สำนักงานวิทยทรัพยากร
จุฬาลงกรณ์มหาวิทยาลัย

CHAPTER IV

EXPERIMENTAL

4.1 Raw Materials

Commercially available, α -Al_2O_3 powder (TM-DAR, Taimei Chemicals, Co.,Ltd., Japan) used in this study was alumina powder with purity of 99.99%. Their important properties, the mean particle size was determined by dynamic light-scattering technique (DLS; Malvern Zetasizer 300HSA, UK) while microstructure was characterized by scanning electron microscope (SEM; JEOL, JSM-6400).

A commercially available NH_4^+ salt of polymethaacrylic acid (NH_4^+ -PMA; Aron A6114, MW 6000, Toagousei Co., Ltd., Tokyo, Japan) was used as deflocculant to obtain a uniformly dispersed slurry and Na^+ salt of carboxymethylcellulose (Na^+ salt of CMC; Blanose 7M , Hercules Incorporated ,UK) was used as binder. Other specifications of the raw materials from distributors are shown in Appendix B.

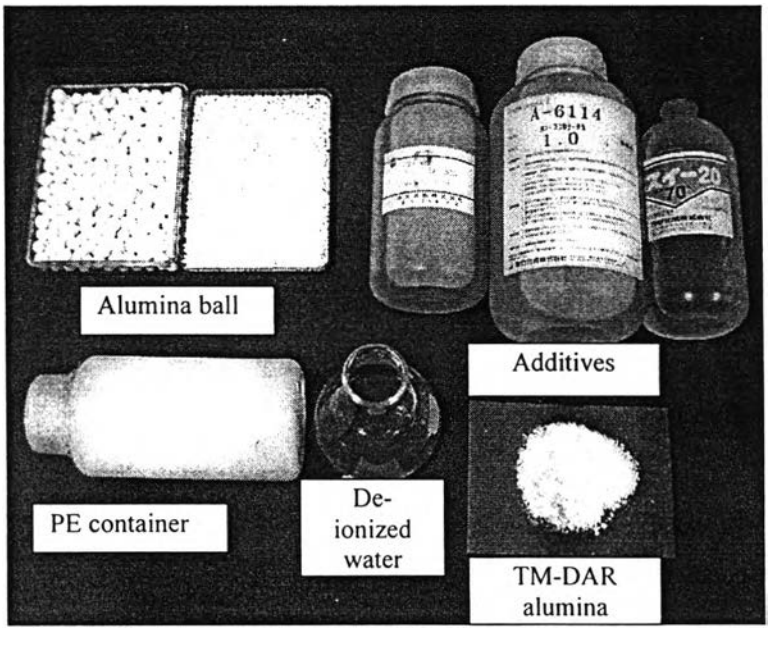

Figure 4.1 Raw materials and equipments for preparing TM-DAR alumina slurry

4.2 Preparation of stabilized slurries with high solid content

For preparation of alumina slurries, alumina powder, deflocculant and de-ionized water shown in Figure 4.1 were mixed for 24 hr in a Polyethylene (PE) container with high-grade alumina balls, as shown in Figure 4.2. The slurries with solid contents of 70, 75 and 80 wt% (corresponding to 37.0, 43.0, 50.1 vol. %) were prepared by varying amounts of deflocculant in order to determine optimum conditions under which the well-dispersed slurries could be obtained. The rheological characteristics of slurries were measured by using a rotational viscometer (Brookfield DV-E, Brookfield Engineering Laboratories, Inc., Massachusetts, U.S.A.).

(a) (b)

Figure 4.2 The mixing process for preparing the alumina slurries by ball-mill (a) mixing process in PE container (b) high purity alumina ball

4.3 Preparation of stabilized slurry adding with binder

The stabilized slurries with solid content of 70 and 75 wt% were added with binder in a range of 0-0.3% in order to enhance green body strength. Their rheologies were also characterized by the rotational viscometer.

