

# Centrifugal casting of $\text{Al}_2\text{O}_3$ –15 wt.% $\text{ZrO}_2$ ceramic composites

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## Abstract

Aqueous ceramic slurries with different solid contents (60, 70 and 78 wt.%) were prepared by mixing 85 wt.% low-temperature-sinterable high purity  $\alpha$ -alumina and 15 wt.%  $\text{ZrO}_2$  (3 mol%  $\text{Y}_2\text{O}_3$ ) in distilled water and a small amount of poly-carboxylic ammonium as the dispersion agent. Consolidation was performed by using a high-speed centrifugal casting process at 4000 rpm for 1 h. The result showed that the dispersion agent had a great influence on the viscosities of the slurries. It was also observed that the green and sintered densities of the  $\text{Al}_2\text{O}_3$ –15 wt.%  $\text{ZrO}_2$  composites increased with the solid contents. Phase segregation could be effectively eliminated when the solid content of the slurry was 78 wt.%.

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## 1. Introduction

During the last decade interest has been continuously increased in centrifugal casting with fine ceramic slurries [1–6]. Centrifugal casting is characterized by a powerful colloidal processing that leads to massive ceramic parts with high strength and high reliability [7–9]. Tashima et al. [7,8] used a low-temperature-sinterable high purity alumina as starting material to prepare green compacts by a high-speed centrifugal casting process, and the resulting alumina ceramics showed very high bending strength (1330 MPa) and excellent wear resistance. Kim et al. [6] reported that large alumina tubes with good properties were formed by a low-speed centrifugal casting. However, the drawback of the centrifugal casting process is related to the possible mass and/or phase segregation during the consolidation stage [1,10,11]. Fortunately, this disadvantage can be eliminated by starting with a coagulated [1] or flocculated [10] slurry or a highly concentrated slurry [12].

Although a number of investigations on centrifugally compacted single-phase ceramics, especially on alumina,

have been done under various conditions, studies on binary systems such as  $\text{Al}_2\text{O}_3$ – $\text{ZrO}_2$  composites are limited. The purpose of the present work was to prepare  $\text{Al}_2\text{O}_3$ –15 wt.%  $\text{ZrO}_2$  (3 mol%  $\text{Y}_2\text{O}_3$ ) composites via a high-speed centrifugal casting process and to investigate the effect of solid contents of the slurries on slurry viscosities, green and sintered densities, and phase segregation of the composites. For comparison, the  $\text{Al}_2\text{O}_3$ –15 wt.%  $\text{ZrO}_2$  (3 mol%  $\text{Y}_2\text{O}_3$ ) composite compacts were also prepared by cold isostatic pressing.

## 2. Experimental procedure

The starting materials were the  $\alpha$ -alumina powder (TM-DAR grade, particle size 0.2  $\mu\text{m}$ , purity of 99.99%, Taimei Chemical Co., Ltd., Japan) and TZ-3Y powder [ $\text{ZrO}_2$  (3 mol%  $\text{Y}_2\text{O}_3$ ), 28 nm, Tosoh Co., Ltd., Japan]. Aqueous ceramic slurries with different solid contents (60, 70 and 78 wt.%) were prepared by mixing 85 wt.% alumina and 15 wt.% TZ-3Y in distilled water and a small amount of poly-carboxylic ammonium (AH-103P, 43~45 wt.%, Dai-ichi Kogyo Seiyaku Co., Ltd, Japan) as the dispersion agent with a Conditioning Mixer (AR-360M, Thinky Co., Ltd, Japan) for 1 h at a rotation speed of 500 rpm and a revolution speed of 1500 rpm. The viscosities of the slurries were measured

