Reducing the density deviation in alumina by pressure-vacuum hybrid slip casting by employing powders with different particle sizes

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1. Introduction

As the size of display electronics increases, the scale up of ceramic size is inevitably required. However, there are many difficult problems in the forming techniques for large-scale fabrication of ceramic parts and components. One of the most important problems is the spatial density heterogeneity of the forming body, which can lead to non-uniform shrinkage in subsequent drying and sintering processes. This causes deformation and cracking and leads to non-uniformity of material quality, reducing the manufacture yield of the genuine materials with the required in the display industry [1,2].

Slip casting is one of the forming method for large ceramics by removing the dispersion medium from the concentrated slurry. A green body is formed on a porous mold by applying external mechanical pressure, which separated the dispersion medium from the solid through the porous mold [3–15]. The particle size, particle size distribution and morphology of the starting powder greatly influence the packing density of the slip cast body and the microstructure of the sintered body [16]. To obtain ceramics of uniform microstructure, the starting materials are small in size and spherical powders with a tightly controlled powder size distribution are ideal. However, if the size is small or has a narrow size distribution, the price of the powder is rapidly increased. Therefore, the mixed batch in which the small sized powders are added to the commercial powders of microscale and wide particle size distribution may provide economic manufacturing way and can effectively improve the density homogeneity and their performance.

In ideal packing of mono-sized spherical powders, octahedral and tetrahedral voids are created, and the geometric ratios of the void size to the host powder (H powder) size are 0.414 and 0.225 respectively [17,18]. When the voids of a large spherical host powder are all sequentially filled with small spherical powders, the highest packing density obtainable increases from 74.04% to 79.29% and 80.98% depending on the octahedral + tetrahedral void filling, respectively [17,18].

At this time, the volume ratios of the two spherical powders; the host powder and the octahedral void powder, are 93.47 vol% and 6.53 vol%. In the case of the volume ratios of the three spherical powders; the host powder, the octahedral void powder, and the tetrahedral void powder, are 91.45 vol%, 6.38 vol%, and 2.17 vol%, respectively [18]. However, this sequential void filling does not change or adjust the coordination structure in the particle packing.

Most researchers showed high packing density with bimodal distribution of coarse and fine powders, where the addition of large particles to a fine particle matrix can improve initial packing density, but decrease the densification in sintering [19–26]. In such a particle size system, stress differences occur
during firing at high temperatures and large particle and pore structure differences occur due to the difference in the driving force for densification between the small powder region and the large powder region. In particular, densification inhibition situations occur with exaggerated grain growth, separation of pore-grain boundaries and pore coarsening [16,23].

However, we thought that the mixing of coarse, medium and fine powders in a precise way, taking into account the geometric aspects, could improve not only the packing density but also the sinter density. If the coarse particles form a rigid skeleton structure and the small particles occupy interstitial voids (homogeneous mixing inside the packing body), then it will not adversely affect the overall homogeneous densification during firing. This is more effective with short sintering time and at low sintering temperatures, which do not cause excessive growth [16,25]. That is, a mixed batch in which small amounts of fine and medium sized powders are added to the commercial powders of coarse and wide particle size distribution. Preparation with these modified starting powders can effectively improve the performance of the resulting product and provide an economical manufacturing method in the real industrial field.

In this study, commercial Al₂O₃ powders were first ball milled to produce sizes corresponding to the host (H) powder, and void sizes of 8 coordination (O) powder, 4 coordination (T) powder. Compared to a normal wide size distributed powder batch H, the powder batches including medium and fine powders (H + O and H + O + T) were applied in a geometrically precise volume ratio that could have a higher packing density. Pressure-vacuum hybrid slip-casting (PVHSC) was performed from three powder batch aqueous slurries at appropriate pressure conditions. During the drying and sintering of the green body, the humidity and temperature carefully controlled to avoid the appearance of defects. Firing was carried out to maximize densification with minimum excessive grain growth. The drying characteristics, forming density distribution, and sintering density distribution of the cast bodies made from three powder batches were investigated and discussed.

