CHAPTER III

EXPERIMENTAL

3.1 Raw Materials

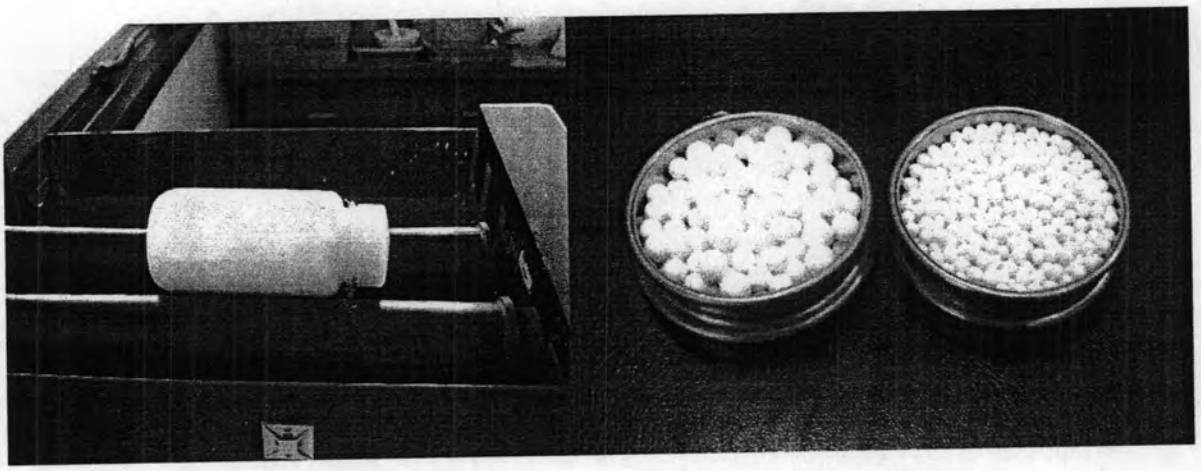
A high-purity (99.99%) commercial alumina powder (TM-DAR, Taimei Chemicals, Co. Ltd., Japan) has been used for preparing alumina slurry. The mean particle size was determined by dynamic light scattering technique (DLS; Malvern Zetasizer 300HSA, UK).

A commercially ammomium salt of poly(methacrylic) acid (NH_4^+-PMAA; Aron A6114, MW 6000, Toagousei Co, Ltd., Japan) as dispersant was added into the slurry to prevent agglomeration of suspended alumina particles.

Additionally, a commercial organic binder, polyvinyl alcohol (PVA, MW 13,000-23,000, Sigma-Aldrich, Inc.,USA) has also been employed to enhance mechanical strength of green body prepared in each experiment.

3.2 Preparation of stabilized alumina suspension with high solid content adding with organic binder

Alumina slurries for slip casting were prepared by dispersing the alumina powder of 70, 75 and 80 wt% in demineralized water. The stabilized suspension consisted of the alumina slurries mixed with the optimal dispersant concentration of 1.18, 1.25 and 1.5 wt% with respect to the alumina content of 70, 75 and 80 wt% was added with binder in the range of 0-1.5 wt%. The amount of dispersant and binder use in this research is expressed on a dry weight of the powder basis. The prepared suspensions were then ball-milled for 24 h by high-grade alumina balls in a Polyethylene (PE) container shown in Figure 3.1.



(a) (b)

Figure 3.1 Preparation of alumina slurries by ball milling
(a) mixing process in PE container (b) high-grade alumina balls

3.3 Preparation of green body from well-dispersed alumina slurries by slip casting in gypsum mold

The well dispersed alumina slip after ball milling process was degassed in vacuum chamber for 30 min before slip casting to eliminate gas bubbles remaining in slurries. The schematic of the process before slip casting is illustrated in Figure 3.2.

