Ceramics International 33 (2007) 739–745
CERAMICS INTERNATIONAL
www.elsevier.com/locate/ceramint

Boehmite modification of nano grade α-alumina and the rheological properties of the modified slurry

Ming-Shyong Tsai [a,*], Fu-Hsu Yung [b], Fu-Han Yang [a]

[a] Department of Chemical and Material Engineering, Southern Taiwan University of Technology, Tainan 710, Taiwan, ROC
[b] Department of Resources Engineering, National Cheng Kung University, Tainan, Taiwan, ROC

Received 29 July 2005; received in revised form 4 November 2005; accepted 6 December 2005
Available online 10 March 2006

Abstract

The boehmite effect on the modification of the surface of a nano grade α-alumina was studied. The pre-aging process was performed by adding boehmite to a nano grade α-alumina (Nα-A) slurry and adjusting the pH value of the mixed slurry to less than 4. After the pre-aging process, boehmite was coated onto the surface of Nα-A particles to change its zero charged point (PZC) from pH = 6–7 to 9–10, similar to that of the boehmite slurry. Mixed and boehmite slurries show thixotropic behavior if the pre-aging process is adjusted to pH = 1. However, there is no thixotropic behavior if the pre-aging process is adjusted to pH = ∼2. Boehmite degrades to a nano grade sol as the pH of the pre-aging process decreases, especially at pH = ∼1. The rheology and the thixotropic properties of the mixed slurry at different pre-aging conditions are discussed in this article.

Keywords: Rheology; Thixotropic; Alumina and boehmite

1. Introduction

Nano size ceramic powders have excellent properties for use in many fields, such as structural, optical, and electrical material, etc. Aluminum oxide is an important ceramic oxide in modern industry because it is inexpensive and possesses good mechanical and optical properties. Alumina slurry settles easily under the action of gravity, which restricts alumina slurry applications. Several patents were registered for alumina and boehmite composites. Wassermann and Meyer [1] discussed a process for making dispersible aluminum hydroxide. Water dispersible aluminum hydroxide is prepared by treating an acid dispersible aluminum hydroxide with 1–9 wt.% of gaseous acid. Meyer et al. [2] discussed a process for producing boehmite alumina. This preparation was carried out by obtaining an alumina suspension from a neutral aluminum alkoxide hydrolysis and then aging the alumina suspension in an autoclave at 1–30 bar at 100–235 °C. Hurlburt and Plummer [3] proposed a method for making boehmite alumina where alumina powder was modified by boehmite with a compound X $(SO_3M)n$ (X is an organic moiety, M is a monovalent cation) at elevated temperature. The rheological properties of the ceramic slurry have been reported in the literature [4–7]. Moreno et al. [4] and Schilling et al. [5] studied the effect of additives on the rheology of alumina slurry. They indicated that surfactants can reduce the viscosity and thixotropic effect of a high solid content slurry. Rao et al. [6,7] studied the suspension properties of SiC slurry by measuring the zeta potential and the sedimentary height of SiC slurry at various pH values. They indicated that pH has a great influence on the zeta potential and slurry suspension properties.

A very simple way to modify Nα-A with boehmite was developed in this study. Nα-A slurry is modified with boehmite by pre-aging at a pH less than 4. The sedimentary heights, the rheological properties and the zeta potential of the slurries are discussed to understand the properties of mixed slurry.

2. Experimental

2.1. Sedimentary height measurement

A 30 wt.% slurry of nano grade α phase alumina (Nα-A) (Design & Quality Enterprise Co., Ltd, Taipei) was the raw material in this study, which was modified with ammonium

* Corresponding author. Tel.: +886 0922739828; fax: +886 288662425741.
E-mail address: tsaims@mail.stut.edu.tw (M.-S. Tsai).

0272-8842/$32.00
doi:10.1016/j.ceramint.2005.12.015

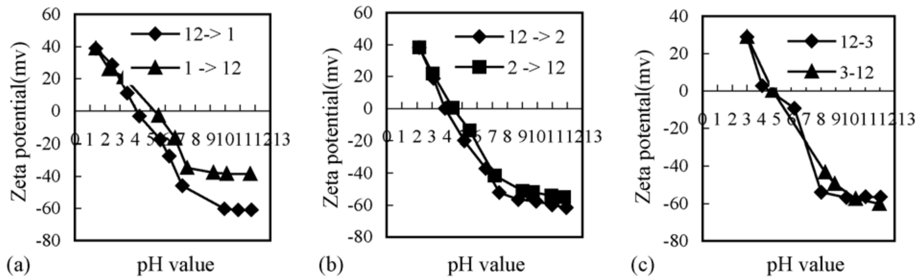

Fig. 1. The zeta potential of pure α-alumina slurry at different pre-aged processes: (a) pH = 12 → 1 → 12, (b) pH = 12 → 2 → 12, and (c) pH = 12 → 3 → 12.

polymath acrylate. The commercial slurry was filtered and subsequently dried in a microwave furnace. Five grams of the dried nano grade α-Al_2O_3 powder was then weighted with 100 g de-ionized (DI) water to form a slurry for the sedimentary height measurement. The mixed powder slurry consisted of 5 g nano grade α-Al_2O_3, 5 g boehmite (Condea, trade name: dispersal) and 100 g DI water. These slurries were divided into 10 parts that were pH adjusted to the desired values from 1 to 10, respectively, and then introduced into cylinders that were placed on a flat, vibration free plate for 2 weeks. The sedimentary heights of the slurries were recorded.

