PREPARATION AND PROPERTIES OF POROUS ALUMINA CERAMIC PRODUCED BY AN EXTRUSION PROCESS

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Abstract

Porous alumina ceramic was fabricated by an extrusion method with the addition of titania as a sintering additive up to 20 wt%. Alumina powders were mixed and kneaded with titania and organic additives including binder (carboxy methyl cellulose, 7 wt% based on dry weight of alumina and titania; all wt% mentioned in this study based on the amount of inorganic powders excluding water), co-binder (polyvinyl alcohol, 1 wt%), plasticizer (polyethylene glycol, 0.5 wt%), lubricant (glycerin oil, 1 wt%), and solvent (water, 23 wt%). The resulting dough was extruded, dried at room temperature for 48 hours, and sintered at 1200°C for 1 hour in an electrical box furnace. The phase composition, microstructure, mechanical strength, and porosity of the sintered porous bodies were also investigated. With 5 and 10 wt% of TiO_2, the bending strength of the sintered specimens is higher than 18MPa with porosity of more than 40%.

Keywords : Alumina, titania, porous ceramic, extrusion

Introduction

Porous ceramics are widely used as filters, catalyst carriers, separation membranes, and bio-ceramics. There have been many studies focused on the preparation of porous ceramics. Generally, porous ceramics have good properties such as high mechanical strength, abrasion resistance, and chemical and thermal stability (Isobe *et al*., 2006). Furthermore, permeability is one of the most important properties of porous ceramics for filters because this property directly relates to the pressure drop during filtration (Isobe *et al*., 2007). Thus, the development of porous ceramics requires sufficient mechanical and chemical stability as well as permeability.

The main raw materials of commercial porous ceramics are alumina, zirconia, titania, silica, and mullite (Dong *et al*., 2006; Jedidi

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et al., 2009). Alumina is the most widely used of the oxide ceramics because of its hardness, good corrosion resistance, high thermal resistance, and ease of processing (Sathiyakumar and Gnanam, 2003).

In order to promote ceramic sintering kinetics or reduce the sintering temperature, there are 2 techniques which may be applied, using superfine powders and using sintering additives (Wang *et al*., 2007). The additives, such as MgO, TiO_2, ZrO_2, Y_2O_3, and La_2O_3, have an influence on the sinterability, microstructure, and mechanical properties of alumina (Sathiyakumar and Gnanam, 2003). Normally, the solid-state reaction between Al_2O_3 and TiO_2 above the eutectoid temperature 1280°C can form aluminum titanate. Aluminum titanate in the Al_2O_3/TiO_2 system increases the concentration of vacancies and the transfer rate of mass (Park *et al*., 2003; Qi *et al*., 2010).

Porous ceramics can be prepared by several methods such as slip casting, centrifugal casting, extrusion, and isostatic pressing. Among these methods, an extrusion process has an advantage in the production of rods, tubes, and honeycombs. In this research, porous alumina ceramics consisting of alumina and titania were prepared by the extrusion method using alumina dough containing organic additives including binders (sodium carboxy methyl cellulose), co-binder (polyvinyl alcohol), plasticizer (polyethylene glycol), lubricant (glycerin oil), and solvent (distilled water). The phase composition, microstructure, mechanical strength, density, porosity, and pore size of the sintered porous bodies were also investigated.

Materials and Methods

Raw Materials and Sample Preparation

Commercially available Al_2O_3 (AL-05, Hindalco Industries Ltd., Mumbai, India) and TiO_2 (R818, China) powders were used as starting raw materials in this work. According to the data from the suppliers, the average particle size of the Al_2O_3 and TiO_2 was 5μm and 0.3μm, respectively.

1 wt% of polyvinyl alcohol (PVA) was dissolved in 23 wt% of distilled water (based on dry weight of the alumina and titania) by using a magnetic stirrer at 80°C for 1 h. The alumina powders were first mixed with 0, 5, 10, 15, and 20 wt% of TiO_2 powders. Subsequently, the powders were then mixed and kneaded with 7 wt% of sodium carboxy methyl cellulose (CMC), 0.5 wt% of polyethylene glycol (PEG), 1wt% of glycerin oil, and the solution of polyvinyl alcohol. The binder composition was the same as that used in a previous study (Gosuphan, 2009). The dough was extruded to form the samples with a 10 mm diameter and 120 mm length by the hydraulic extrusion pushing type. The extruded green bodies were dried at room temperature for 48 h and 110°C for 24 h. The dried samples were then sintered at 1200°C for 1 h in an electrical box furnace with a heating rate of 5°C/min. Five kinds of samples with different compositions are referred as A_1 (Al_2O_3: TiO_2 = 100:0), A_2 (Al_2O_3: TiO_2 = 95:5), A_3 (Al_2O_3: TiO_2 = 90:10), A_4 (Al_2O_3: TiO_2 = 85:15), and A_5 (Al_2O_3: TiO_2 = 80:20). The flow chart of sample preparation is shown in Figure 1.