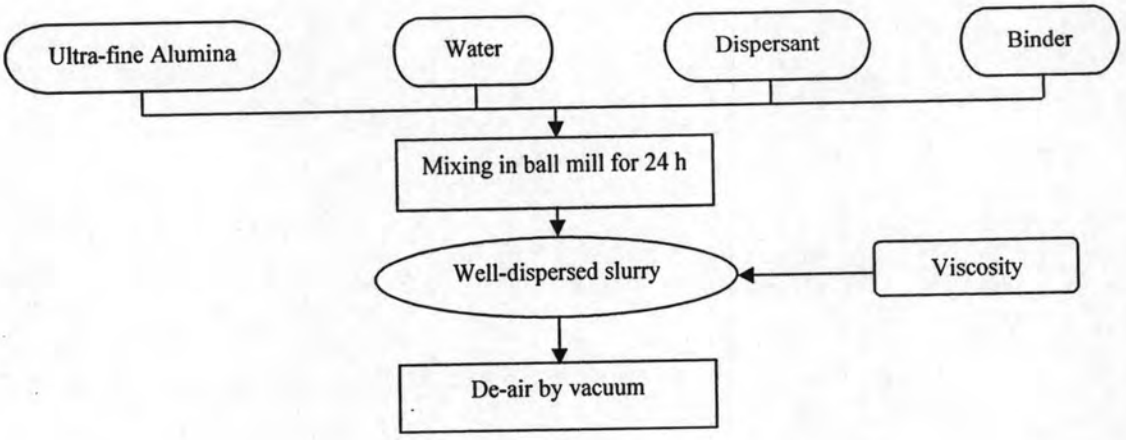


Figure 3.2 Flowchart of process before slip casting

The slip casting method for prepare alumina green body commonly use porous gypsum mold as a pattern form. In this research, there are three types of assembled gypsum mold such as pellet shape (30 mm diameter and 4 mm thickness), cylindrical shape (12 mm diameter and 70 mm length) and alphabet shape (as shown in Figure 3.3).

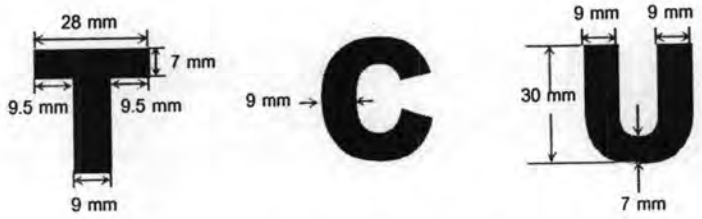
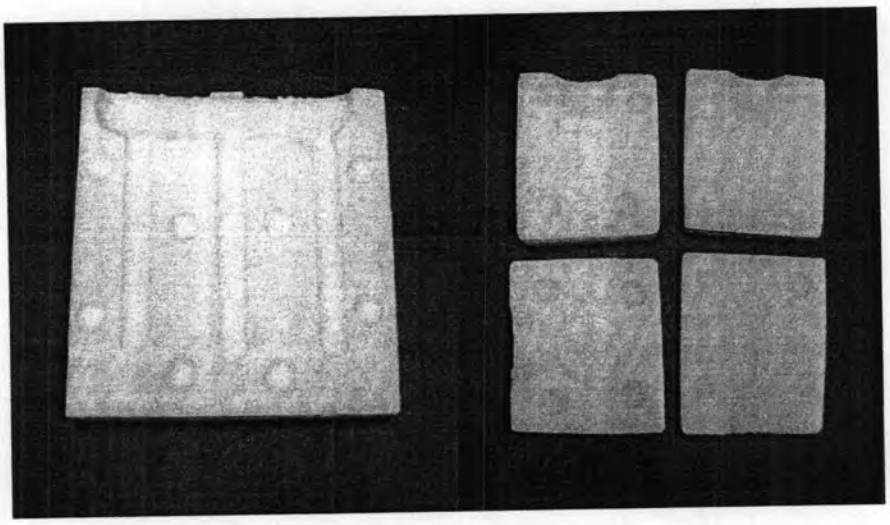


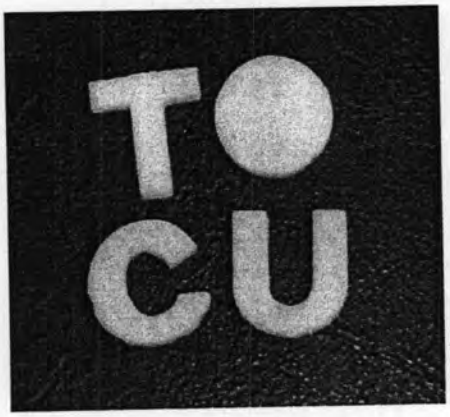
Figure 3.3 Template of alphabet shape with 4 mm thickness

The gypsum mold was prepared by added gypsum plaster (from Lafarge Prestia Co., Ltd., Thailand) into water by the ratio of 70:100 (water : plaster) and soaked in water for 1 min. Gypsum paste was then stirred with spatula for 5 min to be a homogeneous slurry and was shaken for 1 min in order to remove the bubble. The gypsum paste was poured into the prototype mold and placed at room temperature for 2-3 h to set the desired shape of gypsum mold. Before using gypsum mold for casting the slip in each cycle, it must be dried at 45°C to reduce the water content and to enhance the water absorption efficiency. The gypsum molds in this work are demonstrated in Figure 3.4 (a).