## 2. Experimental procedure

Two commercial Al₂O₃ powders with irregular polyhedral shapes were used as raw materials: SA-32 (Average size of 1.54 µm, 99.6% Purity, Nippon Light Metal Co., Ltd., Japan) and AES-11 C (Average size of 0.57 µm, 99.9% Purity, Sumitomo Chemical Industry, Japan). To prepare the three powders of different sizes for the three powder mixtures, the commercial alumina powders were subjected to ball milling under different conditions (Table 1). The target and obtained average sizes of the host, secondary and tertiary powders (denoted H, O and T powders) were 1.36, 0.56, and 0.31 µm, respectively. The particle size distributions of the H, O, and T powders after ball milling are shown in Figure 1. H powders have micro-size and wide size distribution like typical commercial powders. However, O and T powders are additive particles with designated amount and size in geometric aspects to improve packing density. We thought that the narrower size distribution of O and T powders, the higher the density. Their average sizes are small and the particle size distributions are narrow. The mixing amount for the powder batches are shown in Table 2.

Slips were prepared using each of the three powder batches and cast into a plate body by PVHSC (pressure vacuum hybrid slip casting) [27–30]. Al₂O₃ powder batches at a concentration of 40 vol% were dispersed in ion-exchanged water containing a dispersant [ammonium polycarbonate, Ceraperse 5468CF, (NH₄)₂CO₃)n, San Nopco Korea Ltd., Korea], a plasticizer [polyethylene glycol,(C₃H₆O)n, Mw 360 ~ 440, Guoli Chem. Co., Ltd., Korea], and a binder [polyvinyl alcohol,(CH₂CHOH)n, Kuraray Co., Ltd., Japan] to form slurries. The dispersant content of the slip was 0.3±0.4 wt% in the slip (H batch: 0.4 wt%, H + O batch: 0.3 wt%, H + O + T batch: 0.3 wt%), and the content of the other additives was 0.5 wt% each. The slurry for PVHSC was homogenized with the Al₂O₃ medium and vacuum-treated for de-airing.

A porous polyvinyl chloride (PVC) casting mold was used to produce an alumina plate (110 × 110 × 20 mm³) by PVHSC, as shown in Figure 2. The PVHSC processing was performed in a plate die using a polypropylene fiber-woven membrane filter (Hwashin Special Textile Filter Co., Ltd., Korea) supported on a porous PVC plate with pores 2 mm in diameter. The slip inlet load was increased to 0.5 MPa in five equal steps by a compressor, and air was pumped out continuously by applying a pressure of −0.05 MPa on the downstream side of the filter to enhance the removal of the filtered water, as shown in Figure 3.

The spatial partial density within pressure-filtered bodies inevitably varies along the pressure direction because of internal friction between the particles as well as the particles and the mold walls [15,22,23,29]. Spatial density distribution (deviation) in the green body causes cracking and deformation of the ceramic body during subsequent drying and firing [6–15,20,31]. During the drying of the green body, the humidity and temperature carefully controlled to avoid the appearance of stress-induced defects [27–30]. The as-

### Table 1. Starting Al₂O₃ powders prepared by ball milling.

| Nomination | Raw powder | Milling condition | Target size (µm) | Obtained size (µm) |
|------------|------------|-------------------|------------------|--------------------|
| H powder   | SA-32      | Wet ball milling for 6 h | 1.36             | 1.36               |
| O powder   | SA-32      | Planetary milling for 2 h  | 0.56             | 0.56               |
| T powder   | AES-11 C   | Planetary milling for 9 h  | 0.31             | 0.31               |
fabricated green cakes were allowed to dry at 25°C and 80% relative humidity to ensure a slow drying rate and then dried at 50°C for 24 h in a dryer (Figure 4). The as-dried specimens were prepared for measurement by cutting the alumina block into small cuboid pieces from the upper, middle, and lower sections and grinding their surfaces carefully with a fine SiC paper, as shown in Figure 5(a). The green densities were determined geometrically.

Small cuboid pieces and whole bodies made from each powder batch were sintered in an electrical furnace with MoSi₂ heating elements in air. Firing was carried out at 1650°C to maximize densification with minimum excessive grain growth. Of course, even in the case of a green body having a homogeneous density distribution, in order to obtain a homogeneous sintered body, it can be obtained only by appropriately adjusting the firing temperature and time [16,17,25]. During sintering, the specimens were heated at 10°C/min to the maximum temperature of 1650°C.