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with a Digital Viscometer (DV-E, Brookfield Eng. Lab. inc, USA) at 100 rpm. These slurries were consolidated by a high-speed centrifugal casting machine (H-26F, Kokusan Co. Ltd., Japan) at 4000 rpm (centrifugal force: 3600 g) for 1 h in steel moulds with a diameter of 45 mm and a height of 115 mm. After centrifugation, the supernatant was poured off. The saturated, consolidated bodies in the moulds were placed at room temperature for 12 h and then dried in an oven at 40 °C for 24 h. After drying, the green compacts were released easily from the moulds and put in the oven again at 40 °C for 24 h. For comparison, the  $\text{Al}_2\text{O}_3$ –15 wt.% $\text{ZrO}_2$  (3 mol.%  $\text{Y}_2\text{O}_3$ ) composite samples were also prepared by uniaxial dry pressing in a steel die at 20 MPa and then followed by cold isostatic pressing at 200 MPa. Finally, those green compacts were sintered for 2 h in air at temperatures ranged from 1300 to 1450 °C with a heating rate of 10 °C/min.

Green and sintered densities were measured by the Archimedes method. Because of their irregular shapes, the green compacts were treated at 700 °C for 1 h with a heating rate of 5 °C/min before the measurement of green densities. In order to investigate phase segregation of samples prepared by centrifugal casting, three sintered pellets with a thickness of about 9 mm which were made from either 60 or 78 wt.% slurries were, respectively, ground to obtain the top, middle and bottom regions with a thickness of about 3 mm. After that, the densities and phase compositions of samples at different regions were determined. Phase compositions of the composites were examined by means of X-ray diffraction analysis using  $\text{CuK}_\alpha$  radiation in a Rigaku RAD-2C diffractometer. Surface morphology was observed by SEM on polished and thermally etched surfaces.

### 3. Results and discussion

The viscosities of  $\text{Al}_2\text{O}_3$ – $\text{ZrO}_2$  slurries with different amount of dispersion agent are listed in Table 1. As

expected, the viscosity of the slurry without any dispersion agent increased rapidly with increasing solid content from 70 to 78 wt.%. In this case, it was very difficult to pour such a slurry into the moulds. On the other hand, the viscosity of 78 wt.% slurry decreased dramatically from 5350~5400 to 850~900 mPas by adding 0.8 wt.% dispersion agent. However, the viscosity of the slurry increased rapidly when the added dispersion agent was less than 0.4 wt.%.

Fig. 1 shows the relative densities of  $\text{Al}_2\text{O}_3$ – $\text{ZrO}_2$  ceramics with different solid contents in slurries as a function of sintering temperature. It was quite clear that the green and sintered densities of the  $\text{Al}_2\text{O}_3$ – $\text{ZrO}_2$  composites increased with the increasing solid contents in slurries. At the sintering temperature of 1400 °C, the relative density of the samples prepared from 60 and 78 wt.% slurries was 95.6, 98.9%, respectively. In addition, it was also observed that the green and sintered densities of samples prepared by centrifugal casting from 70 and 78 wt.% slurries were higher than those produced by cold isostatic pressing. As well know, green compacts with higher green densities can be sintered at lower temperatures to reach high final densities. In

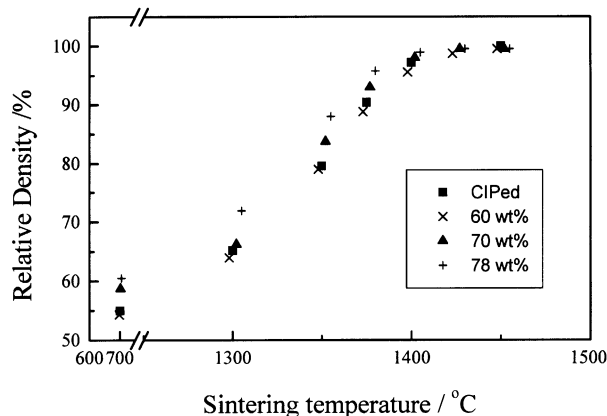


Fig. 1. Relationship between relative density of  $\text{Al}_2\text{O}_3$ – $\text{ZrO}_2$  ceramics with different solid contents of slurries and sintering temperature.