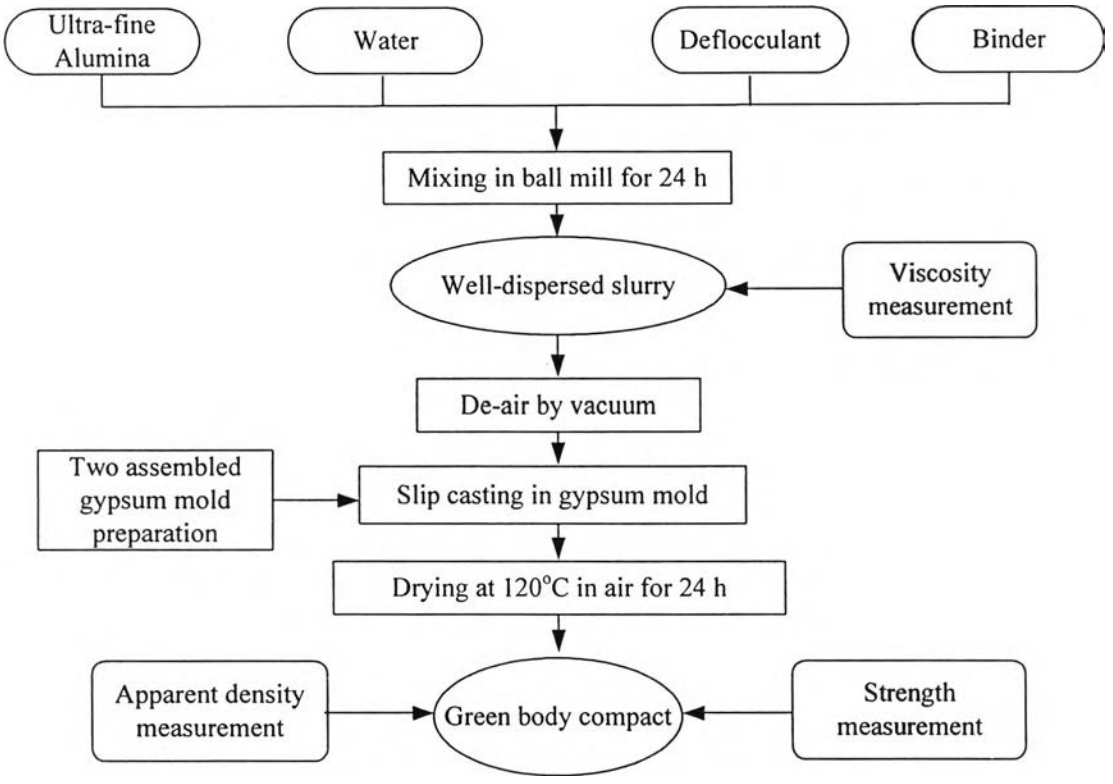

Figure 4.3 Flow chart of green body compact prepared by slip casting process.

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

The slip casting process diagram for preparing of alumina green body is schematically shown in Figure 4.3. The experiment was performed in the range of 70-80 wt% Al_2O_3. They were degassed in vacuum before slip casting in order to eliminate the gas bubbles remains in slurries.

Slip casting were conducted by pouring slurries into the two types of assembled gypsum molds. One type mold is for forming pellet as shown in Figure 4.4. The other is for forming cylindrical rod. The type of mold is depended on purpose of characterization as shown in Table 4.2. Gypsum mold slurry was prepared by gradually adding the gypsum (from Lafarge Prestia Co., Ltd., Thailand) into water in the ratio of 70 : 100. Then, gypsum slurry was mixed by hand for 1 min and shake for 3 min in order to remove the large bubble that remains in slurry. The gypsum slurry was poured into the prototype mold and placed at room temperature for 2-3 h to give

the desired shape of gypsum mold. Before using gypsum mold for slip casing with each casting cycle, it must be dried at 40 ^{o}C to reduce the water content and to enhance the water absorption efficiency in gypsum mold.

