Consolidation Induced Microstructural Inhomogeneities in $\text{Al}_2\text{O}_3\text{--ZrO}_2$ Composites Prepared by Centrifugal Slip Casting

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遠心鋳造方法によるアルミナ–ジルコニア複合セラミックス作製における微細組織不均一性

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Centrifugal slip casting was applied to the fabrication of an $\text{Al}_2\text{O}_3\text{--}5\text{ vol}\%\text{ZrO}_2$ (3Y-TZP) composite and the effect of solid contents in the slurry was investigated on various kinds of inhomogeneities and segregations. When a relatively thick slurry (40 or 45 vol%) was used, mutually hindered settling occurred quite effectively and thus essentially no segregations were observed in the distribution of grain sizes and the dispersion of the two phases. Even the zirconia aggregates contained in the starting powders were found distributed over the entire body of a sample. On the other hand, when a slurry of moderate thickness (30 vol%) was used, the Al_2O_3 and ZrO_2 particles were still uniformly distributed but the zirconia aggregates were found accumulated in the thin layer of the bottom section. The results were discussed with a model calculation and shown to be in general agreement with the theoretical prediction.

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1. Introduction

Nano-composites of ceramics have received considerable interests because of their potentials as strong and reliable structural components^{1–3)} and superplastically deformable ceramics.^{4,5)} In order to obtain a fine grained uniform structure, we need to start with fine powders and sinter the compact at a low temperature. Fine grade commercial powders of various kinds are now available in the form of sufficiently small elementary particle sizes. However, these powders generally contain aggregates of elementary particles and they would result in various kinds of inhomogeneities in the sintered body.⁶⁾ Although these aggregates may be broken apart by ultrasonication^{7,8)} or ball milling⁸⁾ or may be removed by sedimentation method,⁹⁾ it should be bore in mind that aggregates would rebuild if the suspensions are dried again into powders.⁶⁾

It has been proposed that colloidal processing is quite effective in preparing a uniformly dispersed slurry of fine particles and consolidating directly into a high density green compact for sintering.^{6,10)} Normally one of the following three methods are used for consolidating powders from slurry, namely slip casting, pressure filtration and centrifugal casting methods.^{6,10)} Although each method has its own merits, the centrifugal method has its distinctive features over the other two.¹¹⁾ The method is fast and convenient technique for making a compact. In addition, since it uses metal or glass moulds, drawbacks of contamination from plaster mould and low durability of mould which is often involved in slip casting method can be avoided. Furthermore, since an acceleration force is applied directly

to the individual particles in the slurry, a high and uniform packing density is expected in the compact.

The main possible disadvantage of centrifugal slip casting method is microstructural inhomogeneities induced by segregation, which is a result of the differential settling velocities of particles of different size and density. Hindered settling, which occurs in a high solid concentration suspension or in a flocculated suspension, has been the most frequently used strategy for minimizing the segregation in centrifugal casting.^{11–15)} The strategy is also used in slip casting method and the effect of slurry states on the segregation have been studied in considerable depth.^{14,16)} However, available research on centrifugal slip casting has been mostly concerned in the consolidation of monolithic ceramics.^{11–13,15,17)} In case of $\text{Al}_2\text{O}_3\text{--ZrO}_2$ composites, segregation between the two components would be the major concern with centrifugal casting, but only limited information is currently available.