Characterization

The density and porosity of the sintered porous bodies were determined by the Archimedes technique using water as an immersion medium (Isobe *et al*., 2006). The water absorption of the samples was also measured. The linear shrinkage measurement of the samples before and after sintering was determined by a *vernier caliper*. The mechanical strength of the sintered samples was measured by a 3-point bending test. The sample with a diameter of 10 mm and length of 120 mm was measured with a span length of 80 mm. The phase compositions were identified using X-ray diffractometry (Bruker-AXS, Cu-K_α radiation (λ = 0.154 nm) Bruker Corp., Billerica, MA, USA,). The microstructure of the sintered bodies was observed using a scanning electron microscope (JSM-6480LV, JEOL Ltd., Tokyo, Japan).

Results and Discussion

Table 1 shows the data of bulk density, linear shrinkage, water absorption, porosity, and 3-point bending strength of the specimens fired at 1200°C for 1 h When the amount of titania was increased, the bulk density and linear shrinkage of the samples increased; however, the water absorption decreased. Moreover, the porosity of the samples decreased from 48.63 to 39.94% with increasing the amount of titania from 0 wt% to 20 wt%, respectively. Titania is the sintering aid which increases bulk density and decreases porosity. Thus, doping titania into porous alumina leads to a better sintering behavior (Qi *et al*., 2010).

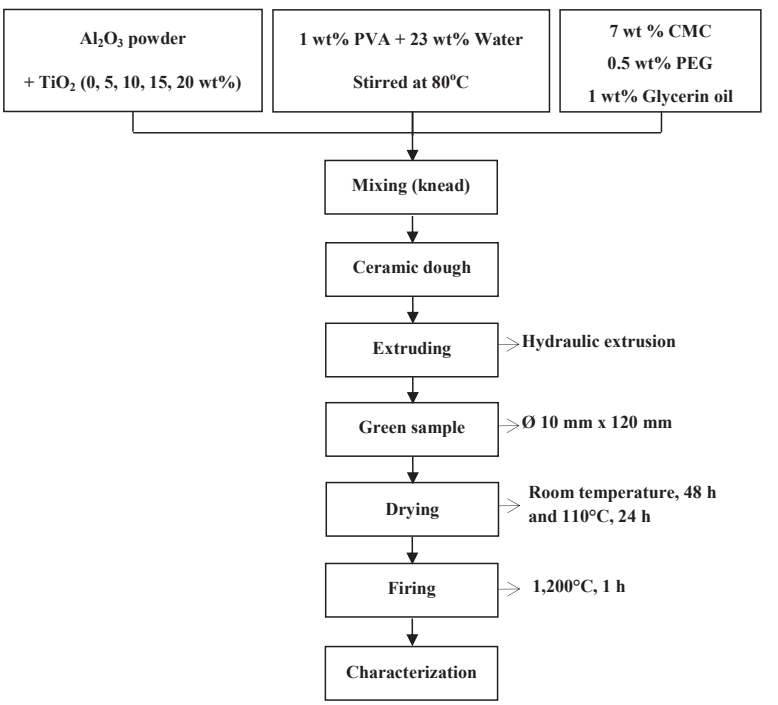

Figure 1. Flow chart for the preparation of porous alumina ceramics

Table 1. Properties of obtained porous alumina ceramics sintered at 1200°C

Sample	Bulk density (g/cm^3)	Linear shrinkage (%)	Water absorption (%)	Open porosity (%)	Three-point bending strength	
					(MPa)	SD.
A_1	1.98	-	24.51	48.63	4.45	3.72
A_2	2.16	2.85	20.71	44.83	23.22	2.46
A_3	2.25	3.86	18.93	42.57	18.11	2.54
A_4	2.28	4.88	18.24	41.56	20.04	3.79
A_5	2.32	5.67	17.19	39.94	23.73	3.49

The strength of the sintered samples tends to increase with increasing the titania addition. With 5, 10, and 15wt% of TiO_2 (A_2, A_3, and A_4), the bending strength of the sintered specimens is higher than 18 MPa with porosity of more than 40%. The maximum strength of 23.73 MPa is obtained for A5 (Al_2O_3: TiO_2 = 80:20). The properties of the sintered samples are shown in Table 1. However, the strength of A_2-A_5 is slightly changed because of the low sintering temperature. Thus, the sintered sample