After casting, the green compacts were taken out from mold and were dried in the oven at 45°C for 24 h and then re-dried again at 80°C for 6 h and 120°C for 24 h, respectively. As the immediately vaporized moisture in the green body, the steps of drying were usefulness to prevent the breakage of the green compact. The alumina green body prepared by slip casting was shown in Figure 3.4 (b).



(a)



(b)

Figure 3.4 Equipment for preparing alumina green body
(a) The assembled gypsum mold with pellet and alphabet shape
(b) The alumina green body with pellet and alphabet shape

3.4 Calcination, HCl treatment and sintering condition

After dried in the oven, the green compacts were calcined at 800°C for 2 h with a heating rate 10°C/min. The calcination process was used to eliminate the organic content in the specimens and also prevent the slaking of the sample during the next step of acid leaching.

The calcined specimens were then subjected to acid treatment process by dipped in the 1M of HCl for 1 h so as to remove calcium ion contamination from gypsum

mold during casting. The acid in calcined bodies was washed with de-ionized water until no AgCl could be detected (using $AgNO_3$ for HCl detection) and dried.

The treated alumina specimens were dried and then sintered at 1250–1400°C at the heating rate of 10°C/min for 0-5 h in an atmosphere furnace (Vecstar furnace, England) and vacuum furnace (Hi muti-5000, Fujidempa, Japan).

After the pre-sintering process, the sintered alumina specimens with higher than 95% of theoretical density were post-sintered with hot isostatic pressed (HIPed) (Dr.HIP, KOBELCO) at 1300°C for 2 h under 150 MPa in argon atmosphere to achieve fully density and good transmittance The work flow of experiment after slip casting is shown in Figure 3.5.

