Research Article

An Optimization of Particle Size and Additives of Slip Cast Alumina Samples to Reduce Warpage and Porosity

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Alumina body densification using slip casting was investigated with low warpage rates. In order to optimize slurry and sintering, 1600 °C has been used. As a starting material, three types of alumina were used. Alumina has been used in conjunction with PMAA, and carboxymethyl cellulose in order to prepare the slurry. It was found that warpage rates were reduced when coarser particles were included in the composition. An acid treatment of the slurry was carried out in order to improve densification of the alumina body and to reduce warpage. This study examines the microstructure of a sintered alumina body. In the investigation, it was found that preparing a dense product reduced porosity and warping rate by 1.1% and 4%, respectively.

1. Introduction

Al2O3, commonly referred to as aluminum oxide, is a high-performance ceramic material that possesses excellent thermal, chemical, and physical properties. Additionally, alumina is used as abrasives, firebricks, and IC packages. Alumina is mainly produced by the Bayer method using bauxite, which delivers more than 45 million tons to the world each year. The corundum/sapphire super lattice illustrates the period of Al2O3. For electronic packaging, corrosion resistance ceramics, erosion-resistant ceramics, and erosion-resistant ceramics, Al2O3 is usually utilized and concentrated in high temperatures. The most thermodynamically stable form of alumina is the polymorphic phase α-Al2O3. There are 5.2 cell sides and a rhombohedral plane angle of 55° for each unit cell of α-Al2O3. In the Bayer process, Al2O3 powders have the greatest purity of 99.6–99.9%, which makes them suitable for assembling refractories, spark plugs, and substrates of coordinated circuits [1–3].
more basic parameter for sintering [7]. High densities and little grain sizes of alumina ceramics results in high strength and better mechanical properties [8].

The powder particle shapes may be needle-like, spherical, plain, platelet, or equiaxial. As compared to all other shapes the spherical-shaped powder of various sizes results in the best packaging capacity. The high-level physical and chemical properties of alumina ceramic request demand ultrafine and high purity alumina powder [9]. Due to their large surface area, fine ceramic particles often form agglomerates during processing, resulting in suspensions with extremely high viscosities and poor packing properties. We have therefore added coarser particles to reduce warpage. In order to achieve reduction in warpage and porosity, we have varied the particle size, amount of fines, amount of coarses, amount of solids, amount of dispersant, and the water/plaster of Paris content of the molds.

2. Experimental

Experiments were carried out on raw alumina powders A (particle mean size 0.7735 μm), B (particle mean size 0.85156 μm), and C (particle mean size 3.69587 μm). As part of this study, Particle size (PS) and Particle Size Distribution (PSD), of the starting powders were manipulated, and the drying-shrinkage behavior of the powders was evaluated to assist us in reducing the warpage rate at the green stage. For slurry-based colloidal processing, high powder loading is critical to obtain a high packing density of the green body, which facilitates minimal drying and sintering shrinkage, reducing the final component warpage [10]. However, high powder loading is complex for fine powders due to the high tendency of agglomeration, so coarser particles were also used. Weights of the above materials were carefully taken based on their stoichiometric composition (99 wt% Al₂O₃). The above composition was mixed with CMC and PMAA at concentrations ranging from 0.1 to 0.6 weight percent. To generate enough repulsive force, a dispersant agent is necessary to build up an electrical double layer and, in turn, reduce suspended particle zeta potentials by compressing double layers by absorbed counter ions [15], Slurry processes have a major impact on colloidal microstructure and mechanical properties as a result of the quantity of polymer dispersant used. When air bubbles are entrapped during milling, the slurry’s high viscosity will cause them to be difficult to remove, eventually resulting in defects [16, 17]. It is therefore important to optimize the amount of dispersant.

The prepared alumina slurry is poured into the designed mold. The characteristics of the mould played an important role in the reduction of green warpage. Variation in the water to the plaster ratio has a direct influence on product characteristics [18]. According to Lambi and Adegoke [19], the hardness, strength, durability, and weight are directly related to the mix ratio of water and plaster used in producing the mould. The percentage of water absorption for the samples prepared from different water to plaster ratios is determined. POP mould with an average absorption water level (74.52%) was used. In order to fill the hollow, the continuous slurry is added since the slurry immediately absorbs water from the plaster mould. As the mould absorbs no more water, the slurry begins to harden and becomes hard, then the drain casting is done. After 2 hours molding is done. Due to shrinkage, the green product comes out of the plaster mould automatically once it leaves the wall. Microstructures of green plants are commonly characterized by (i) Archimedes immersion, which measures density; and (ii) electron microscopy, which measures qualitative properties. A period of around 24 hours was required for the green products to be dried under atmospheric conditions at the RT. At this stage, some water is removed.