Chang et al.¹⁴⁾ reported that a severe phase segregation took place in the 20 vol% dispersed slurry of $\text{Al}_2\text{O}_3\text{--}30\text{ vol}\%\text{ZrO}_2$ composite, but segregation could be avoided if coagulated or flocculated slurries were used. Although the dispersed slurry of a higher solid content can also eliminate the phase segregation, detailed information is lacking. The main purpose of the present work is to experimentally study the dependence of the phase and density segregation on the solid content of a dispersed slurry during centrifugal casting and to find a suitable condition for the fabrication of a uniform composite in an $\text{Al}_2\text{O}_3\text{--ZrO}_2$ system. The experimental results will be discussed in reference with the calculated results after a recently proposed simplified model of centrifugation.¹⁸⁾

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2. Experimental

2.1 Starting powders

The composite system investigated was Al_2O_3 -5 vol% ZrO_2 (3Y-TZP); the second phase composition was typical one for a nano-composite. Alumina powder (TM-DAR, Taimei Chemical Co.) was of 99.99% purity with the following impurities (in ppm): Si 7, Fe 6, Na 3, K 1, Ca 1, Mg 1 and the reported average grain size was $0.14\ \mu\text{m}$. The ZrO_2 powder (TZ-3Y, Tosoh Co.) was of 100% tetragonal phase with the following main constituents and impurities (mass%): Y_2O_3 5.250, Al_2O_3 <0.005, SiO_2 0.005, Fe_2O_3 <0.002, Na_2O 0.023 and the reported average grain size of $0.12\ \mu\text{m}$. The SEM micrographs and the particle size distributions are shown in Fig. 1 and Fig. 2, respectively, for both powders. The Al_2O_3 particles were of equiaxed morphology and had a very narrow particle size distribution. On the other hand, the 3Y-TZP particles appeared as agglomerates of finer crystallites and exhibited bimodal distribution of the particle size. Although the powders were ultrasonicated with 20 Hz and $\sim 200\ \text{W}$ (Model US-600T, 36 mm diameter horn, Nihonseiki Kaisha Ltd., Tokyo) for 4 min before the measurement of particle size distribution, considerable portions of the agglomerates appeared to have remained unbroken.

Fig. 1. SEM micrographs of the starting powders: (a) Al_2O_3 and (b) ZrO_2 .

Fig. 2. Particle size distributions of the starting powders.

2.2 Centrifugation

The powders were mixed and dispersed in distilled water by ball milling (Al_2O_3 or TZP balls) in a plastic pot for 24 h with the addition of a specified amount of polyelectrolyte dispersant (Polycarboxylic acid ammonium, $M_w = 10000$, Dai-ichi Kogyo Seiyaku Co., Ltd., Kyoto). The processing parameters, such as the amount of dispersant, the time for centrifugation, etc. were determined by a trial-error process¹⁵⁾ beforehand and are listed in Table 1.

Centrifugal consolidation was performed in glass tubes of dimensions of $\phi 10 \times 100\ \text{mm}$ by using a centrifugal machine (Model H-26F, Kokusan Co., Ltd.). The distance from the bottom of the tubes to the rotation center was about 140 mm. The prepared powder suspensions (6–8 ml, depending on the solids concentration) were centrifuged with a maximum rotation speed of 4000 rpm for 2–2.5 h until steady cakes were obtained. The resulting acceleration was $2507\ g$ at the bottom of the tubes and $\sim 1254\ g$ at the head of slurry. The clear supernatant was then poured out, by which no flow of powder particles were observed.

2.3 Sintering

The cakes were dried at 110°C for 12 h, then pyrolyzed at 700°C for 1 h with a heating rate of $100^\circ\text{C}/\text{h}$, followed by pre-sintering at 1000°C for 30 min with a heating rate of $400^\circ\text{C}/\text{h}$. The pre-sintered specimens were sliced into disks at a 4 mm interval. The sliced disks were numbered 1, 2, 3, etc., from the bottom to the top, as shown in Fig. 3. Totally 10 or 11 disks were obtained from a single column. Density of each piece was measured with Archimedes method using distilled water. To calculate relative density, 3.99 and $6.08\ \text{g}/\text{cm}^3$ were used as the theoretical density of Al_2O_3 and 3Y-TZP, respectively. The measured densities were regarded as the green density of each specimen, which was believed to be a tolerable approximation, although these values should be slightly higher than the real green densities. Final sintering was performed for 2 h at 1300°C for Al_2O_3 and ZrO_2 and at 1450°C for Al_2O_3 -5 vol% ZrO_2 (3Y-TZP) composites. The heating and cooling rates were $400^\circ\text{C}/\text{h}$.

