

Effects of pore structure on thermal conductivity and strength of alumina porous ceramics using carbon black as pore-forming agent

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ABSTRACT

In the present work, carbon black (CB) works as a pore-forming agent in the preparation of alumina porous ceramics. The pore structures (*i.e.* mean pore size, pore size distribution and various pores size proportions) were characterized by means of Micro-image Analysis and Process System (MIAPS) software and mercury intrusion porosimetry. Then their correlation and thermal conductivity as well as strength were determined using grey relation theory. The results showed that the porosity and mean pore size increased against the amount of CB, whereas the thermal conductivity, cold crushing strength and cold modulus of rupture reduced. The $< 2 \mu\text{m}$ pores were helpful for enhancing the strength and decreasing the thermal conductivity whereas the $> 14 \mu\text{m}$ pores had the opposite effects.

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1. Introduction

Alumina porous ceramics are widely applied in many areas such as thermal insulation, working lining with the service temperature up to 1600°C [1–5]. In the past few decades, alumina hollow spheres are used as the starting materials in the manufacturing process of such refractories. However, such starting materials are commonly fabricated by the blowing process after melting industrial alumina in electric-arc furnace, which leads to a large amount consumption of electricity [6–10]. It is necessary to develop an economical process for the fabrication of alumina porous ceramics.

Up till now, burning pore-forming agents in the materials to generate pores is a promising approach for the fabrication of such ceramics. Various pore-forming agents such as rice husk, sawdust, starch, poppy seed and polymethylmethacrylate (PMMA) microspheres *etc.* are successfully applied [11–16]. For instance, Mohanta *et al.* [14] reported the use of rice husk as the pore former for the fabrication of an alumina porous ceramics. Such ceramic has a porosity of 66%, a mean pore size of $50\text{--}516 \mu\text{m}$ and a thermal conductivity of $1.2 \text{ W m}^{-1} \text{ K}^{-1}$. In addition, alumina porous ceramics can achieve a thermal conductivity of $0.45 \text{ W m}^{-1} \text{ K}^{-1}$ with the porosity of 70% and a mean pore size of $150 \mu\text{m}$ using sawdust as the pore-forming agent. However, the strength of such ceramics is only 0.8 MPa [16]. It can be seen that irregular and larger pores are formed

in the porous ceramics due to the agglomeration and nonuniformity of pore-forming agents [17–18], leading to the lower strength and higher thermal conductivity [19]. Moreover, impurities such as K_2O , Na_2O and SiO_2 may also deteriorate the high temperature performance of the porous ceramics [20–21].

Theoretically, the spherical micro-pores and nano-pores can improve the mechanical properties and decrease the thermal conductivity of porous ceramics [22–24]. Compared with conventional pore-forming agents, carbon black is characterized as small particle size in nano-scale and spherical shape. Furthermore, such agent has no impurities left after burning out at high temperature. Consequently, in the present work, carbon black was introduced as the pore-forming agent of alumina porous ceramics. Additionally, metallic aluminum powder was added in the preparation of such ceramics, which will be oxidized at approximate 600°C to improve the strength at low temperature. The Weibull modulus was also used to evaluate the stability of strength. Moreover, the effects of pore structure on the thermal conductivity and strength of alumina porous ceramics were investigated in detail.

2. Experimental

2.1. Raw material and specimen preparation

In the experiment, tabular alumina ($\leq 0.074 \text{ mm}$ and $\leq 0.045 \text{ mm}$, 98% Al_2O_3 , Qingdao Almatiss Premium Alumina Co., Ltd., China); $\alpha\text{-Al}_2\text{O}_3$ ($2 \mu\text{m}$, 99% Al_2O_3 , Kaifeng Special Refractories Co., Ltd., China);

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Table 1
The batch composition of the samples.

Sample no.	C0	C1	C2	C3	C4	C5	C6
Tabular alumina	20	20	20	20	20	20	20
Micron sized α -Al ₂ O ₃	74	69	64	59	54	49	44
ρ -Al ₂ O ₃ ($\leq 5 \mu\text{m}$)	5	5	5	5	5	5	5
Carbon black	–	5	10	15	20	25	30
Metal aluminum powder (0.045 mm)	1	1	1	1	1	1	1
PVA	+8	+8	+8	+8	+8	+8	+8

carbon black (CB, N774, 50–100 nm, Wuhan Kebang New Material Co., Ltd., China), ρ -Al₂O₃ (5 μm , 85% Al₂O₃, Kaifeng Special Refractories Co., Ltd., China) and aluminum powder ($\leq 0.045 \text{ mm}$, 98% Al, Xinxiang Zhongyuan Alumina Material Co., Ltd., China) were used as starting materials, with commercially-available poly vinyl alcohol solution (PVA, liquid, the specific gravity is 1.02 g/cm³) as a binder. The batch compositions are shown in Table 1. The mixtures of raw materials were first wet-milled at a rotating rate of 300 rpm in a planetary balling for 3 h (corundum balls as the abrasive media) using absolute ethyl alcohol as the disperse media. The mass ratio of the raw materials:corundum balls:absolute ethyl alcohol was 1:3:1. Then the as-prepared mixtures were mixed for 30 min with 8 wt% PVA solution in a mixer with the rotating speed of 80–100 rpm after dried at 110 °C for 24 h. After kneading, the mixtures were pressed at 5 MPa into prismatic specimens (25 × 25 × 140 mm³) and cylindrical specimens (Φ 36 × 36 mm²) and then cured at 110 °C for 24 h. Finally, the specimens were fired at 1550 °C for a soaking time of 3 h in ambient atmosphere with a heating rate of 2 °C/min.