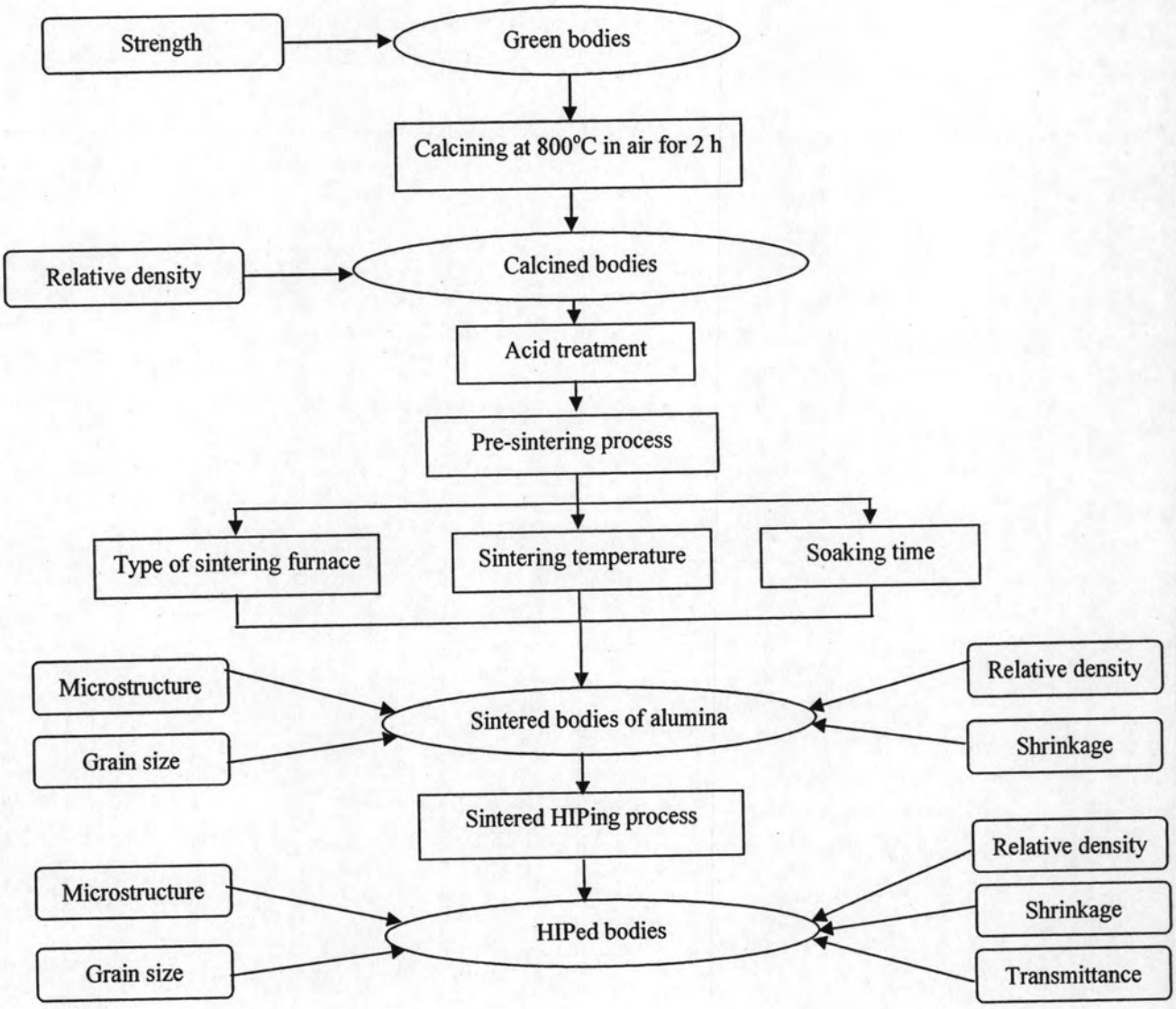


Figure 3.5 Schematic of process after slip casting

3.5 Characterization

3.5.1 Viscosity of alumina slurry

The rheological properties of alumina slurry were characterized by a viscometer (Brookfield DV-E, Brookfield Engineering Laboratories Inc., Massachusetts, USA). The alumina slurry was measured the viscosity with shear rate in range of 3-100 rpm.

3.5.2 Strength of green body

The green bodies with cylindrical shape (12 mm diameter and 70 mm length) were measured the strength by three points bending using a strength testing machine (HT-8116, Hungta Instrument Co.,Ltd.).

3.5.3 Bulk density of calcined and sintered body

Bulk density of alumina specimens was determined by the Archimedes method and calculated by equation (3.1).

$$\text{Bulk density} = \left(\frac{W_d}{W_{sat} - W_{sus}} \right) \times \rho \qquad (3.1)$$

Where, W_d is dry weight, W_{sat} is saturated weight, W_{sus} is suspended weight and ρ is water density at measurement temperature (Reed, 1995).

In addition, the relative density was calculated from its bulk density and theoretical density using the following equation:

$$\% \text{ Relative density} = \frac{\text{Bulk density}}{\text{Theoretical density}} \times 100 \qquad (3.2)$$

In this experiment, the theoretical densities of pure Al_2O_3 used for calculation is equal to 3.97 g/cm^3 was used for calculation.

3.5.4 Microstructure and grain size observation

The microstructure and grain size of alumina specimens was characterized by scanning electron microscope (SEM) (JSM-6480LV, JEOL Ltd., Tokyo, Japan) or atomic force microscope (AFM) (Nano Scope IV, Veeco Micrology Group). The specimens were manually polished with silicon carbide powder, grit No. 600 and 2500, and finished with 1 μm diamond paste. The polished specimens were etched at sintering temperature for 15 min. The average grain size was determined from the SEM or AFM pictures using Image-Pro Plus version 3 program (Media Cybernetics, Inc., USA).

3.5.5 Dried and sintered shrinkage

Shrinkage of specimens was measured using vernier caliper. The shrinkage after drying and sintering was determined by the equation (3.3)

$$\% \text{ Linear shrinkage} = \left(\frac{L_g - L_d}{L_g}\right) \times 100 \qquad (3.3)$$

Where, length of green specimen (L_g) and length of dried specimen (L_d)

3.5.6 Transmittance of alumina specimens

The transmittance of specimens after HIPing was characterized by UV-VIS spectrophotometer (Perkin Elmer Lambda650) with the wavelength between 200-900 nm. Before measured by UV-VIS spectrophotometer, the specimens were ground to thickness of 0.8 mm by BIWAJIMA GIKEN, Japan.