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Effects of Mechanical Treatment on the Formation of α-Al_2O_3 from Gibbsite

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Abstract In this study preparation of fine alpha alumina powders derived from Bayer gibbsite was studied. Preparation of the alumina powders was performed by decomposition of the gibbsite into transition alumina phase followed by controlled transformation to the alpha phase. Gibbsite was thermally treated at 350 °C and 900 °C to obtain a transition form of alumina. The purpose of the heat treatment at 350 °C was to increase the surface area of the gibbsite particles and obtain a loosely packed structure that may reduce the size of the rather coarse precursor gibbsite during the grinding step. Mechanical treatment (by using ultrasonic forces and impact forces) was utilized to increase the transformation rate to the alpha alumina in the transition phase matrix and influence the nucleation and growth rate of the solid –solid phase transformation. These powders were calcined at 1100, 1200 and 1450 °C for 1 to 8 hours.
Results indicated that transformation to the alpha phase was accomplished in the powders pre-heated at 900 °C, ultrasonically treated or ground, and then calcined at 1200 °C for 2 to 8 hours or at 1450°C for 2 hours. Ultrasonic treatment accelerated the transformation rate to the alpha phase at 1100°C in 2 hours. Powders that were calcined at 1100 to 1200 °C for 1 hour had a significant kappa content together with the alpha phase. Additionally the powder prepared without mechanical treatment and calcined at 1100 °C was mainly in the kappa phase.

Introduction

The advanced ceramic powders such as aluminum oxides and hydroxides generally require chemical conversion of raw materials into intermediate compounds, which lend themselves to purification and subsequent chemical conversion into the final desired form.In general ceramic materials undergo reconstructive transformation. They transform by nucleation and growth with high activation energies. For the fabrication process it is necessary to convert the ceramic powder to the stable form before consolidation because low densities are usually obtained on sintering if the powder undergoes transformation during heating [1]. Nucleation and growth of these powders that undergo transformation during calcination may be affected by parameters and techniques such as seeding, mechanical treatment, and thermal treatment environment.
Many researchers have attempted to influence the transformation to the alpha alumina by using additives such as α- Al_2O_3, CuO, MgO, α-Fe_2O_3 and α-Cr_2O_3 [1,2,4]. Mechanical pretreatment (compaction or dry ball milling) of transition alumina powders significantly affect the kinetics of the transformation and very high compaction pressures (>2.5GPa) can prevent the formation of a vermicular microstructure characterized by the coexistence of contiguous solid and pore phases [3].
The recent studies generally involve the importance of seeding of the transition alumina phases before transformation to alpha alumina. On the other hand few studies have been reported on the use of mechanical treatment to improve nucleation and growth of alpha alumina. Research on the effect of ultrasonic forces on the nucleation and growth of alpha alumina have not been reported yet.
The aim of this study was to prepare the fine alumina powders starting from Bayer gibbsite (α-aluminum hydroxide) and investigate the effect of grinding and ultrasonic treatment on the transformation to α-alumina.

Experimental

Starting material was gibbsite that was obtained by Bayer process from bauxite used in this work (Seydişehir Aluminum Company, Turkey). Gibbsite was reported to have a chemical composition of 64% Al_2O_3, 0.3% Na_2O, 0.015 % Fe_2O_3, 0.015% SiO_2. 80% of the powder had a particle size > 40 μm .
Alumina powders were prepared from gibbsite by using eight different methods in this work. The processing steps for these methods are shown in Table 1.The purpose of these methods in the processing was to investigate their influence on the nucleation and growth rate of the solid-solid phase transformations. Gibbsite was wet ball milled for 1 to16 hours in the first four methods. Multifix ball mill was used with a plastic container of 4.9 cm radius and cylindrical zirconia grinding media in 9.8 mm height and 9.5 mm diameter. The grinding speed was 115 rpm (85% of the critical speed). The ground gibbsite was further heat treated at 350°C for 4 hours in the first 2 methods. The gibbsite was heat treated at 350°C to create a network of submicroscopic cracks that may help grinding. Gibbsite was directly heat treated at 900 °C for 3.5 hours in methods 5 to 7 (M-5 to M-7). The effect of ball milling and ultrasonic treatment was investigated on the transition to alpha alumina transformation. For the ultrasonic treatment an ultrasonic bath (Elma Transsonic 660/H (35 kHz)) was used.
The phase identification of selected samples was done by X-ray diffractometer (Philips,Expert Pro) using CuKα radiation. Structures were characterized by SEM (Philips, XL30 SFEG). Prepared powders were dry pressed and sintered at 1450°C for 2 hours in a high temperature furnace. The sintered densities of the pellets were determined by Archimedes method.

