Sintering Behaviour and Microstructure Development of Indian Bauxite

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Abstract

Two varieties of Indian bauxite with different impurities were used to study their sintering behaviour and phase development with sintering temperature starting from 1000-1650^{0}C. There phase development and microstructure characterisation were done by X-ray diffraction and Scanning electron microscopy. It was found that both the sintered samples not only contains large amount of vitreous phase, but also contains considerable amount of low melting $FeAlTiO_5$ phase. Presence of vitreous phase and low melting $FeAlTiO_5$ phase is primarily responsible for inferior high temperature properties of bauxite. Bauxite samples exhibit a RUL of 1450^{0}C. Prevention of low melting phases can increase the RUL to >1600^{0}C.

1. Introduction

India has the fifth largest reserves of bauxite in the world (about 5%). Indian bauxite contains lot of impurities. Major impurities present in Indian bauxites are SiO_2, Fe_2O_3, TiO_2 & CaO. These impurities form low melting phases at higher temperature which impairs the high temperature properties. China, Guinea and Brazil are the major international suppliers of bauxite for refractory applications.

Good quality refractory grade bauxite potentially should have high refractoriness (~1840^{0}C) as can be interpreted from the eutectic point in Al_2O_3-SiO_2 phase diagram which depict the two main constituents in their pure form. However, the material under consideration has a low refractoriness under load. This behavior is normally attributed to the presence of a liquid phase in this temperature range due to the presence of impurities like TiO_2 and Fe_2O_3 in the bauxite.

In this investigation two varieties of Indian bauxite with different chemical composition were selected to study their densification behaviour. Attempt has also been made to study the compositional effect on the microstructure of the sintered product. Finally, refractoriness under load was measured and the results are correlated with the microsturcture.

2. Experimental Procedure

Two varieties of bauxite with different impurity levels collected from Gujarat region of India. Chemical analyses of these materials were done by standard wet chemical method. Bauxite ores were crushed and dry milled to pass through 100 mesh sieve followed by attrition milling in a zirconia pot with zirconia ball in ethanol medium for 2 h. The slurry thus obtained was dried at 40°C for 24 hrs and passed through 100 mesh sieve to get the desired powder.

Powders thus obtained were mixed with 5% (W/V) PVA solution as binder and uniaxially pressed under 100 MPa pressure into pellets of 2.5 cm dia and 1 cm height. These pellets were then dried at 110±5°C and sintered in the temperature range of 1500-1650^{0}C. The heat treatment was performed in a programmer-controlled electric furnace. Heating rate was maintained at 5°C/min upto 1000^{0}C and then 3^{0}C/min upto the final temperature with a 2 h soaking at peak temperature. Sintered specimens were characterized in terms of bulk density, apparent porosity, phase assemblage and microstructure. The bulk density and apparent porosity of the sintered samples were measured by water displacement method using Archimedes' principle.

Phase identification was done by the X-ray diffraction technique. The diffraction patterns of the finely powdered samples were obtained in a X-ray diffractometer using nickel filtered Cu-Kα radiation and recorded over a Braggs' angle (2θ) range of 10–70°. Microstructure evaluation of the sintered compacts was done by scanning electron microscopy using sputtered gold coating on the polished surface after thermal etching. Elemental analysis of the individual phases was done by EDX using carbon coated sintered polished samples.

3. Results and discussion

The chemical analysis (loss free basis) of two varieties of bauxites is shown in **Table 1**. Chemical composition indicates that, they are impure in nature and there is a wide difference in chemical composition between them. Bauxite-1 is relatively purer with Al_2O_3 content of 84.58% compared to that of bauxite-2 is 79.53%. TiO_2 content of both the bauxites was nearly same. However, the main impurity Fe_2O_3 in bauxite-2 is as high as 6.90% compared to 5.01% in bauxite-1.

CaO content (2.44%) is also significantly higher in bauxite-2, compared to that of bauxite-1 which is 0.81%.

Table 1 Chemical analysis of raw bauxites (loss free basis)

Constituents, wt%	Bauxite-1	Bauxite-2
Al_2O_3	84.58	79.53
SiO_2	5.08	6.54
TIO_2	4.52	4.58
Fe_2O_3	5.01	6.90
CaO	0.81	2.44

X-ray diffraction patterns of both the bauxites are similar as can be seen in **Fig. 1**. Phase analysis study indicates that the major crystalline phase is gibbsite with a small amount of boehmite. Both the bauxites used in this study are gibbsitic in nature.