2.4 Characterization of microstructures

Microstructure was observed on the polished and thermally etched surface of a specimen by SEM (Scanning Electron Microscopy). The distributions of Al_2O_3 and ZrO_2 were determined with EPMA (Electron Probe Micro-

Table 1. Processing Conditions as Used in the Experiment

Slurry	Dispersant content (mass% dry solids)	Solids volume (vol%)	Centrifugation time (h)
Al_2O_3	1	45	2.5
ZrO_2	2	30	2
$\text{Al}_2\text{O}_3/5\text{vol}\%\text{ZrO}_2$	1	30, 40, 45	2

Fig. 3. Schematic drawing of the consolidated cake.

analyzer, JXA-8900RL, JEOL) equipped with WDS (Wave-Dispersive Spectrometer). Randomly selected areas were analyzed for each specimen at a magnification of $1000\times$ in identical probe conditions. Average values were derived from three measurements. The average matrix grain sizes were measured by the line intercept length method. The ZrO_2 particle sizes were analyzed by an image analysis software (WinROOF) from 5–15 SEM micrographs and each measured area was converted into an equivalent circle to obtain its equivalent diameter.

3. Results

3.1 Density profile

Composites were prepared from suspensions of 30, 40 and 45 vol% solids contents. Hereafter, these samples are referred to as 30, 40 and 45 vol% composite, respectively, according to the solid content of the respective suspension. The green densities for the composite samples are shown in Table 2 along with those for the monolithic samples for comparison. The average green densities were 57.2, 58.5, and 59.5% for 30, 40, 45 vol% composite samples, respectively, indicating a slight dependence of green density on solid concentration of the starting slurries. This result is in agreement with a previous work for the alumina ceramics.¹⁵⁾ The green density of the monolithic alumina and zirconia samples were 61.3% and 50.8%, respectively. The addition of 5 vol% zirconia reduced the green density of the alumina compact.

The green density profiles of the monolithic and composite ceramics are shown in Figs. 4(a) and (b), respectively. In addition to the increased overall green density with increasing solid content, a slight decrease in green density from the bottom to top was observed in both monolithic and composite ceramics. Since the density gradient was not significantly different between the monolithic and composite ceramics, the observed density gradient must be due to the varying compressive stress along the height rather than the mass segregation which was likely to occur in composite slurry. The density gradient suggests a slight compressible nature of the particle network. Even though dispersed suspensions are generally considered to form incompressible network of particles, the use of high molecular weight dispersant would have resulted in the compressive nature, as suggested by Bergström et al.¹⁹⁾

The density profiles of the respective sintered bodies are also plotted in the Figs. 4(a) and (b). Despite the notable green density variations along the specimen's height as well as with different solid loadings, the bulk density of sintered bodies was uniform along the height and the same for the different solid loadings, except for the very bottom part of the sample prepared from 30 vol% slurry. As will be shown shortly, the latter effect was due to the segregation of heavy zirconia aggregates at the bottom. The fairly high and constant density of the sintered bodies suggested that the green density variations along the specimen's height as well as with different solid loadings were well within the tolerable degree with respect to densification.

3.2 Distributions of Al_2O_3 and ZrO_2

Phase segregation can occur in the centrifugal compaction process of composite slurry, because particles of different phases generally have different density and different size/shape distribution. Figure 5 shows the relative amount, but not calibrated, of Al_2O_3 and ZrO_2 as measured by EPMA in different sections of the sintered samples. Again, all samples exhibited nearly constant distributions of alumina and zirconia, except for the bottom section of the 30 vol% specimen. The higher zirconia and thus lower alumina composition at the bottom of the 30 vol% specimen was consistent with the aforementioned higher density of the

Table 2. Relative Density of the Samples

Sample	$\text{Al}_2\text{O}_3/5\text{vol}\%\text{ZrO}_2$			Al_2O_3	ZrO_2
Solids loading (vol%)	30	40	45	45	30
Green density (%)	57.2	58.5	59.5	61.3	50.8
$\Delta\rho_g^*$ (%)	1.9	2.1	1.6	1.8	1.6
Sintered density (%)	99.3	99.1	99.2	98.8	99.4

* Difference in green density between the top and bottom sections.

