FABRICATION OF ALUMINA WARES BY SLIP CASTING TECHNIQUE

M. Arif, S. K. Durrani1, M. Akram, N. Hussain and B. A. Hasan2
1Materials Division, Directorate of Technology, PINSTECH, Islamabad.
2Department of Chemical and Materials Engineering, PIEAS, Islamabad.
* durranisk@gmail.com, arif3453@yahoo.com

ABSTRACT

Various processing methods are being used for alumina ceramic casting. In the present work, slip casting process was used to minimize the material wastage as compared with other conventional processing routes. Fine grade of alumina powder (1µ, 0.3µ and 0.05µ) was used. The suspension was prepared with water and poured in to a hami-hydrated Plaster of Paris (local market) and its characteristics were studied by controlling the pore size of capillaries and volume fraction of open channels. After drying the casting and slip from the mould, water ratio in alumina is adjusted to maintain various technological parameters. Pre-sintering was employed to the green stuff at 1150°C for controlling the heating/cooling rate. Sintering was carried out at 1650°C. The sintering densities and shrinkage was studied by using dilatometry. The microstructure and phase analysis was performed by scanning electron microscope and X-ray diffraction. The mechanical and thermal properties were measured in detailed with the help of TG/DTA.

INTRODUCTION

Ceramics are composed of the most abundant elements on earth: silica, alumina, and magnesia. Ceramics are classified as crystalline or non-crystalline. Crystalline ceramics form lattice structures of repeating patterns. Non-crystalline ceramics have low porosity and their atomic structure is not ordered, e.g., glass. Traditional ceramics are made from naturally occurring earth elements referred to as clay. The principal elements found in these clays are oxygen, silicon, and alumina. Ceramics is further classified as structural and refractory ceramics. Refractory ceramics are used in crucibles for melting metals, and for brick linings of large steel making furnaces or kilns for fire ceramic white-ware. They are usually composed of alumina (Tm=2050°C) and silica along with other oxides: MgO (Tm=2850°C), Fe2O3, TiO2, etc., and have intrinsic porosity typically greater than 10% by volume. Specialized refractories, BeO, ZrO2, mullite, SiC, and graphite with low porosity are also used. Ceramics have various properties such as poor conductor of heat and electricity, excellent insulators and have lowest thermal expansion of all materials.

In traditional ceramics, silica, clay, and refractory materials (alumina, zirconia) in powder forms are carefully blended. Most ceramic products are formed by casting. Slip casting is used to form hollow shapes. Slip is low viscosity ceramic slurry i.e. a well mixed ceramic powders (Al2O3) suspension or slurry in solvent (water) is called slip. The slip is poured into a Plaster of Paris mould; the solvent of suspension is extracted into the pores of the mould via the capillary driving force or capillary suction. The slip particles...
are, therefore, consolidated on the surface of the mould to form a layer of particles or a gel-layer. When the desired thickness is achieved the remaining slip is poured from the mold leaving the desired product\textsuperscript{3}. The layer thickness depends on setting time; hollow objects are formed by draining mould (drain casting); if the mould is not drained during the process, a solid component results (solid casting). On drying the cast shrinks and pulls away from the mould, permitting release and extraction by disassembling the mould\textsuperscript{4,5}. A casting process has a merit of being able to reflect complicated shapes. In the present work we have tried to improve the characteristics of fabrication of alumina-wears using porous ceramic mold slip casting process because there is minimum material wastage as compared with other conventional process routes and material utilization is maximum as well as dimensional accuracy, uniformity of microstructure and reduction of machining cast.

1. EXPERIMENTAL
Materials and Methods
The chemicals \(\text{Al}_2\text{O}_3\) (99.9\% Fluka), Plaster of Paris used were A.R. grade and used without further purification. Three types of alumina powders (1\(\mu\), 0.3\(\mu\) and 0.05\(\mu\)) were used.

Mould preparation
Plaster of Paris (anhydrous, hygroscopic) was used for mould preparation. The setting time was studied using different ratio of water and plaster of Paris, as shown in Table 1.

Three types of alumina powder 1\(\mu\), 0.3\(\mu\) and 0.05\(\mu\) were used for slurry preparation. Alumina: water ratio was studied by using different ratio of alumina in fix amount of water by using ultrasonic bath for homogenization. Specific gravity and viscosity measurement were measured. Two different viscosity modifiers or deflocculates HCl and AlCl\textsubscript{3} were added to increase the flowing properties and decrease the viscosity. Once the slurry