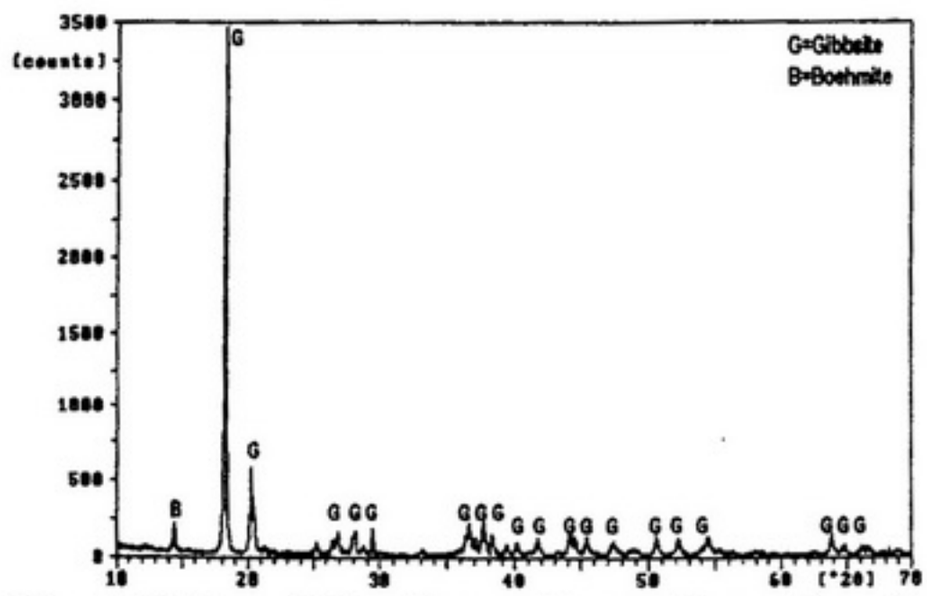

Fig. 1 X-Ray diffraction pattern of raw bauxite indicating gibbsite is the main crystalline phase

Densification:

Densification behaviour of both the bauxites is shown in **Figs. 2(a)-(b)**. In case of bauxite-1 (Fig. 2a), with increase of temperature above 1300°C, the bulk density increases and apparent porosity decreases up to 1500°C, after that it falls slightly up to 1650°C. Apparent porosity is more or less constant above 1550°C. This may be due to the formation of the vitreous phases at higher temperature. The lower specific gravity at 1300°C may be due to formation of various unstable phases of alumina, having lower specific gravity that that of stable corundum phase. From 1300°C it increases continuously upto 150°C due to more and more conversion of different alumina phases into corundum. In case of bauxite-2, the bulk density increases with increase in firing temperature accompanied by fall in apparent porosity (Fig. 2b). The change in the specific gravity of the samples follows the same trend as in case of bauxite-1.

