Re-fluidising the aged gel for high-density alumina green body

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Abstract: A green body with a density as high as possible is critical to diminish the crisis of deformation or cracking when large-size parts are sintered. Here, a new method, i.e., re-fluidising the aged ceramic gel is developed to prepare the high-density green body. Alumina slurry with 56 vol% solid loading and copolymers of isobutylene and maleic anhydride were aged without vapourisation and re-fluidised by non-intrusive shearing after removing the exuded water. The re-fluidised slurry was re-casted. The resultant wet gel was dried and deboned at a low temperature. The relative density of the obtained green body was 64.6\%, 1.5\% higher than that without aging and re-fluidising. The linear sintering shrinkage of the body decreased by 0.7\%. The enhanced green density is explained from the viewpoint of the solid loading and the structure of the slurry.

Keywords: high density; green body; re-fluidising; alumina; colloid formation

1 Introduction

High-density green body is not only advantageous for the preparation of ceramics with a fine grain size and high strength because they can be densified at a low sintering temperature, but also critical to diminishing the crisis of deformation or cracking of ceramic parts of large size because they possess a low sintering shrinkage ratio. Colloid formation as a near-net-forming process is a popular method of preparing a high-density and homogeneous green body [1]. To obtain the high-density green body, researchers usually try to increase the solid loading of slurry based on Derjaguin, Landau, Verwey, and Overbeek (DLVO) theory. However, the viscosity of the slurry will increase with the increase in the solid loading, which leads to problems such as poor degassing and difficult casting operations. Therefore, increasing the density by increasing the solid loading of the slurry meets a bottleneck in the fabrication process.

Recently, a simple forming method called spontaneous coagulation casting (SCC) has been applied to fabricate advanced ceramics by using copolymers of isobutylene and maleic anhydride (commercially named Isobam) [2]. The mechanism of SCC is hydrophobic interaction and hydrogen bonding. Based on this, a series of agents were synthesised for the forming of both dense and porous ceramics by grafting a surfactant with a short or long chain on a dispersant [3]. Many dense

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ceramics such as Al₂O₃, YAG, MgAl₂O₄, Y₂O₃, and AlON, as well as porous ceramics such as Al₂O₃ and mullite, have been formed by the SCC system [4–10]. Di et al. [11] obtained an alumina green body with 57.1% relative density from the slurry with 50 vol% solid loading. By using the same alumina powder (median particle size of 0.45 μm) and an Isobam agent with low molecular weight, Sun et al. [12] obtained a green body with 60.4% relative density from the slurry with 56 vol% solid loading. However, it is difficult to further increase the relative density by merely increasing the solid loading.

There are two significant features of the ceramic gel by the SCC system which were mentioned by few researchers. On the one hand, when the slurry is cast and sealed, the wet gel automatically exudes water from the inside, which is called syneresis. The solid loading of the wet gel thus is increased after removing the exuded water. On the other hand, the wet gel based on the SCC is a physical gel [13–15]. The physical gel is a form of soft matter, and any mechanical force that exceeds the loading limit of the gel will break it into a free-flowing mass [16], which means that the wet gel can be re-fluidised and coagulated again after casting. Therefore, it is expected that the high-density green body can be manufactured by using this new idea.

Normally, vibration and stirring are considered valid methods of re-fluidising the gel. Balzer et al. [17] found that the alumina wet gel prepared by improving the ionic strength tended to flow when vibrated or even shaken by hand. Stirring is also a valid method of re-fluidising wet gel because of the internal shearing effect, but bubbles will be introduced. For the high-solid-loading slurry, degassing is difficult, and the trapped bubbles produce defects in the final ceramics.

In this work, taking advantage of the features of SCC alumina gel, the high-density green body was prepared by aging the gel without vaporisation and by re-fluidising the gel with non-intrusive shearing.

2 Materials and methods

2.1 Raw materials and slurry preparation

Commercial alumina powder (AES-11, Sumitomo Chemical, Japan) of a median particle size of 0.45 μm was selected as the raw material. The Isobam 104 and Isobam 600AF (molecular weight: 55,000–65,000 and 5500–6500, respectively; Kuraray, Japan) were chosen as both dispersant and gelling agents. According to Sun et al. [12], the Isobam 104 of 0.1 wt% and Isobam 600AF of 0.2 wt% (relative to the weight of alumina powder) were dissolved in ultra-pure water. The Isobam solution was poured into an attrition mill chamber, and then the alumina powder was gradually added. Alumina slurry with 56 vol% solid loading was homogenised by milling at a speed of 200 r/min for 1 h, degassed, and finally cast into organic glass moulds (inner diameter: 40 mm, height: 30 mm) to prepare the sample denoted as 56 vol%-%0.

