5: EDX analysis of (1-x) Al_2O_3-xZrO_2 ceramics with variation of ZrO_2 contents from sintered at 1600°C for 2 h with heating/cooling rates of 10 °C min^{-1}

Table 2: Chemical compositions of the Al_2O_3-ZrO_2 ceramics system from EDX analysis

Content of ZrO_2 (mol%)	Compositions (at%) 1°C min^{-1} Al(K)	Zr(K)	O(K)	10°C min^{-1} Al(K)	Zr(K)	O(K)
5	28.92	3.40	67.68	28.92	3.40	67.68
15	28.01	3.82	68.17	30.27	3.83	65.90
25	23.12	8.01	68.87	25.81	6.03	68.16
35	25.75	8.89	65.35	22.68	7.82	69.50
45	19.75	9.74	70.51	19.06	9.96	70.98
50	19.68	10.18	70.14	19.10	9.89	71.04

CONCLUSIONS

The highly dense of Al_2O_3-ZrO_2 ceramics system were successfully. They possess microstructure and mechanical properties, which can be greatly varied by composition and heat treatment condition. The heating/cooling rates and compositions of ceramics are important parameter in controlling ceramics properties. It is found that the smaller grain size were obtained from the short time in fast heating/cooling rates, where high hardness were observed. A higher amount of ZrO_2 reduced grain sized and micro hardness value.

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