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Correlations among processing parameters and porosity of a lightweight alumina

Lvping Fu*, Huazhi Gu, Ao Huang, Hongwei Ni

The State Key Laboratory of Refractories and Metallurgy, Wuhan University of Science and Technology, Wuhan, Hubei 430081, China

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ABSTRACT

In this study, lightweight alumina was fabricated using α-Al_2O_3 micropowder as the raw material and corn starch as a pore-forming agent. Orthogonal experiments were designed to investigate the effect of particle size, pore-forming agent addition, and sintering temperature on the density and porosity of the lightweight alumina. The experimental results were analysed using a one-way analysis of variance and non-linear fitting, and the correlation between each processing parameter and property was discussed. The results indicate that the bulk density and total porosity of lightweight alumina are mainly affected by the pore-forming agent addition, while the sintering temperature is the main contributor to the apparent and closed porosity of the samples. Based on the Brook theory, dynamics analysis was performed on various samples. The difference in physical properties of various samples arose from differences in the relationship between grain boundaries and pore migration velocity. By adjusting the processing parameters, lightweight alumina with low bulk density, low apparent porosity, and high closed porosity could be obtained.

1. Introduction

Energy and resources have important strategic positions in economic development. With the diminishment of resources, energy preservation and emission reductions in industrial furnaces become increasingly important. Low-thermal-conductivity lightweight refractories show better heat insulation functions when they are laid closer to the wear lining [1–3]. Therefore, the goal of designing lightweight wear linings for industrial furnaces has attracted increased attention in the refractory industry. However, the introduction of pores for lightweighting could increase the risk of molten slag penetration and affect the mechanical properties of the refractory material. The miniaturisation of pore size and reduction of apparent porosity are expected to improve the slag resistance and mechanical properties of lightweight materials [4–10]. Hence, the key challenge for the development of lightweight wear linings lies in the fabrication of porous materials with the minimum possible apparent porosity and pore size [11,12].

Many studies have investigated the effects of preparation parameters, including raw material particle size [13–16], heat treatment [17–19], and pore-forming agent addition [20–22] on the properties and microstructures of porous materials. Yan et al. [13] investigated the effect of particle size on the pore characterisation of porous cordierite–mullite ceramics, and found that decreasing particle size corresponded to reduced porosity and pore size. Miao et al. [17] reported that, as the sintering temperature was increased from 1300 °C to 1450 °C, the apparent porosity of a porous anorthite–mullite–corundum ceramic changed slightly, but the pore size increased. Zhang et al. [20] fabricated porous lead zirconate titanate (PZT) ceramics with different porosities using various pore-forming agents. Although the physical properties of porous material are assumed to be closely associated with the processing parameters, the correlation between each processing parameter and physical property remain poorly understood. The exact effects of various processing parameters on different physical properties, especially for different types of porosities (apparent and closed porosity), cannot be well described, despite their importance in developing lightweight wear lining materials with guaranteed slag resistance and good mechanical properties.

Therefore, in this study, lightweight alumina was fabricated by using α-Al_2O_3 micropowder as a raw material and corn starch as a pore-forming agent. Orthogonal experiments were designed to investigate the effect of particle size, pore-forming agent addition, and sintering temperature on the density and porosities (including total, apparent, and closed porosities) of the lightweight alumina. The experimental results were analysed using the one-way analysis of variance (ANOVA) method and non-linear fitting, and the correlation between each factor and property was discussed. Moreover, based on the Brook theory, dynamics analysis was performed to explore the main reason

* Corresponding author. Permanent address: No. 947 Heping Avenue, Qingshan District, Wuhan, Hubei Zip: 430081, PR China.
E-mail address: lvping_fu@163.com (L. Fu).

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Table 1
The compositions of the raw materials (wt%).

Raw materials	Compositions			
	Al_2O_3	SiO_2	Na_2O	IL
α-Al_2O_3 micropowder I#	99.41	0.03	0.33	–
α-Al_2O_3 micropowder II#	99.80	0.06	0.10	–

underlying the differences in the physical properties of various samples with different processing parameters.

2. Experimental

2.1. Raw materials

Two kinds of α-Al_2O_3 micropowder with different particle sizes were selected as raw materials: α-Al_2O_3 micropowder I# (D_{50} = 2.36 μm, Kaifeng Special Refractory Co., Ltd., China), and α-Al_2O_3 micropowder II# (D_{50} = 1.53 μm, Hubei Smile New Materials Co., Ltd., China); their compositions are listed in Table 1. Both α-Al_2O_3 micropowder types exhibit high purities (Al_2O_3 content > 99.4 wt%) and low SiO_2 contents. α-Al_2O_3 micropowder I# has a higher Na_2O content than α-Al_2O_3 micropowder II#.

Corn starch (D_{50} = 14.287 μm, Shandong Hengren Starch Co., Ltd., China) was chosen as a pore former and binder.