Table 1. Powder processing methods.

Powder Preparation Methods	**M-1 A**	**M-1 B**	**M-2**	**M-3**	**M-4**	**M-5**	**M-6 A**	**M-6 B**	**M-7 A**	**M-7 B**	**M-7 C**	**M-8**
1st Grinding (Before heat treatment)	●	●	●	●	●							●
2nd Grinding (After heat treatment)	●	●	●				●	●				●
Final Grinding (After calcination)						●			●			●
Ultrasonic Treatment									●	●	●	●
Heat Treat. @ 350 °C 4 hours	●	●	●									●
Heat Treat. @ 900 °C 3.5 hours						●	●	●	●	●	●	●
Calcination @1100 °C 1 to 2 hours			●			●	●		●			●
Calcination @1200 °C 2 hours	●			●	●							
Calcination @1200 °C 8 hours		●						●		●		
Calcination @ 1450°C 2 hours											●	

Results

The SEM micrographs in Fig.1 show that relatively large gibbsite particles have a porous vermicular microstructure with submicron particles. Heat treatment at 350 °C and ball milling prior to calcination at 1450 °C results in a powder with increased porosity and similar particle size (Fig. 1d).

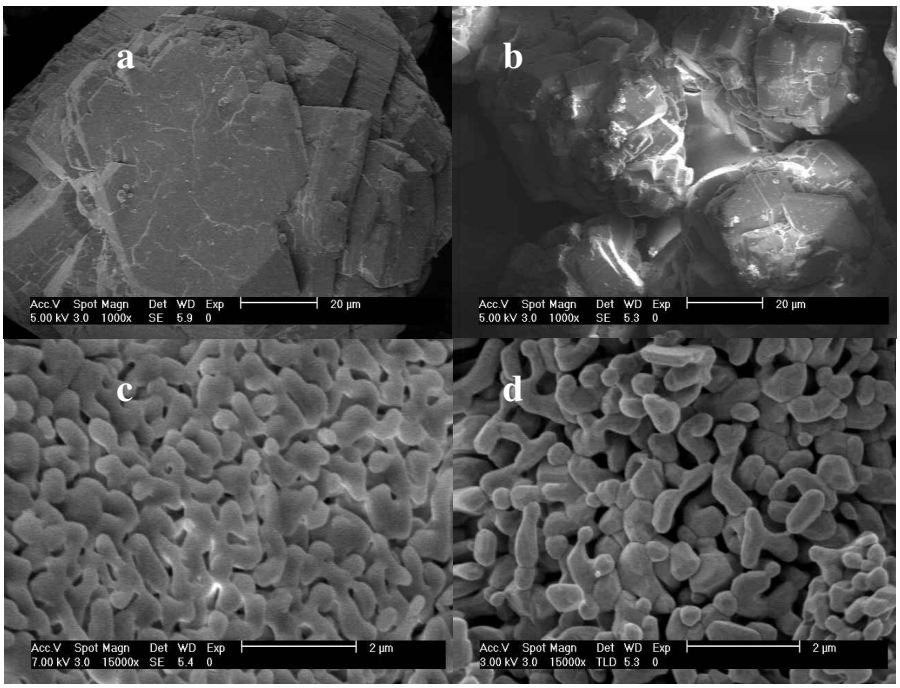

Figure 1.a) Gibbsite after heat treatment at 350 °C. b) Gibbsite without treatment c) Gibbsite, calcined at 1450 °C for 2 hrs. d) Gibbsite ground before calcination at 1450 °C for 2 hrs.

The XRD patterns of the powders prepared by the processing routes showed that almost complete transformation to the alpha phase was obtained for the powder ultrasonically treated before calcination (Fig.2). Calcination at 1100 °C for 2 hrs results in a multiphase structure for the gibbsite powder without treatment. On the other hand, grinding and ultrasonic treatment improved the transformation rate to the alpha phase.