<table>
<thead>
<tr>
<th>Sr.#</th>
<th>Water: plaster</th>
<th>Weight of mould (gram)</th>
<th>Water (ml)</th>
<th>Setting time of water (Sec.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1:1</td>
<td>56.14</td>
<td>5</td>
<td>43</td>
</tr>
<tr>
<td>2</td>
<td>1:4</td>
<td>58.67</td>
<td>5</td>
<td>12</td>
</tr>
</tbody>
</table>

was prepared it was poured in the mould (plaster of Paris). In drain-casting the mould is filled with slurry. The sequence of steps for slip casting of alumina is given in Figures 1 and 2. The thickness of the cast increases progressively with time, until the required thickness was attained. Then the mould is inverted and excess slurry drain. After drying, the cast is removed from the mould and allow to dryness in the oven at 100\(^\circ\)C for 24 h. For finishing and trimming the green casting was placed in the tube furnace for pre-sintering at 1150\(^\circ\)C with heating rate of 2\(^\circ\)C for 2h. The compacts were sintered using two-step heating program. In first step the compact was heated up to 1150\(^\circ\)C for 2-4 h to ensure good nucleation and to initiate crystal growth. In second step, heating was carried out with different heating rates up to sintering temperatures of 1650\(^\circ\)C. The sample was kept at the sintering temperature for a sufficiently long time to achieve the desired crystal growth. Density is determined using Archimeades principle.
Characterization
The raw material (Al₂O₃) and the prepared mold green and sintered (Al₂O₃) ceramic materials were characterized using various techniques; for the measurement of weight loss the combined TG-DTA analysis and for shrinkage/expansion behaviour dilatometry were performed using Netzsch STA-409 thermoanalyzer and dilatomter (Netzsch, DIL, 409) with heating rate of 10°C/min up to 1100°C in static air respectively. The crystalline and phase analysis were conducted by X-ray diffraction using Rigaku Geiger flux instrument with Cu Kα radiation (λ = 0.154056 nm). The XRD data were collected in the 2θ range from 15°<2θ<80° by step-scanning 0.5° increments and scanning rate of 5°/min.

![Figure 1. Block diagram of steps in slip casting.](image)

2. RESULTS AND DISCUSSION
The mixing process of powder and water is very important because the powders uniform dispersion has a great effect on the strength of porous ceramic mold. Slurry is poured into the alumina mold. Once slurry hardens enough to, it is separated from the alumina mold. Then the green part which means hardened slurry in non-sintering, has to be removed water at 100μm with vacuum condition. Micro-pores are caused by getting rid the green part out of water during vacuum drying, have an effect of preventing cracks throughout the sintering process. At last process, the sintering converts the green part to the sintered part. Because the porous mold is made from ceramic, the process of rising and decreasing temperature should be done slowly. Otherwise cracks are taken place on the surface of porous ceramic mold. The results of Table 1 indicates that 1:1 water/plaster ratio give better result than 1:4 ratio and the cast alumina easily slip from the mold. The amount of water has much effect on the efficiency of molds. Decreasing the water contents would decrease the pore size but increasing suction pressure.

In this study, we adopt the porous ceramic mold as a casting mold for aluminum casting. Therefore it doesn't need to have the strength like as conventional ceramic products do. So we prepare two types of specimen to evaluate the properties i.e. an A-type specimen for TG/DTA and diltometry studies and B-type for phase analysis of sintered alumina. Figure 3 shows the TG-DTA thermograms of Al₂O₃ powder derived from precursor solution. The sharp endothermic peaks were observed at 200°C and 1200°C. The position of the minimum on DTA curve is basically determined by the chemical composition of the separated phase or by its transformation temperature. The exothermal crystallization peak, its position and its shape, characterize the crystallization process. The exothermal maximum corresponds to the separation of the crystalline phases. All the volatiles were completely removed at 900°C as observed on TG/ DTA curves.
Figure 4 displays the dilatometry curve of the green slip cast alumina mold at 1000°C. The curve shows the change in length during sintering of pressed pellet. There was no change in length on heating up to temperature of 275°C. However, a large

Figure 2. Fabricating process of porous Al₂O₃ ceramic mold. (a) Pure Al₂O₃ powder (b) the temporary mold, which is made from Plastic of Paris + Water (c) the slurry was prepared, which consists of Al₂O₃ powder and water (d) green ceramics Al₂O₃ (e) Al₂O₃ ceramic mold heated at 1150°C (f) sintered Al₂O₃ ceramics at 1650°C.

Figure 3. TG/DTA curves of alumina used for slip casting.

Weight loss (%)  

0 200 400 600 800 1000 1200

Temperature °C

0 200 400 600 800 1000 1200 1400

102.5 100.0 97.5 95.0

TG

DTA
shrinkage occurred as a result of evolution of decomposed species between 400 and 800°C. There is gradual increase in dimensional changes (lattice parameters) which are due to development of slip cast alumina phase. On the basis of dilatometry results, sintering was conducted between 900-1100°C in order to obtain the full crystalline phase.

Figure 5(a-b) shows XRD pattern of the Al₂O₃ powder (Figure 5a) and slip cast sintered pellet at 1650°C (Figure 5b) for 2h in air. All peaks appearing in both diffractograms show single phase crystalline Al₂O₃ with a well-pronounced rhombohedral structure; however, crystallinity enhances after sintering at 1650°C. The main peak centers at 2θ = 35.15º and corresponds to crystal plane with Miller indices of 104 which is characteristic of Al₂O₃. The XRD peaks were indexed in terms of rhombohedral structure according to standard JCPDS 82-1468⁶.
3. CONCLUSIONS

- Different Shapes of crucibles and boats were formed by slip casting process.
- Surface area of increase with decreasing particle size, in the present case for getting the best Slurry, the size of alumina 0.05 micron was provided the best result than 1 and 0.3 micron.
- Slip casting alumina-wears can be used as parts for high temperature operated instruments, furnace parts, turbine and boiler parts, machine parts and gears also surgical and medical parts.

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