A period of 24 hours was then needed for the green product to completely dry (∗ 99%) in the oven at 110°C. As soon as the alumina specimen and dried sample are polished with sandpaper, impurities are removed from their surface. The green warpage has been reduced by all possible means; after this sintering of green products is done at under 1600°C temperature with varied soaking time. Heat is used in the sintering process to achieve the desired microstructure of the green body. Based on the complexity of the starting material, the stage may involve quite a few changes [20, 21].

A Siemens D-500 powder diffractometer with Cu K-radiation was used for X-ray diffraction (XRD) to identify the phases in sintered samples. A 5°C/min heating rate was used to prepare the powders from the compacts for XRD analysis. The bulk density of the samples was measured by the Archimedes principle. Zwick 1445 universal testing machine was used to measure the modulus of rupture (MOR) of rectangular samples sintered at 1600°C. The samples were subjected to 10 physical tests, with the results averaged. Polished and thermally etched samples were examined using the Cambridge Instruments-5526 scanning electron microscope after being polished and thermally etched for 1 h at a temperature lower than the sintering temperature.

Figure 1 shows the SEM and XRD image of as received Al₂O₃ powder particles. The Aumina powder particles are in
uneven shape. XRD pattern corresponded to pure alpha Al$_2$O$_3$ phase as per alpha phase JCPDS (Match Entry C 96-100-0033) [22].

3. Results and Discussion

It is possible to determine the average particle size and the range of particle sizes based on the particle size distribution. Table 1 shows the particle size distribution of the raw alumina powder namely fines, medium, and coarse respectively. The particle size measurement reveals bi-modal size distribution which is depicted in Figure 2.

Several factors contribute to slip casting performance, including particle density, homogeneity, and rheological behavior. The specimen was manufactured directly from the suspension of ceramic powders [23]. Solid loading, particle shape, and particle-particle interaction forces control the suspension properties and the structure of slip cast bodies [24]. A slurry’s structure is commonly responsible for shear-thinning behavior. A higher specific surface area of fines coupled with a dominant role played by surface forces explains the more pronounced shear-thinning characteristics of fines. As the shear rate increased, the coarser particles became progressively thicker, whereas the fines were shear-thinning over the entire shear rate range. Hydrodynamic interactions between particles become more important at high shear rates. A rise in PS can even lead to their dominance, resulting in a tendency for the particles to become more individualized. It is also important to note that coarse particles need to have a greater average separation distance in order to slide over one another [25]. Increasing alumina powder coarseness increased the green density, reaching a maximum on alumina powder coarsening. Particle packing in suspension results in a gradual decrease in viscosity with increasing particle size. Table 2 shows different compositions by varying fine and medium.

One can observe from Table 4 that the density of a homogeneous slurry increases with the solid content, but it also depends on the flow behavior. The flocculation of alumina particles occurs in a less viscous slurry or in a highly viscous slurry. The particles come close enough to hinder each other’s movement because of the attractive forces caused by high alumina loading. The dispersed particles start to agglomerate as a result of an insignificant repulsive force acting among them. The gravity forces begin to settle particles in less viscous slurries (slurries with high solid content) at optimal conditions. To reduce agglomeration and warpage dispersant has to be introduced in the slip. PMMA generates highly charged surfaces of the particles that will result in a strong double layer repulsion and decreases the electrostatic attractive forces [27]. This means that it is possible to achieve stable aqueous colloidal dispersion. Since most of the changes of alumina particle surface charge have been nearly beside the isoelectric point at the very limited range of pH 9.8. Table 3 shows that from viscosity measurements, the amount of dispersant agent significantly contributed to decreasing the viscosity and determining the effective concentration of the dispersant that allows the viscosity to reach a minimum steady, and thereby analysing the viscosity and shear rate relationship we may come to understand the optimal use of a dispersing agent in the slurry.
Table 1: PSD of the starting powders.

| Particle size | Fine       | Medium     | Coarse     |
|---------------|------------|------------|------------|
| Median size (µm) | 0.77347   | 0.84447    | 3.50281    |
| Mean size (µm)   | 0.77356    | 0.85156    | 3.69587    |
| Standard deviation (µm) | 0.0729     | 0.0891     | 1.6298     |
| Diameter on cumulative (%) | 0.7735     | 0.8445     | 3.5028     |

Figure 2: Particle size distribution (a) fines; (b) medium; (c) coarse.