2.2 Aging and re-fluidising

The as-prepared slurry with 56 vol% solid loading was cast into six moulds. After being sealed, the samples were aged in a constant temperature (25 ℃) chamber for 1–6 d. The moulds were then opened to remove the exuded water which was located on the top surface of the wet gel. The weight of the removed water was determined by comparing the weight of the mould with that of the alumina slurry before and after removing the exuded water. Then, the wet gel was moved to a vacuum degassing mixer (ARV310, THINKY, Japan) and became flowable by non-intrusive shearing owing to the rotation and revolution of the mixer. The re-fluidised slurry was cast again, and the samples were denoted as 56 vol%-%1, -2, -3, -4, -5, and -6. The samples 56.5 vol%-%0 and 56.5 vol%-%1 from the alumina slurry with 56.5 vol% solid loading were prepared in the same way as mentioned above.

2.3 Drying, debonding, and sintering

Drying was carried out in a constant temperature (25 ℃) and humidity (85%) chamber for 48 h and then placed in a drying oven (50 ℃) for 24 h. The debonding condition was 800 ℃ for 6 h in a muffle furnace under an air atmosphere. After that, the green body was cut into five equal-size wafers along the vertical direction. The top wafer of the green body (56 vol%-%0, -1) was radially divided into four parts, which were then sintered at 1400, 1450, 1500, and 1550 ℃ for 2 h.

2.4 Characterization methods

The rheological behaviour of the as-prepared and re-fluidised slurry was characterized by using a rotational rheometer (HAAKE Viscotester IQ Air, Thermo Electron LED GmbH, Germany) with a parallel plate...
(20 mm in diameter) at 25 °C. To explore the microstructure difference between the as-prepared slurry and re-fluidised slurry, freeze-drying was used. The as-prepared slurry was degassed and poured into liquid nitrogen. The slurry re-fluidised by non-intrusive shearing was directly poured into the liquid nitrogen. The frozen slurry was dried under a vacuum. The fracture surface of the dried body was observed by using a scanning electron microscope (SEM; Magellan 400, FEI, USA). The maximum and minimum diameters of the samples’ top parts were measured, and their average value was taken as the diameter of the sample. The densities of the wafers were measured based on Archimedes’ principle. The average density of five wafers from one sample was defined as the density of the sample. The relative density was calculated based on the measured density divided by the theoretical density (3.987 g/cm³). The porosity, cumulative pore volume, and pore-size distribution of the green body were determined by using a mercury intrusion porosimeter (Poremaster 60, Anton Paar, Austria). The sintering shrinkage $\gamma$ was determined based on Eq. (1):

$$\gamma = 1 - \frac{L_s}{L}$$

where $L$ and $L_s$ are the length of samples before and after sintering, respectively.

### 3 Results and discussion

#### 3.1 Characterization of slurry

##### 3.1.1 Increase in solid loading after aging

Based on the weight of the gel and exuded water, the solid loading of the gel after aging was calculated according to Eqs. (2)–(4):

$$V_{\text{syn}} = V_{\text{Al}_{2}O_3} / (V_{\text{Al}_{2}O_3} + V_{\text{H}_2\text{O}})$$

$$V_{\text{Al}_{2}O_3} = M\alpha / \rho_{\text{Al}_{2}O_3}$$

$$V_{\text{H}_2\text{O}} = [M(1 - \alpha) - M_{\text{ex-\text{H}_2\text{O}}}] / \rho_{\text{H}_2\text{O}}$$

where $V_{\text{syn}}$ is the solid loading of the gel after aging, $V_{\text{Al}_{2}O_3}$ and $V_{\text{H}_2\text{O}}$ are the volumes of $\text{Al}_{2}O_3$ and $\text{H}_2\text{O}$ in the slurry, respectively, $M$ is the slurry weight, $\alpha$ is the mass fraction of alumina in the slurry, and $M_{\text{ex-\text{H}_2\text{O}}}$ is the weight of the exuded water after aging. The densities of $\text{Al}_{2}O_3$ and $\text{H}_2\text{O}$ are given as $\rho_{\text{Al}_{2}O_3} = 3.987 \text{ g/cm}^3$ and $\rho_{\text{H}_2\text{O}} = 1.000 \text{ g/cm}^3$, respectively.