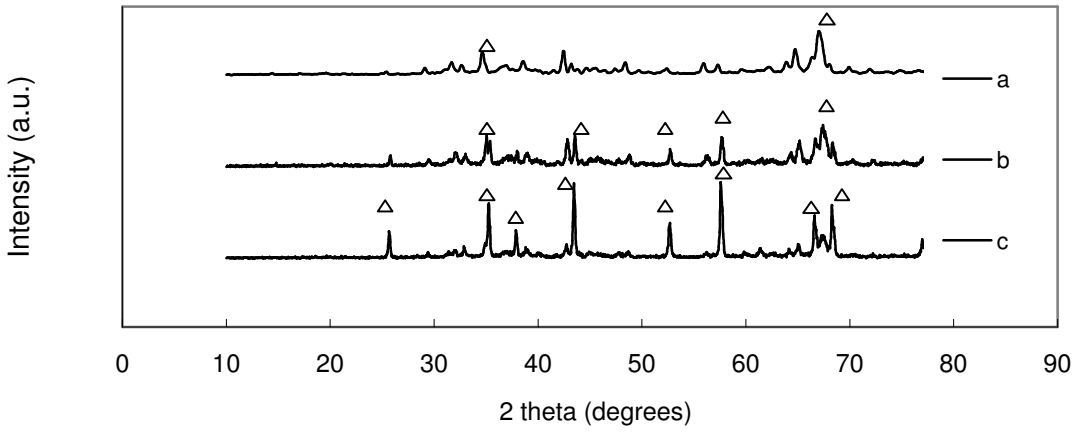

Figure 2. XRD patterns of powders prepared with different processing routes. a)Gibbsite no treatment b) 1 hr grinding before calcination c) 2 hrs ultrasonic treatment before calcination. All calcinations at 1100 °C for 2 hours. (△) Alpha phase

The XRD patterns of the powders M-1B, M-6B and M-7B (all calcined at 1200 °C for 8 hours, M7-B ultrasonically treated) indicated that differences in processing do effect the nucleation of the α phase in the κ phase (Fig. 3). Heat treatment of the gibbsite at 900 °C followed by calcination at 1100°C resulted in the formation of almost pure κ phase. Ball milling (M-6A) and ultrasonic treatment (M-7A) prior to calcination at 1100 °C for 1hr caused partial transformation to the α phase. Complete transformation was obtained at 1200 °C.

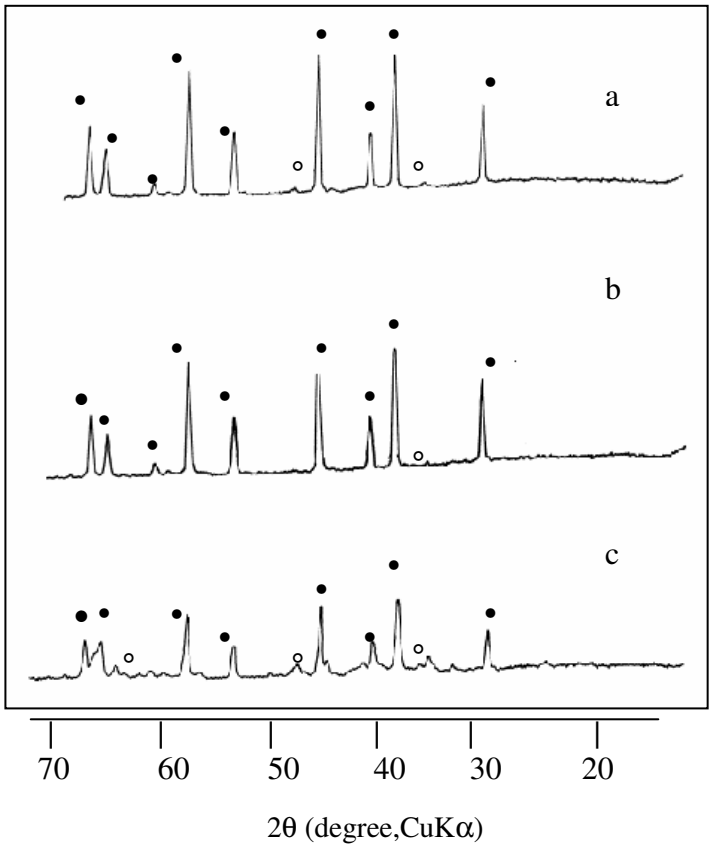

Figure 3. XRD patterns of the powders a) M-7B , b) M-6B, c) M-1B. (•) alpha (°) kappa

Density measurements exhibited that 91% theoretical density was obtained in the powder that was ultrasonically treated before calcination. The highest sintered density was obtained for the powder M-7A as 3.603 g/cm^3 (91% of theoretical density). The powder pellet of M-5 had a rather low density as 3.116 g/cm^3. The ultrasonic treatment that was applied to M-7A was absent in the powder M-5. Effect of ultrasonic treatment on the powders may be attributed to the formation of the micro cracks during the treatment.

Conclusions

The effect of mechanical treatment on the formation of α-alumina was investigated in this work. Results indicated that ultrasonic treatment facilitates the transformation and almost pure α-alumina was obtained from gibbsite at 1100 °C. Grinding and intermediate heat treatments also affect the transformations.

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