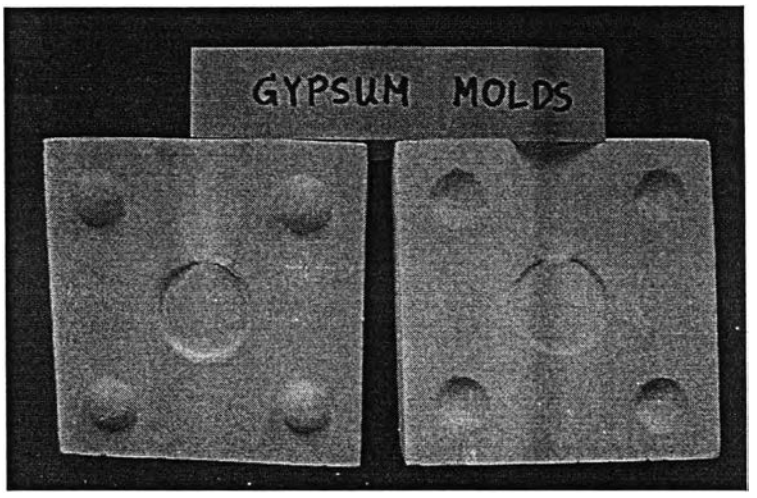

Figure 4.4 Two assembled gypsum mold for preparing alumina green body in pellet shape

After casting, the green compacts were taken out from mold and were dried at 40 ^{o}C for 24 h and then re-dried again at 80 ^{o}C for 6 h and 120 ^{o}C for 24 h, respectively. If it was firstly dried at high temperature, the moisture in the green body was immediately vaporized, resulting in breakage of the green compact.

The dried green bodies were characterized by using the Archemedes' method with mercury as the immersion medium to get its density. The microstructure of fracture surfaces of green bodies was observed using scanning electron microscope (SEM; JEOL, JSM-6400, Japan). The strength of green bodies was evaluated by the three-point bending method with universal testing machine (Instron 5843, Instron Corporation, Canton, MA, USA).

Table 4.1 Dimension of the prepared alumina green body

Type of Figure	Dimension	Purpose of characterization
Pellet	30 mm diameter and 4 mm thickness	● ◇ ■
Cylindrical rod	12 mm diameter and 70 mm length	✿

Remarks:

●	Green body density
✿	Green body strength
◇	Sintered body density
■	Sintered body transmittance

4.5 Post-treatment, calcination, HCl treatment and sintering condition.

Based on the process diagram in Figure 4.3, the obtained green bodies were calcined at 800 ^{o}C for 2 h in air to eliminate the organic content remains in the specimens. Both the heating rate and cooling rates were 10 ^{o} C/min.

In order to investigate the effect of acid treatment on the microstructure and physical properties of sintered body, the calcined bodies were divided into 2 groups. One was not treated with acid, the other was treated.

With acid treatment process, the calcined bodies were submerged in the 1 M HCl for 1h in order to eliminate the Ca^{2+} from gypsum that would penetrate into the green bodies during casting in gypsum mold. Then, the acid in calcined bodies were washed with de-ionized water until no AgCl could be detected (using $AgNO_3$ for HCl detection) and dried. The extinction of element impurities ion can be analyzed by Energy-Dispersive X-ray spectrometer (EDS, ICA 300, Oxford, UK).

The treated and untreated samples were sintered at 1250, 1300 and 1350 ^{o}C for 2h in air atmosphere with the heating and cooling rate of 10 ^{o}C /min. The microstructure of sintered bodies was characterized by scanning electron microscope (SEM). The relative density was characterized by Archemedes' method with water as the immersion medium. The average grain size was determined using Image-Pro Plus version 3 program (Media Cybernetics, Inc., USA). The shrinkage was measured using vernier caliper.