Fig. 4. Green and sintered relative density profiles for (a) monolithic specimens and (b) composite specimens prepared from suspensions of different solids concentrations.

Fig. 5. Distributions of Al_2O_3 and ZrO_2 phases in different parts of $\text{Al}_2\text{O}_3/\text{ZrO}_2$ composite specimens as determined by WDS analysis.

bottom section of the same sample (Fig. 4(a)). It is interesting to note that the severe segregation concentrated only in a small portion ($\sim 10\%$) of the total height of this sample.

3.3 Microstructures

Representative SEM micrographs of the sintered mono-

Fig. 6. SEM micrographs showing the microstructures of the top, middle and bottom sections of the monolithic ceramics: (a) Al_2O_3 and (b) ZrO_2 .

lithic alumina and zirconia are shown in Figs. 6(a) and (b), respectively, and those of the sintered composites prepared from the slurries of different thickness are shown in Figs. 7 (a)–(c). For each specimen, the micrographs were taken from the top, middle and bottom parts of the sectioned pieces (see Fig. 3). Despite the relatively low sintering temperature (1300°C), both monolithic ceramics exhibited fairly dense and uniform microstructures in all parts. To be more quantitative, the average grain size in each section was measured for these samples and the results are plotted in Figs. 8(a) and (b). Although the average grain sizes appeared slightly smaller in the top section of both alumina and zirconia specimens, it is not clear whether it has any physical significance or not.

As seen in Figs. 8(a) and (b), the average grain sizes of both alumina and zirconia were nearly the same in the top, middle and bottom sections of each composite. The average grain sizes also remained the same irrespective of the solid loadings of the slurry used. Despite the higher sintering temperature for the composites (1450°C) compared to the monolithic samples, the grain sizes of both alumina and zirconia remained considerably smaller than that of the respective monolithic samples. The mutual hindrance of the grain growth in the composite was clearly seen. In particular, the grain growth of minor phase zirconia was quite effectively suppressed in agreement with Okada et al's result.²⁰⁾

3.4 Aggregate distribution

The microstructures in Fig. 7 show that zirconia particles were dispersed mostly along the grain boundaries and triple junctions of the matrix grains. They were mostly isolated single grains of zirconia, but aggregates of zirconia comprising several grains were also sporadically observed. The distribution of these aggregates appeared more or less uniform

in the 40 and 45 vol% samples, but the aggregates appeared to be accumulated in the bottom section for the 30 vol% composite. Since the observed ZrO_2 aggregates were an important feature for the composites, size distributions of zirconia aggregates were quantitatively measured and illustrated in Fig. 9. Note that the size of only zirconia aggregates but not the isolated zirconia grains were counted.

Figure 9 shows that the zirconia aggregates were distributed in the range of $0.5\text{--}3\text{ }\mu\text{m}$: the range seemed to match the second mode of the bimodal distribution of the starting zirconia powder (see Fig. 2). It is seen that the aggregate distribution was considerably different from each other among the composites prepared from different slurries. Because of the relatively low sintering temperature of these composites, coalescence of zirconia particles into aggregates during sintering was unlikely. Furthermore, since the range of zirconia aggregates nearly matches the second mode of the bimodal distribution of the starting zirconia powders, the observed aggregate were considered to have been inherited from the starting powders. Thus, it was believed that results in Fig. 9 were a credible reflection of the aggregate distributions for the respective green compact, of which differences arose from the different thickness of the slurries used.