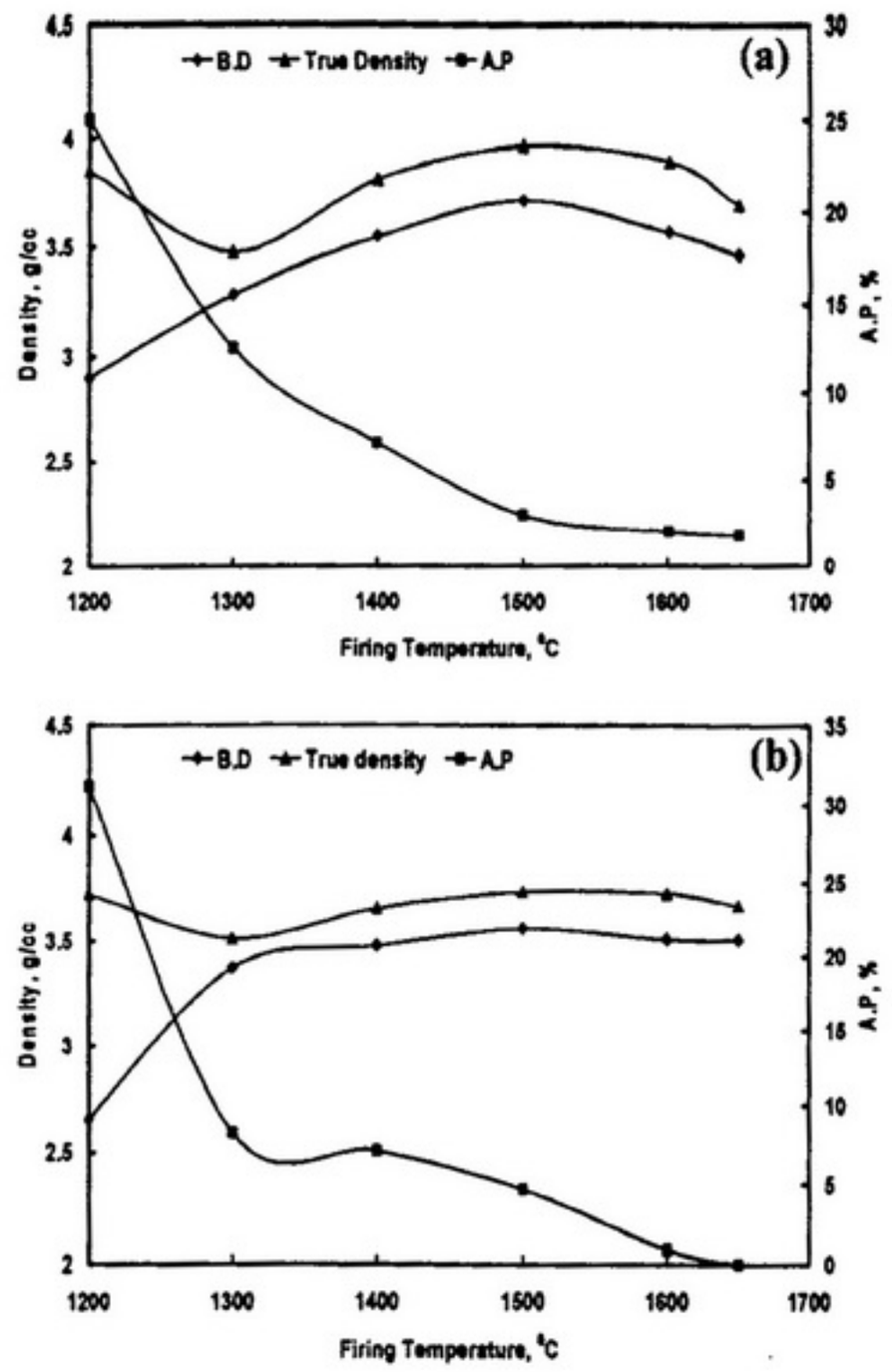

Fig. 2 Variations of physical properties with sintering temperature: (a) bauxite-1 and (b) bauxite-2

The highest bulk density achieved by bauxite-1 samples is always higher compared to that of bauxite-2. True density is also higher in case of bauxite-1 sample. Since bauxite-2 samples contain higher amount of impurities, particularly CaO which favours the low melting phase formation at higher temperature, the vitreous phase content is more in bauxite-2. Higher amount of low density vitreous phase is responsible for the lower bulk density and true density of bauxite-2 samples compared to that of bauxite-1.

Microstructure:

Electron microscopic observation indicates, both the bauxite samples sintered at lower temperature are porous. Scanning electron photomicrographs of the bauxite samples sintered at 1500°C is shown in **Fig. 3(a) – (b)**. The main phase is corundum and corundum grains are acicular in nature. Glassy phase evenly distributed with the corundum matrix. Some white colour phase is noticed in both the samples irrespective of the firing temperature. An EDX spectrum of this phase is shown in **Fig. 4**.

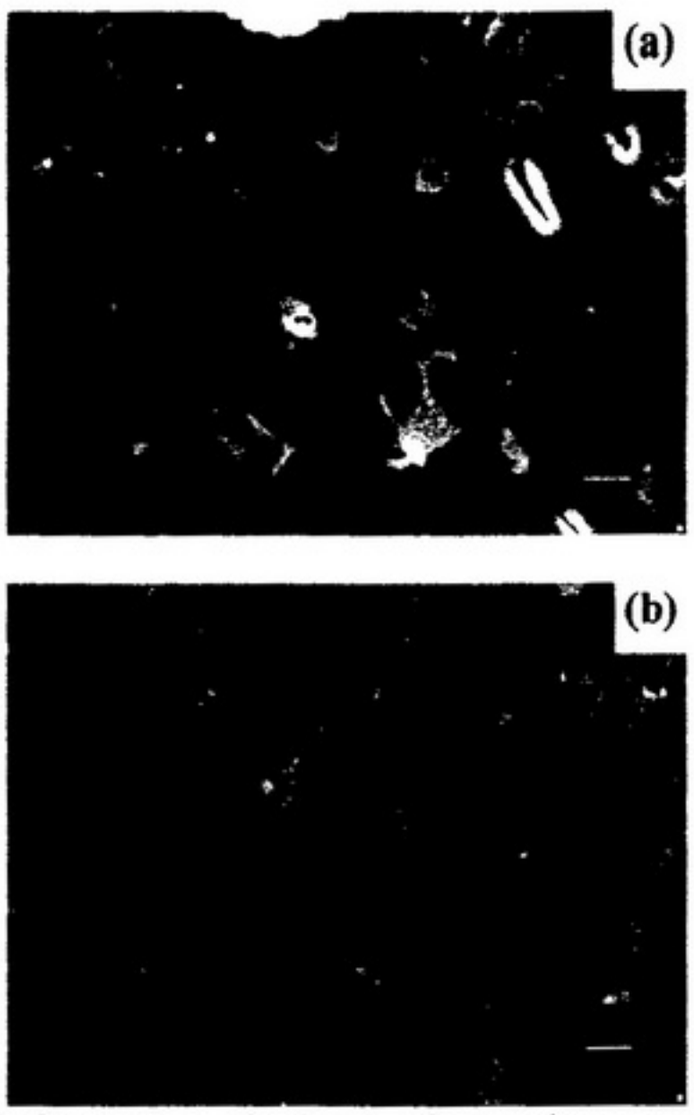

Fig. 3 Scanning electron photomicrographs of bauxite samples sintered at 1500°C: (a) bauxite-1 and (b) bauxite-2