![Figure 1](image1.png)

**Figure 1.** Particle size distributions of the starting alumina powder powders [d(0.1): 10% of the particles are smaller than this diameter, d(0.5): median diameter, and d(0.9): 90% of the particles are smaller than this diameter].

![Table 2](image2.png)

**Table 2.** Al₂O₃ powder batches consisting of mixtures of powders with different particles sizes.

| Nomination       | Mixing ratio (vol%) |
|------------------|---------------------|
|                  | H powder | O powder | T powder |
| H batch          | 100.00    | -        | -        |
| H + O batch      | 93.47     | 6.53     | -        |
| H + O + T batch  | 91.45     | 6.38     | 2.17     |

![Figure 2](image3.png)

**Figure 2.** Schematic diagrams of (a) the PVHSC apparatus and (b) the casting mold.
with an isothermal dwell time of 4 h along with intermediate soaking periods at 350, 600, and 900°C to remove organic residues [18].

The sintered density of the piece specimens was determined by the Archimedes method based on the ratio of the bulk density to the theoretical density of α-alumina (3.987 g cm⁻³). The average and standard deviation of the density of sets of pieces and five whole bodies from three different samples were obtained. The microstructure was observed using field emission scanning electron micro-

**Figure 3.** Applied pressure and suction schedules for PVHSC.

**Figure 4.** Drying rates of the as-cast body with different powder batches as a function of time at 25°C with 80% humidity.
3. Results and discussion

Well-formed alumina green bodies (110 × 110 × 20 mm$^3$) could be fabricated by PVHSC under appropriate casting and drying conditions.

In order to efficiently arrange the particle packing, it is known that the wet forming method from suspension or slurry is superior to dry pressing [21–23]. However, it is difficult to apply a uniform pressure over the entire volume of a large, thick body. Many studies have shown that in slip casting or filter pressing, the conventional practice of applying a static pressure during casting may produce a density deviation in the green body [6–15]. Nevertheless, slurries containing powders of large average size, wide size distribution and high spherical shape can produce wet cakes of more uniform packing structure by pressure vacuum hybrid slip casting (PVHSC) [27–30]. Unlike a typical slip casting process, this method is a hybrid pressure casting, in which the slip is cast by stepwise applying pressure to the casting chamber as well as drawing.

Figure 5. Partial density distribution along the thickness direction of alumina green bodies obtained by PVHSC: (a) schematic of small cuboid pieces obtained from each section in the green body and typical density distributions of the sections in cast alumina made from (b) H batch, (c) H + O batch, and (d) H + O + T batch.
the dispersion medium out of the chamber. The driving force of the slip casting in this process is the gradual pressure obtained by applying pressure stepwise to pass the liquid through the filter (pressure slip casting), or by applying vacuum under the filter to collect the filter medium (vacuum slip casting).

When the slip is loaded into the mold, water is drawn out by capillarity, leaving a layer of cake at the slip/mold interface. The kinetics of the casting process can be explained by Darcy’s differential equation for fluid flow through porous media [10,32–36]. An increase in cake thickness and a consequent decrease in cake permeability with casting time have been reported for pressure-slip-cast Al2O3 bodies [32]. The less permeable microstructures were attributed to particle rearrangement that slows the casting process as the cake thickness increases. The pressure drop across the consolidated layer (cake thickness) increases with increasing volume and casting time when the applied pressure is constant [10,32]. To control the kinetics of the casting process, a stepwise increase in the slip inlet pressure during slip casting can be applied to minimize this variation in the pressure drop [35–37]. Our previous works show that the slip inlet loading with five steps up to a maximum (0.5 MPa) and continuously water outlet pumping of −0.05 MPa are the best casting conditions for the PVHSC process [27–30]. The microstructure of cake subjected to the stepwise pressure loading was more uniform than that produced by static pressure loading.

In the drying process of the formed body, the maximum drying speed should be limited so that cracks or surface distortions do not occur in the ceramic body. During rapid drying, a water gradient is quickly established, resulting in greater free shrinkage. The free shrinkage gradient induces a stress profile in the plate which, in turn, results in the formation of a compression layer in the interior of the body and tension layers near the upper and lower surfaces, resulting in cracking and distortions [27,37–39].