First, for the 45 vol% composite, the aggregates in the size range of $0.5\text{--}2\text{ }\mu\text{m}$ showed similar distributions in different sections, although a few aggregates of the largest class were found only in the bottom section. This result indicated that the 45 vol% composite suspension was thick enough to hinder the segregation of ZrO_2 aggregates. Next, for the 40 vol% composite, the aggregate distribution was more or less the same as the 45 vol% composite, except for the tendency

Fig. 7. SEM micrographs showing the microstructures of the top, middle and bottom sections of the composite specimens prepared from suspensions of different solid contents: (a) 45 vol%, (b) 40 vol% and (c) 30 vol%.

Fig. 8. Average grain size of (a) Al_2O_3 , and (b) ZrO_2 in different parts of the monolithic ceramics and composites prepared from slurries of 45, 40 and 30 vol% solids concentrations.

that the aggregates in all size ranges showed higher concentrations in the bottom part than those of the remaining parts. However, it was noted that the difference was rather small, in agreement with the phase measurement (see Fig. 5).

The 30 vol% composite showed a distinctly different dis-

Fig. 9. Size distributions of ZrO_2 aggregates in different parts of the composite specimens prepared from suspensions of different solid contents: (a) 45 vol%, (b) 40 vol% and (c) 30 vol%.

tribution of zirconia aggregates. No aggregates were found in the top and middle parts for the 30 vol% composite; almost all aggregates were found in the bottom part as shown in Fig. 9(c). This severe segregation of zirconia aggregate in the bottom part of the 30 vol% composite is consistent with the higher density and the higher ZrO₂ phase concentration observed in the corresponding section of the sintered composite (see Figs. 4(b) and 5). From these results, it may be considered that all the zirconia aggregates were accumulated only in the bottom section and thus the remaining part was essentially aggregate free.

4. Discussion

Centrifugation is effective in obtaining a high-density green compact, which in turn is quite beneficial for a good sintering ability. On the other hand, it is liable to various kinds of segregations. Segregation phenomenon could be more complex in a multi-phase composite system than in a monolithic system, because both mass and phase segregations can occur. However, as illustrated in Figs. 5 and 8, both the distribution of the phase composition and grain sizes of Al₂O₃ and ZrO₂ were quite uniform through the entire specimen for all three composites, except for the very bottom section of the 30 vol% composite. The bottom section of the 30 vol% composite exhibited a higher density, a higher zirconia concentration than the average and the accumulation of the zirconia aggregates. The first two phenomena seemed to be the results from the last one. On the other hand, ZrO₂ aggregates were found to be more or less uniformly distributed in the entire body of 45 vol% composites.

Segregations are essentially the result of different settling velocities of each particle in a powder suspension. The settling of dispersed particles in a suspension has been well investigated both experimentally^{13)-15),17)} and theoretically.^{18),21),22)} Recently, Biesheuvel and Verweij¹⁸⁾ developed a model to describe the cast evolution during centrifugation of a multi-component suspension of particles with different sizes and densities. According to the model, the velocity of *i*-type particle is given by:

$$v_i = \frac{1}{18\eta} \left[d_{p,i}^2 (\rho_{p,i} - \rho_s) - \sum_{j=1}^n d_{p,j}^2 (\rho_{p,j} - \rho_s) \Phi_j \right] (1 - \Phi_{tot})^{n_i-2} \omega^2 r \quad (1)$$

where $d_{p,i}$ is the diameter of *i*-type particles, Φ_{tot} is total volumetric suspension concentration, Φ_j is concentration of *j*-type particles in the suspension, $\rho_{p,i}$ and ρ_s are the densities of *j*-type particles and the suspension, respectively, n_i is the exponent of the hindrance factor of *i*-type particles.