The relationship between the solid loading and the aging duration is shown in Fig. 1. As the water was exuded, the solid loading gradually increased. After aging for 6 d, the solid loading of the gel increased from 56.00 to 56.93 vol%.

##### 3.1.2 Rheological behaviour

Since the Isobam 104 was adsorbed on the surfaces of alumina particles, the particles were well dispersed by electrostatic repulsion and steric hindrance. The alumina slurry with 50 vol% solid loading was prepared [2], and the solid loading was further increased to 56 vol% by combining the Isobam 104 with the Isobam 600AF [12]. Because the Isobam 600AF with a shorter chain is better stretched and has more $-\text{COO}^-$ groups to interact with the positive charges on the surfaces of alumina particles, the gel from the slurry with 56 vol% solid loading aged for different days was re-fluidised. Also, the rheological behaviour of the as-prepared
slurry (56 vol%-0) and re-fluidised slurry was examined (Fig. 2). All the slurry showed a shear-thinning behaviour and became shear-thickening with the shear rate increasing. The viscosity at 100 s \(^{-1}\) increased from 0.55 Pa·s (56 vol%-0) to 1.42 Pa·s (56 vol%-6) with aging duration. As shown in Fig. 1, the solid loading of the gel increased with aging duration, which was the main reason for the increase in viscosity and shear-thickening.

Storage modulus is an index used to determine gelation ability of the slurry. The storage modulus of the slurry was measured at 25 °C (Fig. 3). For all the slurry, the storage modulus initially increased slowly and then increased rapidly. The storage modulus of 56 vol%-0 was lower than that of 56 vol%-1, but higher than those of the other re-fluidised slurry with the aging time increasing to 3–6 d. It indicated that, the gelation ability was strengthened for the 56 vol%-1, and then slightly weakened for other re-fluidised slurry. In other words, the gelation ability was not always positively related to the solid loading. This relationship between the solid loading and the gelation ability was different from the results of Yang et al. [2]. They claimed that the molecule chains of Isobam adsorbed on the particles easily interacted with each other because of the short distance between alumina particles, which was the reason why the gelation ability increased with the solid loading.

For the re-fluidised slurry, in our opinion, there are two factors affecting the gelation ability. One is the solid loading, and the increase in solid loading is helpful for the gelation. The other is the microstates of slurry (including the states of the Isobam polymer and the alumina particles). Peng et al. [18] proposed that the chemical activity of Isobam chains in the wet green body became weak with the aging time. This may be one of the reasons of the decrease in the gelation ability after aging in the present case. According to Marsico et al. [15], the interactions including chain–particles and chain–chain existed in the gel coagulated from the as-prepared slurry. When the exuded water was removed and the non-intrusive shearing was exerted on the gel, some weak interactions were destroyed. As a result, the gel was broken into agglomerates and particles and became the re-fluidised slurry. Therefore, the re-fluidised slurry was different from the as-prepared slurry in the solid loading and the microstate of slurry. For the 56 vol%-1, the microstate of the polymer between 56 vol%-1 and 56 vol%-0 was similar because of the short aging time (1 d), and the gelation ability increased with the enhancement of solid loading. When the aging time was 3 d or even longer, polymer bridging and chain entanglement were strengthened, some alumina particles were closed to each other, and some stretching chains of the polymers were gradually attached to the surfaces of particles. After re-fluidisation, the polymer chains adsorbed on the surfaces of particles could not stretch as before, which was not conducive to the gelation of re-fluidised slurry. Thus, the gelation ability of the re-fluidised slurry decreased when the aging time increased to 3–6 d.

### 3.1.3 Micromorphology of slurry

The micromorphologies of the as-prepared and re-fluidised slurry after freeze-drying are shown in Fig. 4. The alumina particles were homogeneously packed without obvious agglomerates in the as-prepared slurry (Fig. 4(a)) because the Isobam 600AF and Isobam 104 molecules were absorbed on the surfaces of alumina particles, which were then homogeneously distributed by the electrostatic repulsion and steric mechanism. However, the alumina particles and agglomerates of different sizes were observed in the
re-fluidised slurry (Fig. 4(b)). The agglomerates originated from the breaking of the ceramic gel.