The particle size distributions of the raw materials and pore former were measured by a laser particle size analyser (Mastersizer 2000, Malvern Instruments Co., Ltd., UK). As shown in Fig. 1, the pore size distributions of both two kinds of α-Al_2O_3 micropowder exhibit a single peak. However, the particle size distribution of α-Al_2O_3 micropowder I# shows a larger range centred at 0.4–9 μm, while the vast majority of particles in α-Al_2O_3 micropowder II# are within the range of 0.4–5 μm.

2.2. Sample preparation

In this study, by changing the particle sizes of the raw materials, addition of pore-forming agent, and sintering temperature, a full 2^3 orthogonal experiment was designed. For the particle size, α-Al_2O_3 micropowders I# and II# with different particle sizes were used. The pore-forming agent addition levels of 10 wt% and 20 wt% corn starch were used. The specimens were sintered at either 1830 °C or 1780 °C. The design of the orthogonal experiment is shown in Table 2.

According to Table 2, sample mixtures of α-Al_2O_3 micropowder and corn starch in water, with approximately 40 wt% of the previously mentioned raw materials, were weighted and mixed in a planetary ball mill (QM-BP, Nanjing Nanda Instrument Plant, China) for 30 min to produce slurries. The details of the wet-milling experiment are as follows: alumina balls of 8–14 mm in diameter were used with the charge

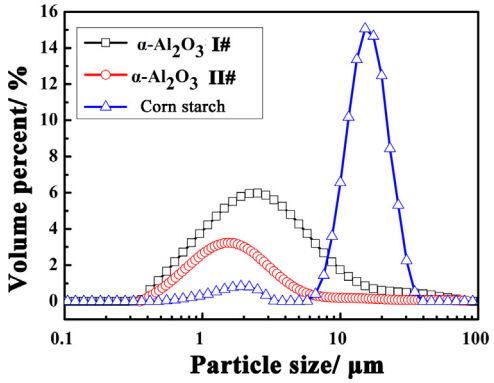

Fig. 1. Particle size distributions of the raw materials and pore-forming agent.

Table 2
Design of the 2^3 orthogonal experiment.

No.	Factors		
	α-Al_2O_3 micropowder	Corn starch	Sintering temperature
1	I#	10 wt%	1830 °C
2	I#	10 wt%	1780 °C
3	I#	20 wt%	1830 °C
4	I#	20 wt%	1780 °C
5	II#	10 wt%	1830 °C
6	II#	10 wt%	1780 °C
7	II#	20 wt%	1830 °C
8	II#	20 wt%	1780 °C

ratio of 7:1 and the rotation speed was 365 r/min. The slurry was poured into a plastic mould and consolidated by starch thermogelation after baking at 80 °C for 24 h. After demoulding, the samples were dried at 110 °C for 24 h. The green bodies were then heated using a chamber electric furnace (SX18-12–8, Luoyang Precondar Instruments for Testing Refractoriness Co., Ltd., China) to obtain the lightweight alumina specimens. The sintering cycle was heating-up at 10 °C/min from room temperature to 1000 °C, at 5 °C/min from 1000 °C to the selected sintering temperatures, as listed in Table 2, and plateauing 3 h at sintering temperatures, and then allowed to cool to room temperature at a rate of 10 °C/min. In such conditions, the effects of heating and cooling sequences on the final sintering state of the material can be neglected when compared to the effect of the 3 h spent at the maximum temperature. Then, this later will further be considered as the reference sintering temperature.

2.3. Testing and characterisation

The bulk densities and apparent porosities of the samples were determined using a gravimetric method based on the Archimedes principle with water as the medium, according to the ISO 5017:1998 standard. The true density was measured by an automatic true density analyser (Accupyc 1330, Micromeritrics Instrument Corporation, Norcross, USA) using 325 mesh-sieved powders from the milled samples. The total and closed porosities of samples were calculated according to the following equations, respectively:

$$\pi_{tot} = \frac{\rho_{tru} - \rho_b}{\rho_t} \times 100\% \quad (1)$$

$$\pi_c = \pi_{tot} - \pi_a \quad (2)$$

where π_{tot}, π_c, and π_a are the total, closed, and apparent porosities, respectively; ρ_t and ρ_b are the true and bulk densities of the samples, respectively.

3. Results and discussion

3.1. Data analysis

In this study, Eq. (3), an empirical formula, was used to describe the influences of the three factors and their interaction effects on the properties of the prepared lightweight alumina [23].

$$p = \alpha_0 + \alpha_1\overline{X_1} + \alpha_2\overline{X_2} + \alpha_3\overline{X_3} + \alpha_{12}\overline{X_1X_2} + \alpha_{13}\overline{X_1X_3} + \alpha_{23}\overline{X_2X_3} + \alpha_{123}\overline{X_1X_2X_3} \quad (3)$$

where p is the property parameter of the samples; $\overline{X_1}$, $\overline{X_2}$, and $\overline{X_3}$ are the unordered variables of the three factors, respectively; and α_0, α_1, α_2, α_3, α_{12}, α_{13}, α_{23}, and α_{123} are the impact factors.

As shown in Table 3, the factors in the experiment are set as unordered categorical variables.

To guarantee the accuracy of the experimental results and the further estimation of the intrinsic scatter of each measurement, for each