Equation (1) was adopted to analyze the segregation in the present composite system. The composite system was simplified as composed of three types of particles, i.e., mono-sized Al₂O₃, mono-sized ZrO₂ and ZrO₂ aggregates; the average size was assigned for each component. The parameters used for the calculation are listed in Table 3, where *i* = 1, 2, and 3 correspond to particles of Al₂O₃, particles of ZrO₂, and aggregates of ZrO₂. The amount of aggregates was estimated to be 10% of the total ZrO₂ from Fig. 2 and the average size was 0.98 μm as measured from SEM micrographs of a sintered body, where the growth of aggregates was assumed not to have taken place during sintering. The density of the aggregates was obtained by assuming impermeable and spherical shape of aggregates with porosity $\varepsilon = 0.35$, which corresponds to a random packing of spheres. Then, $\rho_{aggre} = \varepsilon \rho_L + (1 - \varepsilon) \rho_{ZrO_2}$, where ρ_L is the density of water. The same values were assigned to the exponent of the hindrance factor n_i (*i* = 1, 2) as those used for Al₂O₃ and ZrO₂ in the literature.¹⁸⁾ n_3 was obtained by

Table 3. Parameters and Their Values Used in Calculation

Parameter	Value
<i>r</i>	0.095 m
ω	418.9 rad/s
<i>d</i> ₁	0.14 μm
<i>d</i> ₂	0.12 μm
<i>d</i> ₃	0.98 μm
η	0.0018 Pa·s
ρ_1	3940 kg/m ³
ρ_2	6050 kg/m ³
ρ_3	4282 kg/m ³
<i>n</i> ₁	4.65**
<i>n</i> ₂	4.65**
<i>n</i> ₃	11.49

* 1, 2, 3 represent Al₂O₃, mono-sized ZrO₂, ZrO₂ aggregates, respectively.

** reference 18.

Fig. 10. Settling velocities of Al₂O₃ (V_{Al}) and ZrO₂ (V_{Zr}) particles, and ZrO₂ aggregates (V_{aggre}) as a function of suspension solids loading as calculated using Eq. (1). Also shown in figure is the experimentally determined average boundary moving velocity (V_m).

equating the velocities of ZrO₂ particles and aggregates in Eq. (1) for 45 vol% suspension, as the case of the experimental result.

The calculated results for the three types of particles are plotted against the solid concentrations of slurry in Fig. 10. The settling velocities of mono-sized Al₂O₃ and ZrO₂ particles are in the range of several μm/s. The boundary velocities between supernatant and cake measured in the present experiment are also plotted in Fig. 10. It is noted that the boundary velocities agree well with the calculated velocities of Al₂O₃ particles, although these two types of velocities are not strictly the same parameters; the boundary velocity corresponds to the velocities of the finest particles of Al₂O₃, while the calculated velocity is meant to represent the average velocity of particles. The fair agreement between the calculated and measured velocities suggests that the calculations may be regarded as representing the behavior of the actual slurry.

It is noted in Fig. 10 that the velocity for ZrO₂ particles is slightly higher than Al₂O₃ particles at the highest solid content of the slurry and the difference increases only moderately as the solid content decreases. On the other hand, the velocity of ZrO₂ aggregates becomes very high as

the solid content decreases. Note that the velocities of ZrO_2 particles and aggregates were set equal at 45 vol% slurry. The behaviors well account for the experimental results, i.e. a large segregation was observed only for ZrO_2 aggregates at 30 vol% slurry and Al_2O_3 and ZrO_2 particles were essentially segregation free for the entire range of slurry concentration.