The quantitative analysis indicates this phase is $FeAlTiO_5$. XRD pattern of both the sintered samples indicates the presence of corundum as the main crystalline phase with $FeAlTiO_5$ as the minor phase. Microstructure of samples sintered at higher temperature is more heterogeneous with higher amount of vitreous and $FeAlTiO_5$ phases

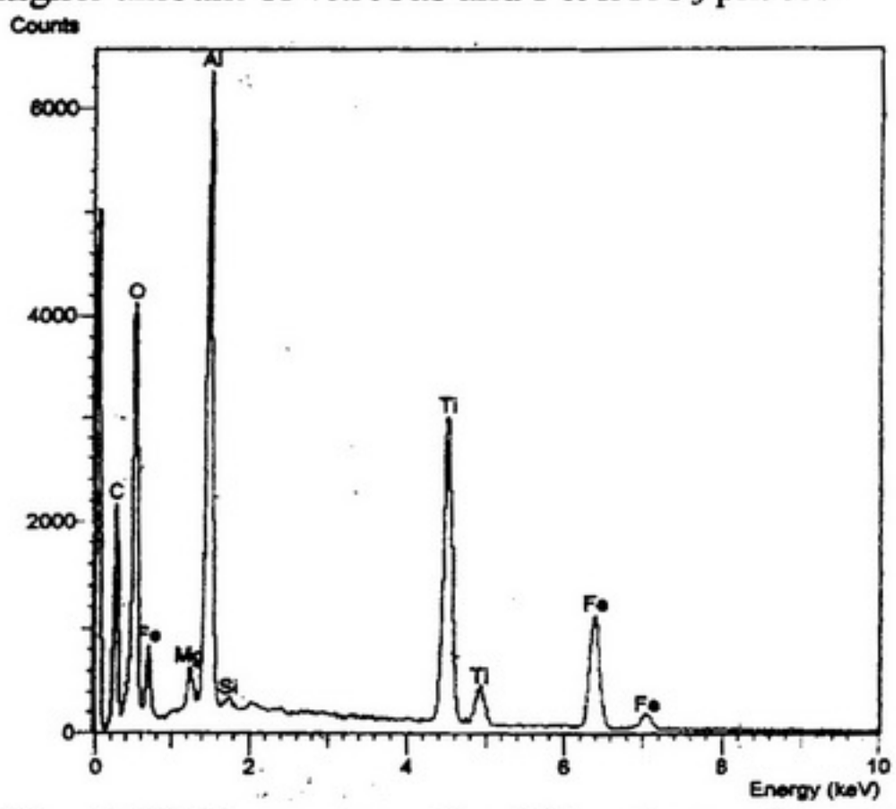

Fig. 4 EDX spectra of white phase observed under electron microscope of sintered bauxite sample as shown in Fig. 3.

Refractoriness under load

Refractoriness under load measured from sintered bauxite-1 and bauxite-2 are in the range of 1400-1450°C. The presence of low melting $FeAlTiO_5$ and vitreous phase is responsible for the low RUL values of both the bauxite samples. Since TiO_2 and Fe_2O_3 can enter into the mullite structure by solid solution, mullite formation from bauxite reduces the amount of $FeAlTiO_5$ phase formation in the sintered samples. Mullite aggregate developed from bauxite-1 with relatively lower amount of CaO exhibit a RUL value of 1550-1600°C.

4 Conclusions

Densification behaviour and microstructure development of Indian bauxite is very much dependent on the chemical composition. Two varieties of Indian bauxite used in this investigation are gibbsitic in nature. Bauxite-1 with lower impurities always exhibit higher bulk density and true density that that of bauxite-2. Both the sintered bauxite contains corundum as the main crystalline phase with considerable amount of vitreous phase and low melting $FeAlTiO_5$. All these phases reduce the high temperature properties of bauxite. The RUL of these bauxites are in the range of 1400-1450°C. Bauxite-1 exhibit relatively higher RUL values compared to bauxite-2. It was observed that prevention of $FeAlTiO_5$ phase improves this RUL to 1550°C-1600°C.

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