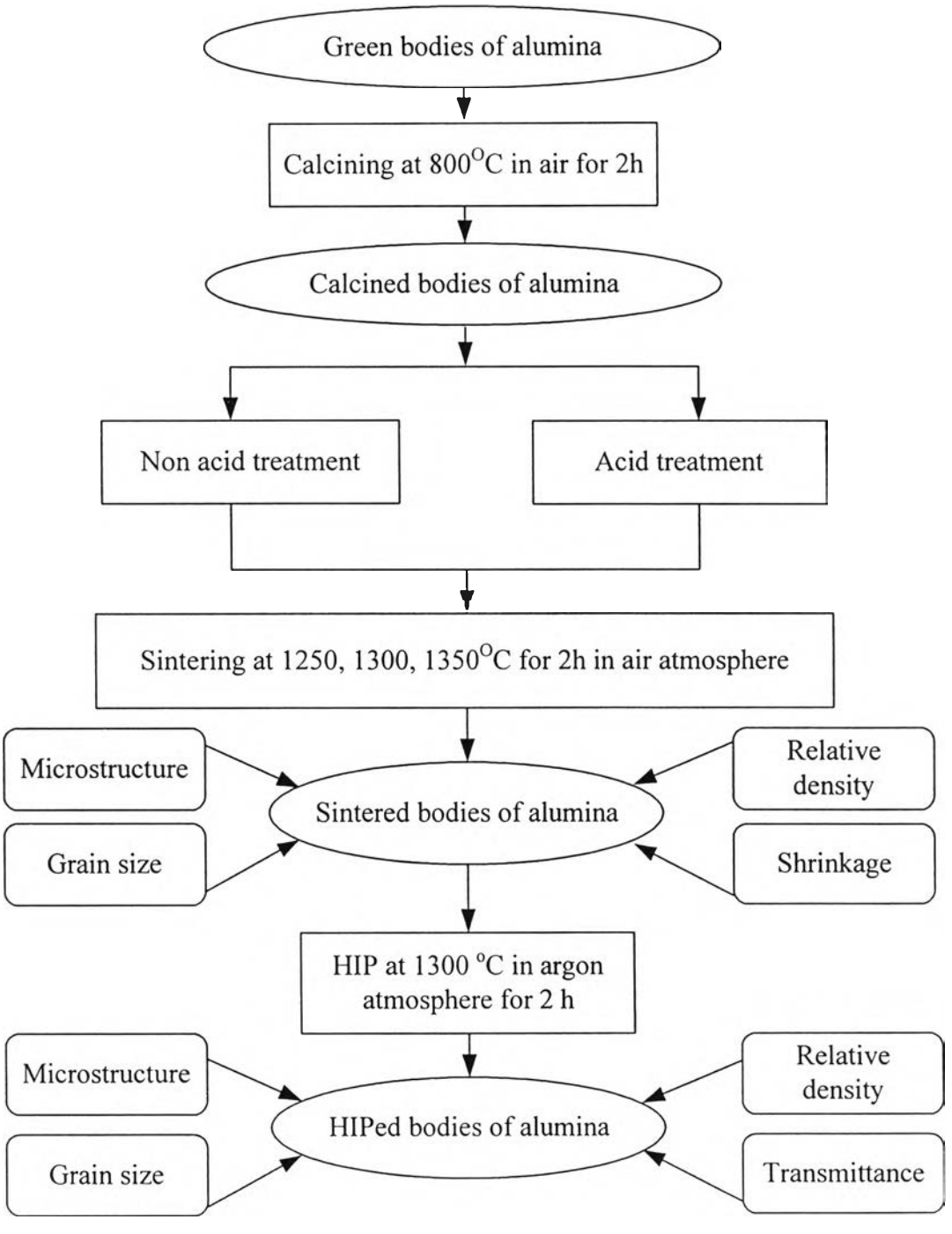

Figure 4.5 Development of optical alumina after slip casting in gypsum mold.

Sintered specimens with higher than 95% of theoretical density were hot isostatic pressed (HIPed) at 1300 ^{O}C, 130 MPa in argon atmosphere in order to give full density. The microstructure (SEM; JEOL, JSM-6301F, JSM 6400, JSL 5410LV, Japan) relative density, grain size and transmission of optical body (UV-VIS Spectrophotometer, UV-1700 Pharmaspec, Shimadzu, Japan) were characterized.