Chang et al.¹⁴⁾ reported a severe segregation between Al_2O_3 and ZrO_2 particles for 20 vol% slurry of Al_2O_3 -30 vol% ZrO_2 composite. Figure 10 shows only moderate difference is noted between Al_2O_3 and ZrO_2 particles at 20 vol% slurry and the calculated result may not appear to explain the severe segregation observed by Chang et al.¹⁴⁾ However, the particles sizes of their powders were different from ours. Recalculation of the settling velocities using their particles sizes, i.e. $0.2\text{ }\mu\text{m}$ for Al_2O_3 and $0.3\text{ }\mu\text{m}$ for ZrO_2 , resulted in the velocities $16.4\text{ }\mu\text{m/s}$ and $131\text{ }\mu\text{m/s}$ for Al_2O_3 and ZrO_2 particles, respectively. The difference is in fact much larger than that between Al_2O_3 particles and ZrO_2 aggregates at 30 vol% in Fig. 10 and qualitatively accounts for the experimentally observed severe segregation. In fact, a more elaborate calculation of the Al_2O_3 / ZrO_2 distribution profile in the cake¹⁸⁾ has accounted for the severe segregation observed by Chang et al.¹⁴⁾ It is seen in Eq. (1), that the settling velocity of particles depends primarily on $d_p^2(\rho_p - \rho_s)$. In the present powders, the diameter of higher density ZrO_2 particles was smaller than that of lower density Al_2O_3 particles. Thus the difference of the above term was kept small, while in Chang et al.'s system the situation was opposite and the difference was enhanced. As pointed out by Biesheuvel and Verweij,¹⁸⁾ Eq. (1) also tells that a change in ω or η influences the particle velocities to the same extent and, thus, does not influence the cast profile, although the change would influence the time necessary for final cast formation.

In the usual practice, both segregation between the component phases and the dispersion of large aggregates are unfavorable. In the present system, segregation between Al_2O_3 and ZrO_2 particles were essentially absent even with 30 vol% slurry. However, even ZrO_2 aggregates remained dispersed in the cake with 40 and 45 vol% slurries. On the other hand, the aggregates were segregated in the bottom section of the cake with 30 vol% slurry. Because, these aggregates appeared to be important structural flaws in the composites, they must be removed. The comparison between Figs. 2 and 9 suggests that these aggregates exist in the as received zirconia powders. Apparently the ultrasonication and ball-milling procedures of the present experiment could not eliminate ZrO_2 aggregates; similar bimodal distributions were reported in the literature.²³⁾ Even though these aggregates might have been broken apart by optimizing the conditions of ultrasonication treatment as reported by Suzuki et al.,⁷⁾ there are other sources of even larger inclusions, such as the metallic debris coming from the eroded ultrasonic horn, wear fragments coming from the milling ball and pot, etc. The present results suggest that a choice of suitable solid concentration of slurry, 30 vol% in the present case, would provide a means to separate the aggregates in the thin bottom layer of the cake and leaving the remaining portion as uniform mixture of component particles. The bottom section can be sliced off before sintering. Alternatively and more preferably, the aggregates and other inclusions in the slurry may be removed by preliminary centrifugation before the mixed homogeneous slurry is centrifugally cast into the cake for sintering.

5. Conclusions

The centrifugal slip casting method was applied for the

fabrication of Al_2O_3 -5 vol% ZrO_2 composite and the effect of the solid content of the slurries (30, 40 and 45 vol%) on the microstructural inhomogeneities was studied.

Slight variation in the green density was observed along the height of a specimen as well as between the samples prepared from slurries of different solid contents. However, because all parts of the three composites attained the same density of $\sim 99\%$ after sintering at 1450°C , the variation of the green density as observed in the present experiment seemed to be unimportant.

The grain sizes of alumina and zirconia were found to be distributed quite uniformly for all three composites. A significant difference was found in the distribution of ZrO_2 aggregates which seemed to be present in the starting powders. These aggregates remained dispersed in the entire volume of specimen for the 40 and 45 vol% composites. However, since they were accumulated in a narrow bottom section of the specimen for the 30 vol% composite, the elimination of the bottom section by slicing off would result in a segregation free green compact. If coarse aggregates or undesirable inclusions were absent in the starting powders, slurry of 30-45 vol% solid content would be quite suitable for preparing quite uniform green compacts.

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