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Characterization of porous alumina bodies fabricated by high-temperature evaporation of boric acid with sodium impurity

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ABSTRACT

For steel-making refractories, porous alumina bodies fabricated by the evaporation of chemical impurities at high temperatures was studied. Boron hydroxide and sodium carbonate were added in different proportions as impurities into the starting aluminum hydroxide powder compacts, which were heated at various temperatures for 1 h to form porous alumina bodies. During heating, the borate compounds that reacted with sodium seems to be formed in the liquid phase, so that all alumina particles were able to grow into platelets. Although the sodium inside the compacts was completely evaporated by 1400 °C, the boron melt was maintained above 1400 °C. Hence, alumina platelets grew with a card-house structure, which preserved the high porosity of the alumina body. After heating at 1600 °C for 1 h, the remaining boron oxide was completely evaporated and sintering between the alumina particles began to decrease the porosity. The compressive strength and porosity of the alumina bodies obtained by heating at 1700 °C for 1 h were 0.8 MPa and 64%, respectively. It is expected that the resulting porous alumina bodies can be used as castable porous clinkers.

1. Introduction

Porous materials are expected to find a growing number of applications in modern-day refractories. The main motivation behind their use is to reduce the energy cost of the production of metals, such as steel, copper, and aluminum alloys. Porous materials have a large insulating effect, which decreases the energy consumption [1–5]. For instance, in steel-making refractories, castables composed of alumina clinker or other stable oxide aggregates (or powder) and alumina cement binder are used [6–15]. It is well known that when these alumina clinker and/or aggregates have high porosity, the insulating performance of the castable body improves. Conventional methods for fabricating porous alumina have typically involved low-temperature annealing or the addition of pore formers [16–23].

In order to fabricate a porous body with high porosity, alumina platelets were useful as starting alumina particles. A porous alumina body fabricated from platelets tends to have a card-house structure, which facilitates the fabrication of the porous body. When alumina platelets were used, the resulting porous alumina bodies had high porosity, but also low compressive strength. Hence, chemical solution immersion followed by reheating treatment has been conducted [24–26]. Thus, castable clinkers with high porosity and sufficient mechanical strength could be obtained. Furthermore, to facilitate the fabrication of a card-house structure using platelets, yeast particles several microns in size were added into starting alumina platelets as a pore former [27]. However, since anisotropic alumina platelets are too expensive as starting material for refractory applications, another fabrication method for porous alumina bodies (clinkers) that does not involve the use of platelets is desirable.

As a simple, novel method for fabricating porous alumina bodies, high-temperature evaporation reactions involving borate compounds and sodium have been attempted. The incorporation of the evaporation of chemical impurities into the formation of porous bodies at high temperature has hitherto not been studied. More specifically, both boron hydroxide and sodium carbonate were added into alumina hydroxide as raw materials to obtain porous alumina bodies after a single-step anneal. Variations in the porosity and compressive strength of the resulting porous alumina bodies with the starting composition and heat treatment temperature were studied. The micromorphology of the porous alumina bodies was observed by scanning electron microscopy. Finally, the formation of the porous structure of the alumina bodies was considered based on the formation of the liquid phase consisting of sodium and an aluminum borate compound and based on the chemical thermodynamics during heating.

2. Experimental

As starting materials, all reagent grade chemical compounds, *viz.*,

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Table 1
Chemical compositions of the starting powder mixtures used.

$Al(OH)_3$	100					
$B(OH)_3$	–	0.5	1.0	2.0	5.0	10.0
$NaCO_3$	–	0.2	0.4	0.9	2.1	4.3
Graphite	5.0					
Water	5.0					

aluminum hydroxide ($Al(OH)_3$: Nippon Light Metal Company, Ltd., Japan; SB93), boron hydroxide ($B(OH)_3$: KCM Corporation Co., Ltd., Japan) and sodium carbonate ($NaCO_3$: Tokuyama Corporation, Co., Ltd., Japan) were used. $B(OH)_3$ and Na_2CO_3 were added simultaneously into the starting $Al(OH)_3$ powder at various weights with a fixed atomic ratio of B:Na = 2:1. Therefore, the starting sample was named according to the weight ratio of $B(OH)_3$. In addition, for ease of release of the mixed powder compact from the steel mold after compaction, a small amount of C powder (scaly graphite: Kojundo Chemical Laboratory Co., Ltd., Japan) was added into the starting powder mixture as a solid lubricant. Table 1 tabulates the chemical compositions of the starting powder mixtures used in this study. First, each powder mixture was pressed at 100 MPa inside a steel mold using a uniaxial press. The resulting cylindrical compacts (25 mm in diameter, 50 mm in height) were heated at various temperatures for 1 h using a heating rate of 100 °C/h. The apparent densities of the final porous alumina bodies were measured using the Archimedes method. As the intrinsic density of alumina, 3.98 g/cm^3 was used. The apparent porosities of the compact bodies were calculated using the apparent density and the intrinsic density.