2.2. Zeta potential measurement

The diluted pure Nα-A and pure boehmite slurries were adjusted to pH = ~12 using aqueous potassium hydroxide. The sample was injected into a cell for measuring the zeta potential. The diluted slurry was then titrated by adding diluted hydrogen nitrate continuously to adjust the pH into the acid range. During titration, the diluted slurry was sampled to measure the zeta potential. After titrating to the desired pH, the diluted slurry was reverse titrated using potassium hydroxide to adjust the pH into the basic range. The zeta potential of this diluted slurry was also measured. The point of zero charge (PZC) at each process of pure alumina slurry, pure boehmite slurry and mixed powder slurry, respectively, were identified by these measurements.

2.3. Rheology measurement

The slurries consisting of 5 wt.% of Nα-A, 5 wt.% pure boehmite and 5 wt.% mixed powders were prepared for rheological property measurements. The thixotropic measurement was performed by recording the slurry viscosity at 25 rpm shear rate for 200 s. If the slurry has no thixotropic behavior, the viscosity will not change with time. If the viscosity decreases with increasing time at constant shear rate, the thixotropic effect is present. The slurry rheology was studied at different pH pre-aging processes pH = 1, pH = 1 → 4 (i.e., pH pre-aging at pH = 1 and then adjusting to pH = 4), pH = 1 → 9, pH = 2, pH = 2 → 4, and pH = 2 → 9.

3. Results and discussions

Fig. 1 shows the zeta potential of pure alumina slurry. In general, regardless of the slurry crystalline structure, pure alumina slurry will have a PZC in pH = 9–10. However, the PZC of the commercial Nα-A in this study was at pH = 4–5. This may be due to the addition of ammonium polymath acrylate in the commercial product. The zeta potentials of the particles did not change with the titration process to pH = 12 → 1 → 12, pH = 12 → 2 → 12, and pH = 12 → 3 → 12, as shown in Fig. 1. Fig. 2(a)–(c) show the zeta potential of the boehmite at different titrating processes for the pH values. The PZC of the boehmite slurry in the pH = 12 → 1 → 12 process was at pH = 9–10, which is quite similar to that for the pH = 12 → 2 → 12 and pH = 12 → 3 → 12 process. This means that the surface property of the boehmite did not change with the pH adjustment. The zeta potentials of the mixed slurries are shown in Fig. 3. The PZC of the mixed slurries were at pH = 6–7 in the pH = 12 → pH = 1, 2, 3, and 4 processes, respectively. However, they changed to pH = 9–10 during the pH = 1, 2, 3, and 4 processes, respectively, → pH = 12, as shown in Fig. 3(a)–(d). In the pH = 12 → 5–6 → 12 processes, the PZC of the mixed slurry did not change during the entire process. The PZC of the mixture of Nα-A and boehmite may be at pH = 5–6. As the pH = <4 → 12 was adjusted, the PZC of the mixed slurry in this

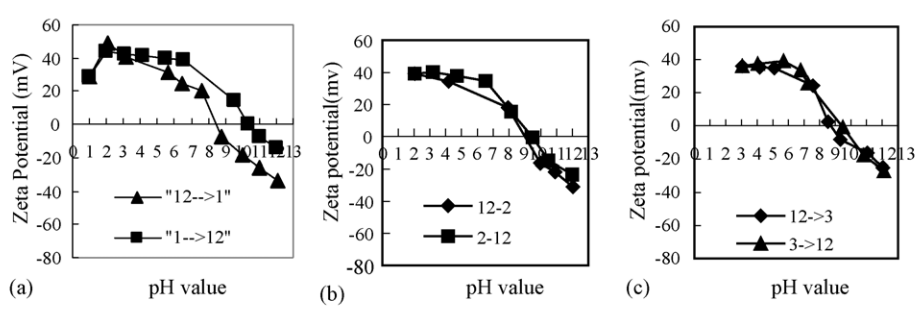

Fig. 2. The zeta potential of pure boehmite slurry at different pre-aged processes: (a) pH = 12 → 1 → 12, (b) pH = 12 → 2 → 12, and (c) pH = 12 → 3 → 12.