Table 2: Different composition of starting raw material.

| Composition | Fines (%) | Medium (%) | Coarse (%) |
|-------------|-----------|------------|------------|
| I           | 70        | 30         | 0          |
| II          | 60        | 30         | 10         |
| III         | 50        | 30         | 20         |
| IV          | 40        | 30         | 30         |
| V           | 30        | 30         | 40         |

Table 3: Rheology of the slip of different composition.

| Composition | Density (g/cc) | Viscosity (ε) | Fluidity (S) | Thixotropy (S) |
|-------------|----------------|---------------|--------------|----------------|
| I           | 1.76           | 2.0           | 20           | 23             |
| II          | 1.78           | 2.5           | 25           | 28             |
| III         | 1.82           | 2.5           | 25           | 28             |
| IV          | 1.74           | 4.3           | 43           | 46             |
| V           | 1.76           | 4.4           | 44           | 48             |

Table 4: Batches with different solid loadings.

| Composition | Solid loading (%) | Observation       |
|-------------|------------------|------------------|
| A           | 40               | Unable to cast   |
| B           | 45               | Unable to cast   |
| C           | 50               | Green shrinkage  |
| D           | 55               | Optimum condition|
| E           | 60               | High friction    |

Table 5: Analysis of the viscosity and shear rate by varying dispersing agent.

| Composition | Dispersant (%) | Viscosity (ε) | Shear rate (1/S) |
|-------------|----------------|---------------|------------------|
| 1           | 0              | 4.4           | 0.022            |
| 2           | 0.1            | 3.6           | 0.0277           |
| 3           | 0.3            | 2.2           | 0.0454           |
| 4           | 0.6            | 1.8           | 0.055            |
| 5           | 0.9            | 1.7           | 0.058            |
| 6           | 1.2            | 1.7           | 0.0588           |
From Figure 3 the viscosity and shear rate relationship has been observed. The suspensions have a significant shear-thinning behavior at intermediate shear rates. Since the coarse particles have been added at high shear rates they showed shear thickening behaviour thus intermediate shear rate showed good results by adding 0.3% of dispersing agent. Approximately 2°C per minute is added to all samples during sintered at 1600°C. Instead of melting, the powder is joined together and its porosity is reduced, which is what is called densification in the fabrication process, which is performed by atomic diffusion in the solid state. The term “solid-state sintering” is often used to describe this type of sintering. A consolidated mass of particles reduces its surface free energy during sintering. Atom diffusion can reduce energy by causing the body to densify. Densification is improved with longer soaking times. Finally, composition III has been prepared by adding an optimal amount of binder (CMC), 0.3% of PMAA, and the specimen was fabricated with low warpage and porosity. The physical and mechanical properties of the specimen were also analyzed.

Flexural strength value gives a material’s ability to resist deformation under load [28]. For measuring flexural strength, three-point bending was used. The value obtained for composition III and found to be 418 MPa. Table 6 shows the physical and mechanical properties of the specimen made of composition III.

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SEM was used to study the microstructural behaviour of the alumina samples are presented in Figure 4. In comparison of the SEM images, composition 3 is clearly characterized by close packing and smaller grain sizes, as well as the higher sintered density. In contrast to the other composition cast samples, there is a better coordination among the smaller grains and greater interparticle spaces between the coarser grains, resulting in higher densification [22].

|   | Bulk density (g/cc) |   |
|---|-------------------|---|
| 1 |                   | 3.75 |
| 2 | Apparent porosity (%) | 1.1 |
| 3 | Water absorption (%)   | 0.07 |
| 4 | Flexural strength (MPa) | 418 |

Figure 4: SEM image of alumina cast prepared using different compositions (a) composition 4; (b) composite 3.
4. Conclusion

Slip casting is used in this study to create an alumina body that contains the inherent packing structure of powder particles. A slurry flowing with water has been observed to have an effect on density and microstructure. An excessive amount of water and a low amount of water in the slurry cause particles to flocculate, resulting in an inhomogeneous slurry. Slurry preparation with 45% water results in heterogeneous slurries.

Changing the composition by adding coarse particles which act as a skeleton and increase the stability of the product. Sintering product density is observed to be affected by soaking time. The densification improves and the porosity decreases as the holding time increases.

A better density can be achieved by better densification of the final body due to the homogeneous grains created by the acid treatment. We were successful to minimize the porosity up to 1.1%. Initially, the warpage rate was 36% and it has been reduced up to 4% by preparing a dense product. Measurements of slurries’ viscosity based on shear rate indicated the state of dispersed alumina particles in aqueous media and contributed to the effective amount of dispersant agent required. Rheological measurements could be proposed as a route for designing the casting technique that causes particles to dissociate. Consequently, the stability of suspensions improves leading to achieving particle packing and high green bulk density of cast body with more uniform microstructures.

The mechanical properties of the alumina body also improve as it becomes denser. The flexural strength is found to be 418 MPa. As a result of increasing the sintering temperature and soaking time, alumina is densified by controlling the particle size and water content. In addition to densifying and homogenizing grain size, acid treatment also produced dense, compact alumina bodies.

Data Availability

Data is available with the corresponding author. Will be submitted upon request.

Conflicts of Interest

The authors declare that there is no conflicts of interest.

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