Figure 4 shows the decrease in water content for the green bodies from three batches as a function of drying time at 25°C and 80% relative humidity. The H + O + T batch, which contained a large amount of fine powder, required a long time of about 250 h to dry completely. The cast bodies from the H and H + O batches required approximately 200 and 225 h, respectively, for complete drying. In the constant drying rate period, during which the slope of the water content versus time is constant, the surface of the cast body remains wet as water travels from the interior to the surface owing to the surface tension and capillary force generated between the powder particles during water evaporation [28,29,36]. This constant drying rate period was longest (approximately 125 h) for the cast body from the H + O + T batch and was approximately 75 and 100 h for the cast bodies obtained from the H and H + O batches, respectively. Further, during the constant drying rate period, the slope of the drying rate for the cast bodies from the H + O + T batch was 0.084 wt%/h, and those of the bodies from the H and H + O batches were 0.095 and 0.126 wt%/h, respectively. This result was attributed to an increase in packing density as the voids between large particles were filled with fine particles, making it difficult for the water in the interior of the cast body to move to the surface. When the constant drying period was complete, the water content of the cast body decreased dramatically, and the decreasing drying rate period began. This period has been found to be dominated by internal diffusion [28,29,36]. Because internal diffusion becomes difficult for the H + O and H + O + T batches, which have a relatively high fine powder fraction, a longer drying time is required.

Figure 5 shows the method of obtaining small cuboid pieces from each section of the green body (Figure 5(a)) and the typical density distribution of the sections within the upper, middle, and lower layers of cast alumina made from each batch (Figure 5(b–d)). The actual experimental packing density is lower than the ideal value with a spherical powder, depending on the particle shape, mixing technique and overall homogeneity [16,22,23]. This difference results in insufficient filling of fine particles in the voids created by the large particles skeleton structure. About 60% green density of the obtained alumina is similar to that of the pressure filtrated alumina from slurries with 10–50 vol% solid loading under low pressure conditions [4,6,15].

The density difference between several cuboid segments within the lower layer was smaller than those within the upper and middle layers, indicating that the lower layer was more uniform than the other two layers. Furthermore, the lower layer had a higher green density than the other two layers. This spatial difference in density is due to the fact that the large particles in three parts form a tightly or loosely filled skeleton structure, and the fine particles do not fill or fill the large gaps sufficiently [22,23].

Table 3 shows the mean values and standard deviations of the green densities of the sections in the alumina cast bodies made using each batch of powder.

| Green body | Layer | Mean density (%) | Standard deviation (%) | Density of whole body (%) |
|------------|-------|------------------|------------------------|--------------------------|
| H batch body | Upper | 58.0 | 1.0 | 58.4 ± 0.6 |
| H batch body | Middle | 58.3 | 0.5 | |
| H batch body | Lower | 59.0 | 0.4 | |
| H + O batch body | Upper | 58.5 | 0.5 | 59.0 ± 0.4 |
| H + O batch body | Middle | 59.2 | 0.4 | |
| H + O batch body | Lower | 59.4 | 0.4 | |
| H + O + T batch body | Upper | 59.1 | 0.3 | 59.4 ± 0.3 |
| H + O + T batch body | Middle | 59.3 | 0.3 | |
| H + O + T batch body | Lower | 59.8 | 0.1 | |
The H + O + T batch body shows a higher density and narrower density distribution than the bodies from the other two batches. The green densities of the formed alumina produced by PVHSC using the H, H + O, and H + O + T batches were 58.4%, 59.0%, and 59.4% of the theoretical value, respectively. Further, the standard deviation of the density was 0.1–0.3 for the body from the H + O + T batch, and the bodies from the H and H + O batches had wider density distributions (standard deviations of 0.4–1.0). The mean green density of the formed body in this experiment is lower than the cake prepared by the pressure-filtration (62.8% and 65%). However, the standard deviation in green density is narrower (0.6% and 1.5%) [6,15], which suggests the more homogeneous packing of the particles due to H-T-O treatment.

As a result, the body obtained by PVHSC of the H + O + T batch exhibited a uniform density distribution along the thickness direction. It is expected that there will be little cracking and deformation due to the low density variation of this body. Deformation arises as different parts of the body shrink to different extents, which, in turn, can be related to spatial variations in density within the body [37–40].