Table 1  
Viscosities of  $\text{Al}_2\text{O}_3$ – $\text{ZrO}_2$  slurries measured at about 35 °C

Dispersion agent addition (wt.%)	Viscosity (mPas)		
	60 wt.% slurry	70 wt.% slurry	78 wt.% slurry
0	377~378	486~488	5350~5400
0.1	~16 000	> 10 M	> 10 M
0.2	~14 600	> 10 M	> 10 M
0.3	3700~3900	~6 M	> 10 M
0.4	45~46	1400~1410	> 10 M
0.6		130~140	3940~3950
0.7			1000~1050
0.8			850~900

order to get the samples with high green densities, the solid content is expected to be as high as possible for centrifugal casting. However, when the solid content increased to 80 wt.%, all green compacts were broken during the centrifugal casting. It seems impossible to prepare unbroken green compacts from a 80 wt.% slurry in the present work. This is due to the fact that 3–4 g water was vaporized during the centrifugal casting process. Meanwhile,  $\text{ZrO}_2$  particles could not be homogeneously dispersed in  $\text{Al}_2\text{O}_3$  matrix and large agglomerates occurred in the 80 wt.% slurry.

Because of the difference in the specific densities of  $\alpha\text{-Al}_2\text{O}_3$  and  $\text{t-ZrO}_2$ , phase segregation may occur during the high-speed centrifugal casting process. Table 2 summarizes the ratios of the strongest integral intensity of  $\text{t-ZrO}_2$  (101) to  $\alpha\text{-Al}_2\text{O}_3$  (104) and the sintered densities at different regions of  $\text{Al}_2\text{O}_3\text{-ZrO}_2$  samples sintered at 1450 °C. When the solid content was 60 wt.%, the densities of the sample slightly increased from the top to bottom region. Moreover, the ratios of integral intensity of  $\text{t-ZrO}_2\text{:}\alpha\text{-Al}_2\text{O}_3$  increased rapidly from the top to bottom region (as shown in Fig. 2). This result clearly indicated that serious phase segregation

occurred in the 60 wt.% slurry. However, phase segregation could be effectively eliminated by starting a 78 wt.% slurry. The ratios of integral intensity of  $\text{t-ZrO}_2\text{:}\alpha\text{-Al}_2\text{O}_3$  and sintered densities of the  $\text{Al}_2\text{O}_3\text{-ZrO}_2$  sample with the 78 wt.% slurry were almost the same at different regions (as shown in Fig. 3). SEM observation shows that  $\text{ZrO}_2$  particles were homogeneously dispersed in the  $\text{Al}_2\text{O}_3$  matrix by the centrifugal casting process and there was no difference in microstructure between the centrifugal compacted and cold isostatic pressed samples (as shown in Fig. 4).

#### 4. Conclusions

The viscosities of the  $\text{Al}_2\text{O}_3\text{-15 wt.\%ZrO}_2$  slurries could be adjusted by adding a small amount of dispersion agent. When the solid content of the slurry was 78 wt.%, the  $\text{Al}_2\text{O}_3\text{-ZrO}_2$  composites with homogenous microstructure were obtained by the centrifugal casting process. Meanwhile, their green and sintered densities were higher than those prepared by cold isostatic pressing.

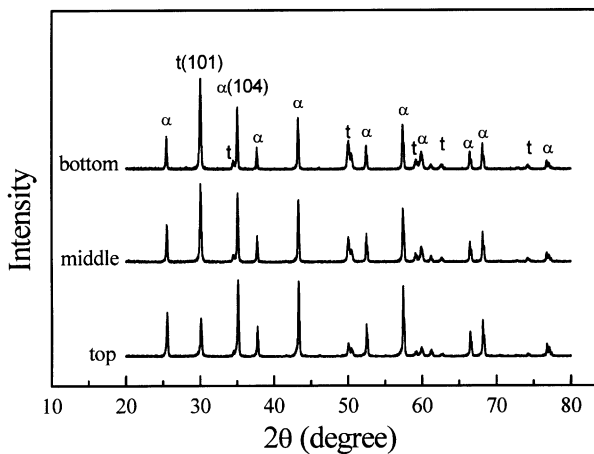


Fig. 2. X-ray diffraction patterns of different regions of sample made from 60 wt.% slurry.