The variation in the reaction products of compact bodies having different compositions with the heat treatment temperature was analyzed using a powder XRD analysis device (Bruker; D2PHASER). In addition, changes in the microstructure of the compact bodies after heating at various temperatures were investigated by scanning electron microscopy (KEYENCE Corporation, Co., Ltd.; VE8800). The compressive strengths of the compact bodies were measured using a material testing device (SHIMADZU Corporation, Co., Ltd., Japan; Autograph AG-Xplus). Three to five samples were tested under each set of test conditions. Finally, in order to determine the composition of the liquid phase formed inside the compact body during heating, the sodium content of each compact body after heating at various temperatures was measured using an XRF analyzer (Rigaku Corporation, Co. Ltd., Japan; Simultix14).

3. Results and discussion

3.1. Characteristics of porous alumina bodies

3.1.1. Porosity and compressive strength

Fig. 1 shows the variation in the average compressive strength of the porous alumina bodies with the amount of $B(OH)_3$ additive after heating at 1700 °C for 1 h. Without $B(OH)_3$, the average compressive strength of the porous alumina bodies was 3.1 MPa. As the amount of $B(OH)_3$ increased, the average compressive strength of the alumina bodies decreased, dropping to 0.85 MPa at a $B(OH)_3$ amount of 5 mass %. When a porous alumina body was fabricated with 10 mass% $B(OH)_3$, the average compressive strength obtained was 0.90 MPa. The marked decrease in average compressive strength in going from 0 to 5 mass% $B(OH)_3$ was thought to be caused by an increase in the porosity of the alumina bodies. Thus, the porosities of the alumina bodies were measured. Fig. 2 shows the variation in the apparent porosity of the porous alumina bodies with the amount of $B(OH)_3$ additive after heating at 1700 °C for 1 h. With no $B(OH)_3$, the apparent porosity of the alumina body was 44.8%. With 0.5 mass% $B(OH)_3$, the average apparent porosity increased to 62.0%. With further increase in the amount of $B(OH)_3$, the apparent porosity increased slightly. When 5 mass% and 10 mass% $B(OH)_3$ were used, the apparent porosities of the resulting samples were almost the same: 62.5% and 62.2%, respectively. Therefore, it was decided that 5 mass% $B(OH)_3$ would be used in subsequent experiments.

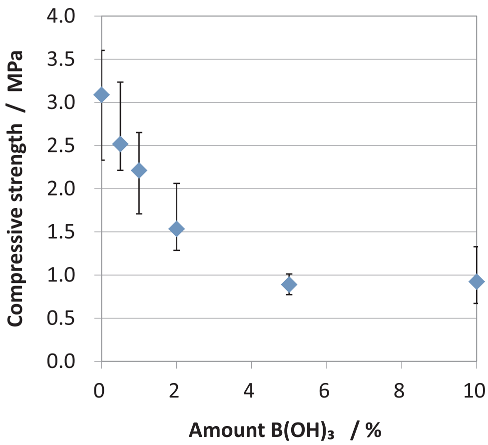

Fig. 1. Variation in compressive strength of porous alumina bodies with amount of $B(OH)_3$ additive after heating at 1700 °C for 1 h.

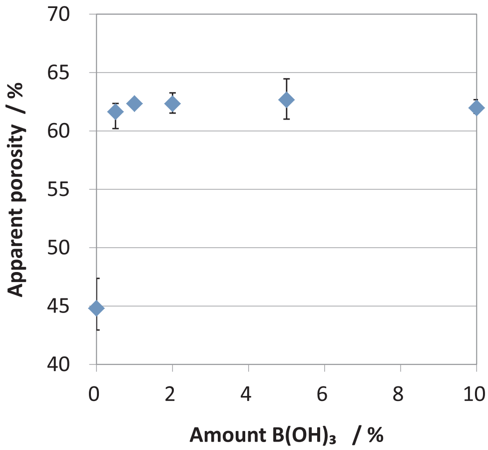

Fig. 2. Variation in apparent porosity of porous alumina bodies with amount of $B(OH)_3$ additive after heating at 1700 °C for 1 h.

Fig. 3 shows the compressive strength of porous alumina bodies with/without 5 mass% $B(OH)_3$ as a function of heat treatment temperature. The compressive strength of the porous samples without B

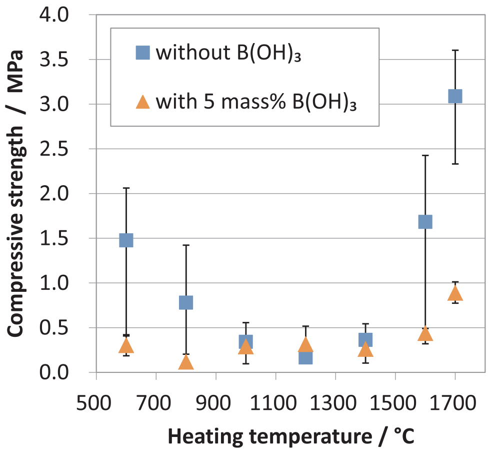

Fig. 3. Compressive strength of porous alumina bodies with and without 5 mass% $B(OH)_3$ as a function of heating temperature.