Figure 6 shows the typical density distributions of the sections within the upper, middle, and lower layers of sintered alumina made from each powder batch. Table 4 compares the mean values and standard deviations of the sintered densities of the sections. The sintered \( \text{Al}_2\text{O}_3 \) produced from the H batch showed a relative density of 98.8%, whereas that produced from the H + O and H + O + T batches had relative densities of 99.2% and 99.5%, respectively, which were more than 0.4% and 0.7% higher. The density of each layer made using the H batch was as follows: upper layer, 98.5 ± 0.6%; middle layer, 98.7 ± 0.4%; lower layer, 99.1 ± 0.4%. The sintered bodies obtained from the batches that included fine powder exhibited a more uniform density distribution along the thickness direction. The sintered density of the alumina produced from the H + O + T batch was 99.5% of the theoretical value. Further, the standard deviation of the sintered density of each section piece was 0.2%, which is less than that (0.3%) in the formed body. As in the formed body from this batch, the more uniform density of the sintered body suggests less likelihood of cracking and deformation.

Figure 7 shows the microstructure of the \( \text{Al}_2\text{O}_3 \) from each powder batch after sintering at 1650°C. The microstructure of the sintered \( \text{Al}_2\text{O}_3 \) fabricated from the H batch, which contained only coarse powder, corresponded to a density of 98.8%; large intergranular pores were observed, and densification was incomplete. By contrast, the microstructures of the sintered \( \text{Al}_2\text{O}_3 \) from the H + O and H + O + T batches, which included fine powders, showed fully densified microstructure with little voids between the large particles.

Figure 8 shows SEM images of the sintered microstructure of each layer of the \( \text{Al}_2\text{O}_3 \) made from the H + O + T powder batch. Fine microstructure with grains several micrometers in size was observed. No systematic changes in the sintered microstructure were observed across the cross section or height of the specimen. Nearly identical microstructures were

| Sintered body | Layer | Mean density (%) | Standard deviation (%) | Density of whole body (%) |
|--------------|-------|-----------------|------------------------|--------------------------|
| H batch body | Upper | 98.5            | 0.6                    | 98.8 ± 0.5               |
| H batch body | Middle| 98.7            | 0.4                    | 99.2 ± 0.4               |
| H batch body | Lower | 99.1            | 0.4                    | 99.5 ± 0.2               |
| H + O batch | Upper | 99.0            | 0.4                    | 99.2 ± 0.3               |
| H + O batch | Middle| 99.2            | 0.3                    | 99.5 ± 0.3               |
| H + O batch | Lower | 99.5            | 0.3                    | 99.5 ± 0.2               |
| H + O + T batch | Upper | 99.3            | 0.2                    | 99.5 ± 0.2               |
| H + O + T batch | Middle| 99.4            | 0.2                    | 99.5 ± 0.2               |
| H + O + T batch | Lower | 99.6            | 0.2                    | 99.5 ± 0.2               |

Figure 6. Typical partial density distribution along the thickness direction of the sections in the sintered alumina produced by PVHSC using (a) H batch, (b) H + O batch, and (c) H + O + T batch.
found in the upper, middle, and lower layers. Further, no significant differences in grain size distribution were observed between the layers. This observation suggests that it is mainly the high degree of uniformity in the green body that affects the final microstructure of this dense Al₂O₃.

A simple method of adding small particles to a large particle matrix with geometrically accurate size and amount allows us to produce Al₂O₃ ceramics with uniform density distribution and higher density. Possible application of this method to increase the product size with uniform microstructure seems promising.

4. Conclusions

We successfully prepared Al₂O₃ with a uniform density distribution by PVHSC using Al₂O₃ powder batch with adding fine particles to a large particle matrix with geometrically accurate size and amount ratios. Well-formed green bodies were fabricated by PVHSC by applying a stepwise increase in pressure up to 0.5 MPa combined with suction at a pressure of −0.05 MPa. The cast bodies fabricated from the batches including fine powder showed a longer constant drying rate period and drying completion time compared to the powder batch containing only coarse particles. The green densities of the Al₂O₃ made from the H only (typical normal wide size distributed powder), H + O, and H + O + T batches were 58.4 ± 0.6%, 59.0 ± 0.4%, and 59.4 ± 0.3% of the theoretical value, respectively, and showed an increasingly uniform density distribution along the thickness direction. After sintering at 1650°C, the density of the Al₂O₃ obtained using the H + O + T batch, which contained fine particles, was close to the theoretical value, and the density distribution was uniform.

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