### 3.2 Characterization of green body

#### 3.2.1 Drying shrinkage of green body

Table 1 shows the drying shrinkage of the wet body after aging for different days. We observed that the drying shrinkage of the wet body decreased from sample 56 vol%-1 to sample 56 vol%-6, which is similar to the result of Tari and Ferreira [19], who found that the total shrinkage of slip cast body always decreased with the solid loading of slurry increasing. It became a common sense that preparing a high-solid-loading slurry was indispensable to minimising the shrinkage of the green body [20]. However, the shrinkage of the samples 56 vol%- (1–5) was larger than that of sample 56 vol%-0, even though their solid loading was higher than that of sample 56 vol%-0. A possible mechanism is shown in Fig. 5.

For the as-prepared slurry, the alumina particles were homogeneously dispersed in the water (Fig. 5(a)). With the time going, the slurry spontaneously coagulated to form a ceramic gel. During aging, the alumina particles moved toward each other as the water was exuded, and the density of the gel gradually increased (Fig. 5(b)). After drying, the denser green body was obtained (Fig. 5(c)). By non-intrusive shearing, the gel was broken into particles, agglomerates, and water, and the agglomerates were relatively dense in the re-fluidised slurry (Fig. 5(d)). After casting, the particles and agglomerates were formed into the wet gel again, and the different sizes between the agglomerates and particles might have resulted in their loose packing (Fig. 5(e)). As a result, the shrinkage of the green body for the samples 56 vol%- (1–5) was larger than that of the green body of the sample 56 vol%-0, and the denser green body was obtained (Fig. 5(f)).

#### 3.2.2 Density of green body

Figure 6 shows the bulk and relative densities of the green body after debonding at 800 °C for 6 h. The relative density of the sample 56 vol%-0 (63.1%) was higher than those reported by Di et al. [11] (60.1%) and Sun et al. [12] (60.4%) from the alumina slurry with the same powder and solid loading (56 vol%) when a planetary mill was used to prepare the slurry. The enhanced density could be attributed to the effect of the attrition mill which was used in this research by ourselves. Alex et al. [21,22] compared particle-size distributions by an attrition mill and a planetary mill. The attrition mill was propitious to obtain smaller \( d_{50} \) (particle size with 50% cumulative particle distribution) values, and the powders were well deagglomerated. Thus, the bulk density was increased.

The bulk densities of the green body of the sample 56 vol% after aging for 1 and 5 d rapidly increased from 2.517 to 2.558 and 2.576 g/cm³, respectively. Correspondingly, the relative densities increased from 63.1% to 64.1% and 64.6%, respectively. The same
phenomenon was observed between the samples 56.5 vol%-0 and 56.5 vol%-1. The bulk density increased from 2.539 to 2.580 g/cm³, and the relative density increased from 63.7% to 64.7%. The reason for the density improvement of green body was ascribed to the increase in the solid loading. Besides, the gel from the re-fluidised slurry shrank bigger than those from the as-prepared slurry because of the different sizes of agglomerates and particles in the re-fluidised slurry (Fig. 5(d)).

The reason for the density improvement for the samples 56 vol%-1 to 56 vol%-5 was ascribed to the increased solid loading. Slightly low relative density was observed for the sample 56 vol%-6. There may be an optimal aging time for the re-fluidised wet gel. On the one hand, the solid loading was increased after aging, and the existence of agglomerates of different sizes was beneficial to the high density; on the other hand, the increased strength of wet gel after aging may weaken the effect of re-fluidising.

3.2.3 Pore structure of green body

The pore-size distributions and cumulative pore volumes of the green-body samples 56 vol%-0 and 56 vol%-1 (both sampled at the middle wafer among the five wafers) are shown in Fig. 7, and the corresponding data was listed in Table 2. The porosity decreased from 37.6% of the sample 56 vol%-0 to 35.5% of the sample 56 vol%-1. It was ascribed to the fact that the mean pore size decreased from 79 to 67 nm, and the cumulative pore volume decreased from 0.148 to 0.139 mL/g after the re-fluidising process.

