

Ceramics International 33 (2007) 551-556

CERAMICS INTERNATIONAL

www.elsevier.com/locate/ceramint

Effects of composition and temperature on porosity and pore size distribution of porous ceramics prepared from Al(OH)₃ and kaolinite gangue

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Abstract

Porous corundum-mullite ceramics were made from $Al(OH)_3$ and kaolinite gangue by the in situ decomposition pore-forming technique. The porosity of the sample increases with increasing $Al(OH)_3$ content in the raw mixture of $Al(OH)_3$ powder and kaolinite gangue. With increasing temperature from 1300 to 1500 °C the porosity of the sample changes a little because of mullite formation. However, when the sintering temperature increases from 1500 to 1600 °C the porosity decreases rapidly. The pores in the samples consist of two groups. One is composed of micropores with maximum diameter of 100 nm. This kind of pores are located in the pseudomorphs of $Al(OH)_3$ and kaolinite gangue. They are derived from the decomposition of $Al(OH)_3$ and kaolinite, and from burning of carbon and organic substance in the gangue. With increase of pores with maximum diameter of 1000 nm. They are located among the pseudomorphs of $Al(OH)_3$ and kaolinite gangue. With increase of the $Al(OH)_3$ content in the starting powder mixture the % volume of micropores (pore diameter 250 nm) increases, while it decreases with the increase of the sintering temperature.

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Keywords: In situ decomposition; Pore-forming; Porous; Corundum-mullite; Al(OH)3; Kaolinite gangue

1. Introduction

Recently, there has been an increasing interest in the applications of porous ceramics as filters, desiccants, insulators, catalyst supports, bone replacement, acoustic absorbers, sensors and membrane reactors [1–6].

Porous ceramics can be made by adding pore-forming agents such as sawdust, starch, carbon or organic particulates [7] into the starting powders, or by injection molding [8], or by gelcasting [1]. Deng et al. [9,10] made porous alumina ceramics by decomposition of Al(OH)₃. This pore-forming in situ technique exploiting the decomposition of starting powders is a good way to prepare porous ceramics containing well-distributed pores. Mullite has a lower thermal-expansion coefficient [11,12] than alumina. Introducing mullite into corundum ceramics can increase their thermal-shock resistance as well as their mechanical and chemical stability [13,14]. We have used the in situ decomposition pore-forming technique to prepare porous raw materials [15]. On the other hand kaolinite gangue is a large coal mine waste in China. Utilization of this gangue to decrease pollution of the environment is interesting. Compared to microsilica, kaolinite gangue has a pore-forming ability because kaolinite will lose water and the carbon in the gangue will burn out during sintering. It means that the gangue has two pore-forming abilities. Using kaolinite gangue instead of microsilica may increase the porosity of porous ceramics. This article describes the preparation of porous corundum-mullite ceramics, using Al(OH)₃ powder and kaolinite gangue. Open porosity and pore size distribution of porous corundum-mullite ceramics were investigated as a function of the composition of the starting powders and the sintering temperature. Comparison of porosity and pore size distribution of samples made from microsilica and gangue will be presented as well.

corundum-mullite ceramics, using Al(OH)₃ and microsilica as

2. Experimental procedure

The compositions of the starting powders with different kaolinite gangue and $Al(OH)_3$ content are given in Table 1. The starting powders were wet milled for 3 h in a planetary mill using

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Table 1Main composition of different starting powders (wt%)

| | 1# | 2# | 3# | |
|----------------------|-----|----|----|--|
| Al(OH) ₃ | 54 | 68 | 84 | |
| Kaolinite gangue | 46 | 32 | 16 | |
| Mullite ^a | 100 | 71 | 35 | |

^a Mullite: the content of mullite in sintered specimens was calculated from the composition of starting powders.



Fig. 1. The particle size distribution of milled starting powder.

alumina balls and pressed to billets with diameter of 36 mm under 50 MPa. The green billets were dried at 110 $^{\circ}$ C for 24 h, and then heated at 1300, 1400, 1500 or 1600 $^{\circ}$ C for 3 h, respectively. Particle size distribution of milled powders is given in Fig. 1.

The chemical compositions of the raw materials are listed in Table 2.

The particle size distribution was measured by laser particle size analyzer (Matersizer 2000). X-ray diffractometry (Philips X'pert TMP) was used to analyze the phase composition in the sintered specimens heated at different temperatures. Apparent porosity was detected by Archimedes' Principle with water as medium. The pore size distribution was measured by mercury intrusion porosimetry (AutoPore IV 9500, Micromeritics Instrument Corporation) and the microstructure was analyzed by SEM (Philips XL 30 TMP scanning electron microscopy).

3. Results and discussion

3.1. Effect of the composition of the starting powders on the pore structures of the sintered specimens

The relationship between the $Al(OH)_3$ content in the starting powders and apparent porosity is shown in Fig. 2. As shown in

| Table 2 | | | | |
|--------------|-------------|--------|-------------|------|
| The chemical | composition | of raw | materials (| wt%) |



Fig. 2. Variation of the apparent porosities of the specimens sintered at 1500 $^{\circ}$ C for 3 h with the Al(OH)₃ content in starting powders.

Fig. 3. Variation of the pore size distribution of the porous specimens with different starting powders sintered at 1500 $^\circ$ C for 3 h.

Fig. 2, with increasing the $Al(OH)_3$ content in the starting powders the apparent porosity of sintered specimens increases.

The pore size distributions of the specimens prepared from different starting powders sintered at 1500 °C are shown in Fig. 3. They are bimodal. One pore group is representative of micropore the diameter of which is mostly about 100 nm and the other is formed by bigger pores the size of which is mostly about 1 μ m.

| Ee Oa | C-0 | 14.0 | | | | |
|-------|-------|------------------------|----------------------------------|--|--|---|
| 10203 | CaO | MgO | K_2O | Na ₂ O | TiO ₂ | IL |
| 0.041 | 0.15 | 0.04 | 0.013 | 0.039 | _ | 32.60 |
| 0.21 | 0.15 | 0.15 | 0.084 | 0.028 | 0.43 | 17.89 |
| ; | 0.041 | 0.041 0.15 0.21 0.15 | 0.041 0.15 0.04 0.21 0.15 0.15 | 0.041 0.15 0.04 0.013 0.21 0.15 0.15 0.084 | 0.041 0.15 0.04 0.013 0.039 0.21 0.15 0.15 0.084 0.028 | $\begin{array}{c ccccccccccccccccccccccccccccccccccc$ |

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