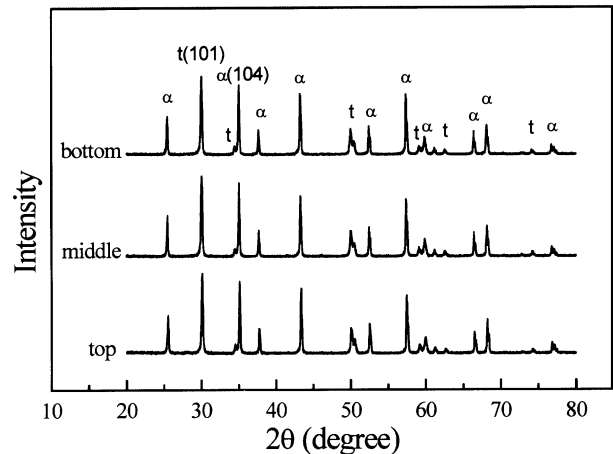


Fig. 3. X-ray diffraction patterns of different regions of sample made from 78 wt.% slurry.

Table 2

Ratios of integral intensity of  $\text{t-ZrO}_2$  (101) to  $\alpha\text{-Al}_2\text{O}_3$  (104) and sintered densities at different regions of  $\text{Al}_2\text{O}_3\text{-ZrO}_2$  samples

Sample position	60 wt. %		78 wt. %	
	$I_{\text{t-ZrO}_2} / I_{\alpha\text{-Al}_2\text{O}_3}$	Density <sup>a</sup> (g/cm <sup>3</sup> )	$I_{\text{t-ZrO}_2} / I_{\alpha\text{-Al}_2\text{O}_3}$	Density <sup>a</sup> (g/cm <sup>3</sup> )
Top region	0.57	4.152	1.28	4.183
Middle region	1.27	4.182	1.30	4.185
Bottom region	1.81	4.205	1.33	4.186

<sup>a</sup> Sintered at 1450 °C.

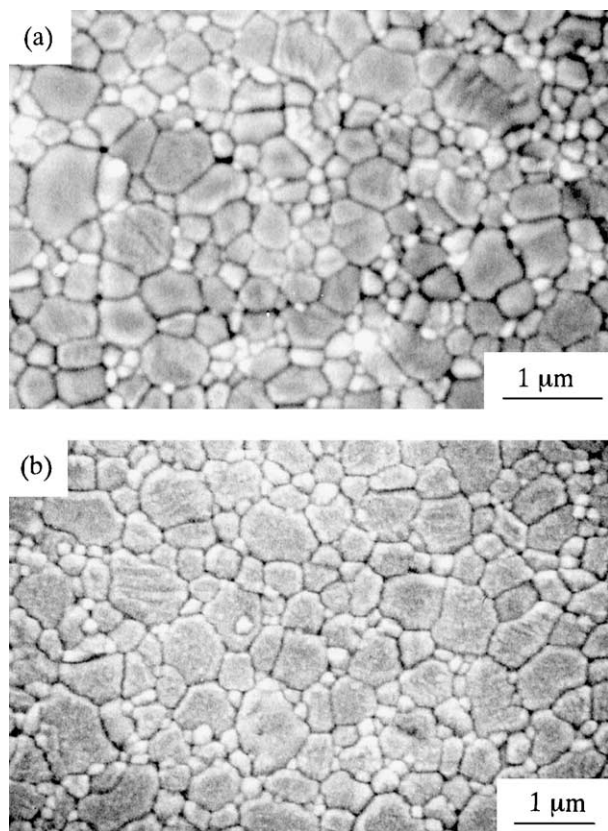


Fig. 4. SEM photographs of the thermally etched surface of  $\text{Al}_2\text{O}_3$ – $\text{ZrO}_2$  composite sintered at  $1425^\circ\text{C}$ : (a) prepared by centrifugal casting with the 78 wt.% slurry and (b) prepared by cold isostatic pressing.

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