The mercury intrusion porosimetry can measure large capillary pores (0.005–10 μm). We found that there were pores less than 0.005 μm in 56 vol%-1, which may be due to the bottle-neck effect. It had a little effect on the calculation of mean pore size and did not affect the porosity. The mercury intrusion data shows that a denser green body was manufactured by the re-fluidising method. This result was consistent with those of the drying shrinkage (Table 1) and bulk density (Fig. 6) of the green body.

3.2.4 Characterization of sintered body

Figure 8 shows the bulk densities and linear shrinkage ratios of the ceramics prepared from the green-body samples 56 vol%-0 and 56 vol%-1 sintered at different temperatures. The densities of the sample 56 vol%-1 were higher than those of the sample 56 vol%-0 sintered at each temperature from 1400, 1450, 1500, to 1550 °C (Fig. 8(a)). With the sintering temperature increasing, the density difference between the samples 56 vol%-0 and 56 vol%-1 gradually decreased from 0.03 to 0.01 g/cm³, even though there was a large density difference between the two green-body samples (0.04 g/cm³). This phenomenon was in agreement with the results of Krell et al. [23], where the difference between the relatively high- and low-density green body was gradually eliminated with the sintering temperature increasing. The linear shrinkage of the sample 56 vol%-1 was lower than that of the sample 56 vol%-0 sintered at each temperature from 1400, 1450, 1500, to 1550 °C. The shrinkage ratio decreased from 14.1% of sample 56 vol%-0 to 13.4% of sample 56 vol%-1 after sintering at 1550 °C for 2 h (Fig. 8(b)). This was because the green body of the sample 56 vol%-1 possessed a higher density (Fig. 6).

It is known that the sintering shrinkage depends on the density or porosity of the green body. Zheng and Reed [24] classified the porosity in the alumina green body into two classes: The first class contains pores smaller than the critical ratio (pore size to mean particle

![Fig. 7 Pore-size distributions and cumulative pore volumes of the green-body samples 56 vol%-0, -1 after debonding.](www.springer.com/journal/40145)
Table 2  Porosity, cumulative pore volume, and mean pore size of 56 vol%-0 and 56 vol%-1 measured by the mercury intrusion porosimetry

| Sample          | Porosity (%) | Cumulative pore volume (mL/g) | Mean pore size (nm) |
|-----------------|--------------|------------------------------|---------------------|
| 56 vol%-0       | 37.6         | 0.148                        | 79                  |
| 56 vol%-1       | 35.5         | 0.139                        | 67                  |

Fig. 8  Influence of sintering temperature on (a) bulk density and (b) linear shrinkage ratio.

The surface microstructures of 56 vol%-0 and 56 vol%-1 after sintering at different temperatures are shown in Fig. 9. The pores in the ceramics of 56 vol%-1 were less than those in the ceramics of 56 vol%-0 when sintered at 1500 °C for 2 h (Figs. 9(a) and 9(b)). This phenomenon still existed when the sintering temperature increased to 1550 °C (Figs. 9(c) and 9(d)). Obviously, the denser ceramic was successfully prepared by the re-fluidising method because of the improvement in the green body density.

4 Conclusions

The alumina ceramic gel formed with the copolymers of isobutylene and maleic anhydride is a physical gel that can be re-fluidised and coagulated again. Another characteristic of this gel is that it automatically exudes water from the inside when it is aged without vaporisation at room temperature. Based on these, the re-fluidising method was proposed in this study, and the following conclusions were drawn.

The alumina ceramic gel from the slurry with 56 vol% solid loading was aged for different days, and the solid loading of the gel increased after removing the exuded water. The aged gel became flowable by non-intrusive shearing. After casting, the re-fluidised slurry turned into gel again. The green body from the re-fluidised slurry possessed a lower cumulative pore volume (0.139 mL/g) and a smaller mean pore size (67 nm) compared to the as-prepared slurry. Correspondingly, the relative density of the resultant green body increased to 64.6%, 1.5% higher than that of the as-prepared slurry. The sintering shrinkage ratio decreased to 13.4%, 0.7% lower than that of the as-prepared slurry. These are propitious for obtaining the high-density ceramic of a large size. It is demonstrated that re-fluidising the aged slurry is an effective method of preparing a high-density green body with a low sintering shrinkage ratio.
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