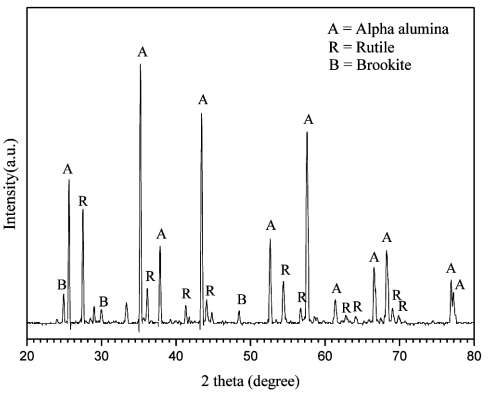

Figure 2. XRD patterns of porous alumina ceramic A_5(Al_2O_3: TiO_2 = 80:20) sintered at 1,200°C (A; alpha alumina, R; rutile, B; brookite)

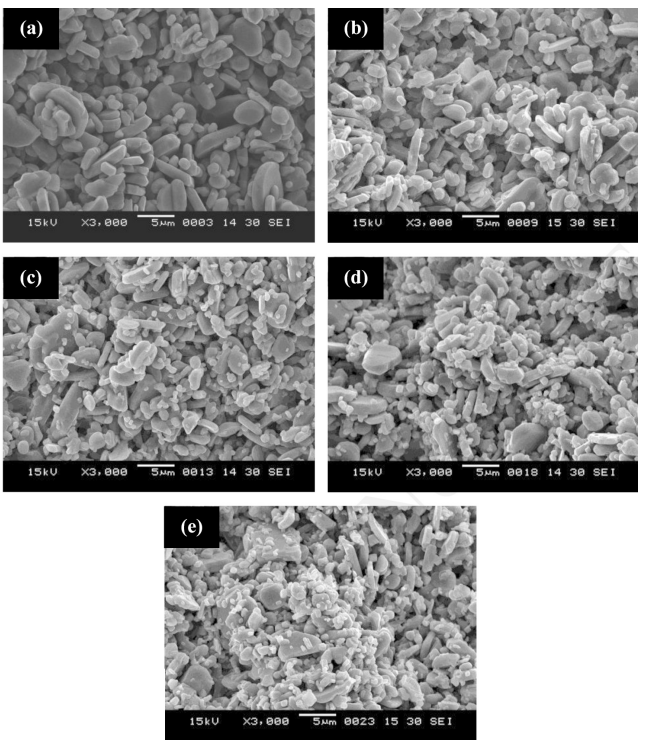

Figure 3. SEM micrographs of porous alumina ceramics sintered at 1200°C; (a) A_1(Al_2O_3: TiO_2 = 100:0), (b) A_2(Al_2O_3: TiO_2 = 95:5), (c) A_3(Al_2O_3: TiO_2 = 90:10), (d) A_4(Al_2O_3: TiO_2 = 85:15), (e) A_5(Al_2O_3: TiO_2 = 80:20)

obtained low density. Kritikaki and Tsetsekou (2009) reported that porous alumina ceramic has a mean bending strength of 17 MPa and a porosity of 41%. The porous alumina ceramics were prepared by the extrusion method using pure α-Al_2O_3 powders with an average particle size of 5μm and were then sintered at 1500°C. The bending strengths of A_2-A_5 are higher than that of reported by Kritikaki and Tsetsekou (2009).

The phase composition of the porous alumina ceramic A_5 (Al_2O_3: TiO_2 = 80:20) sintered at 1200°C is shown in Figure 2. It consists of α-Al_2O_3 and TiO_2 (rutile phase) corresponding to Qi *et al*., (2010). In their study, the prepared macroporous Al_2O_3/TiO_2 membrane support was sintered at various temperatures. The phase composition of the support A85/15 (Al_2O_3/TiO_2 = 85/15) sintered at 1200°C consisted of α-Al_2O_3 and TiO_2 (rutile phase).

Figure 3 shows the microstructure of the porous alumina ceramics sintered at 1200°C with an addition of 0, 5, 10, 15, and 20 wt% of titania (A_1, A_2, A_3, A_4, and A_5, respectively). It can be seen that only coarse-grain is Al_2O_3 in Figure 3a in which the sample is without any addition of titania. The samples with the addition of titania consisted of both coarse-grained Al_2O_3 and fine-grain TiO_2 that are presented in Figure 3(b-e). The fine TiO_2 particles gradually appeared more when the amount of titania was increased. These SEM micrographs show that the pores between the grains resulted in the water absorption and open porosity exhibited in Table 1.

Conclusions

Porous alumina ceramics were prepared by an extrusion method and subsequently sintered at 1200°C for 1 h. The dough for the extrusion was prepared by mixing alumina and small amounts of titania with organic additives. It was shown that the addition of small amounts of titania led to an increase in the bending strength. With the addition of 5, 10, and 15 wt% of TiO_2, the bending strength of the sintered specimens was higher than 18 MPa with porosity of more than 40%. The SEM micrographs demonstrated that the pores between grains gave rise to the water absorption of the samples.

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