2.2. Testing and characterization

The apparent porosity and bulk density of the fired specimens were measured according to the Archimedes method. The porosity values V_p of the fired specimens were obtained from the bulk and true densities using the following relation:

$$V_p = 1 - \frac{\rho_{\text{bulk}}}{\rho_{\text{true}}} \quad (1)$$

where the true density (ρ_{true}) of alumina is 3.98 g/cm³. All the specimens were cut after firing, polished and coated with gold; the microstructure was observed using scanning electron microscopy (SEM, Quanta400, FEI Company, USA). Then microstructure photographs were analyzed with Micro-images Analysis & Process System (MIAPS) software (Beijing Precise Instrument Co., Ltd, China); The specimens were cut into approximately 6 × 6 × 6 mm³

and then examined by Mercury Intrusion Porosimetry (Autopore IV9500, Micromeritics Instrument Corp., USA); Thermal conductivity measurements were performed on disk specimens (20 mm in thickness and 180 mm in diameter), at 300 °C, 500 °C, 800 °C and 1000 °C respectively using water flow plate thermal conductivity apparatus (PBDR-02, Precondar, PR China). Cold modulus of rupture (CMOR) of the prismatic specimens and cold crushing strength (CCS) of the cylindrical specimens were measured using a computer-controlled universal testing machine (Shenzhen Wance Testing Machine Co., Ltd., China) at a loading rate of 0.05 mm/min. Finally, the results of mechanical tests were analyzed according to Weibull statistical theory. The Weibull modulus is a measure of the scatter of the data and corresponds to the slope of the plot $\ln(\ln(1/(1-F)))$ versus $\ln \sigma_f$. F is the failure probability, $n/(N+1)$, with n the ranking of the sample and N the total number of specimens.

3. Results and discussion

3.1. Porosity of Al₂O₃ porous ceramics

The total porosity (V_p), closed porosity and bulk density of the fired specimens are shown in Fig. 1. It is apparent that the V_p increased with the increment of CB, whereas the bulk density decreases. The amount of closed pores was less than 7% and did not change significantly with respect to the CB content. The specimen without CB addition had a total porosity of 33% and a bulk density of 2.63 g/cm³. In contrast, the V_p increased up to 68% and the bulk density was 1.27 g/cm³ when 30 wt% of CB was added.

3.2. Thermal conductivity of Al₂O₃ porous ceramics

The thermal conductivity of the specimens as a function of temperature is shown in Fig. 2. An enhancement of thermal conductivity was observed with increasing of fire temperature. According to the previous research, the thermal conductivity of both monocrystalline alumina materials and polycrystalline ones decreases with rising of the temperature [25–26]. This was attributed to the acceleration of motion of air molecule in the pores of specimens when the temperature increased [27]. The thermal conductivity decreased sharply at varied temperatures with the increase of CB from 0 wt% up to 10 wt%, and only slight decline was found when further increasing the amounts of CB up to 25 wt%. The thermal conductivity rose again when 30 wt% of CB was added.

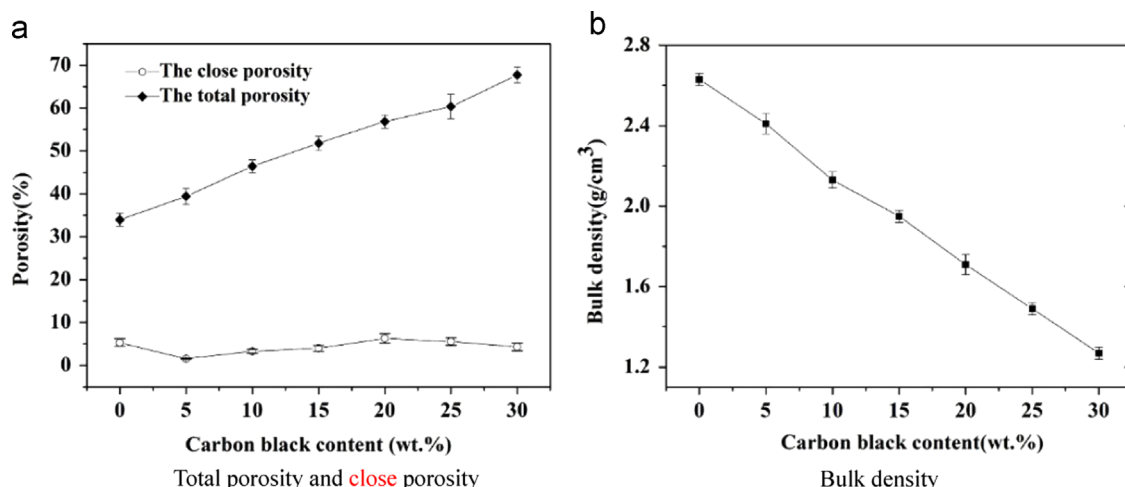


Fig. 1. Total porosity, close porosity and bulk density of fired samples with different carbon black contents.

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