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# Effects of pyrolusite additive on the microstructure and mechanical strength of corundum–mullite ceramics

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**Abstract**

Corundum–mullite ceramics have been successfully synthesized by using the raw materials of bauxite and kaolin clay. The effects of pyrolusite additive on the microstructure and mechanical strength of the corundum–mullite duplex ceramics were systematically investigated. The results show that the duplex ceramics exhibit the optimal performance with the amount of 4 wt% additives at 1350 °C. The liquid glass phases appear during the sintering process due to the presence of the impurities in pyrolusite powder, which is beneficial to the growth of crystal, the gap decrease of grain boundary and pores, and the improvement of compressive strength. Moreover, the component of $MnO_2$ could lead to the distortion of lattice, and further improve the mechanical strength via a solid solution strengthening mechanism.


*Keywords:* Corundum–mullite; Mechanical strength; Microstructure; Pyrolusite

## 1. Introduction

Corundum–mullite material, as one widely used in duplex ceramics for application [1,2], centralizes the advantages of corundum and mullite ceramics and is regarded as a quite promising engineering material at high temperatures because of its remarkable thermal shock resistance, high temperature resistance and creep characteristics [3–5]. Moreover, the duplex ceramic has been widely applied in kiln furniture at high temperature, such as refractory bricks and pushers [6,7]. Recently, extensive research focuses on the effects of different additives for corundum–mullite ceramics [7,8]. Tripathi et al. studied the mechanical properties of the mullite–alumina ceramics sintered at 1400–1600 °C with 2–6 wt% of $ZrO_2$ [7]. The effects of fine $Al_2O_3$ additive on the mechanical properties of corundum–mullite ceramics were also investigated at 1550–1700 °C for 4 h, and the results showed that thermal shock resistance mainly depends on sintering temperature [8]. However, the synthetic temperatures of these ceramic materials were much higher, which will limit the wide range of practical applications. Therefore, it is crucial to choose suitable additive to lower sintering temperature, obtain good densification and improve compressive property of the duplex ceramics.

The pyrolusite powder, possessing outstanding properties, is considered as an ideal additive. It is able to decrease sintering temperature and promote the formation of glass phases [9]. However, related studies have not been conducted on corundum–mullite with the additive of pyrolusite powder especially applying in molten steel lining materials,

Herein, the purpose of this study is to further improve the performance of corundum–mullite and make it possible to industrial application of molten steel lining materials [10]. In our work, corundum–mullite ceramics with the additive of pyrolusite powder were prepared by raw material of bauxite and kaolin clay, in the light of the characteristics of molten steel lining material for excellent high temperature performance and

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radial compressive properties. The effects of pyrolusite powder on microstructure and properties of the ceramic samples were systematically investigated.

## 2. Experimental procedure

### 2.1. Raw materials

Secondary bauxite and kaolin clay (Shanxi Yangquan, China) with particle size less than 0.05 mm were used as raw materials. Pyrolusite powder (Shanxi Yangquan Changqing Co. Ltd., China) with the particle size less than 0.074 mm was selected as additive. The chemical composition of the raw materials is shown in Table 1.

### 2.2. Sample preparation

The secondary bauxite and kaolin clay were firstly crushed into powder, and then the starting materials were prepared by screening (100 #) with a shock pendulum sand screening machine (SSZ). According to the certain proportion, as shown in Table 2, each starting material was weighed accurately and ball-milled in pure water for 1 h with highly pure $Al_2O_3$ balls, and then dried at 100 °C for 24 h. 5 wt.% PVA was added to the prepared powders and sieved through screening (100 #) to break up agglomerates. In order to ensure samples formability and avoid cracks and fracture appearing in the surface of the samples the height of samples was decreased and the mixed powders were dry-compressed into a stainless steel die 50 mm in diameter and 30 mm long under the pressure of 300 MPa. Subsequently, the obtained compacts without pyrolusite powder were put into an electric furnace and sintered in argon atmosphere at different temperatures (1250 °C, 1300 °C, 1350 °C, 1400 °C, and 1450 °C) for 3 h. The samples with 0 wt%, 2 wt%, 4 wt%, and 6 wt.% additive were sintered at 1350 °C and signed as A, B, C, and D, respectively, as shown in Table 2.

Table 1
Chemical composition of raw materials.

| Raw material | $Al_2O_3$ (wt%) | $SiO_2$ (wt %) | $Fe_2O_3$ (wt%) | CaO (wt %) | MgO (wt %) | $TiO_2$ (wt %) | $MnO_2$ (wt%) | L.O.I (wt %) |
|---|---|---|---|---|---|---|---|---|
| Bauxite | 71 | 3.6 | 2.8 | 0.56 | 0.58 | 3.9 | – | 17.56 |
| Kaolin | 35.06 | 47.1 | 0.28 | 2.79 | 0.32 | – | – | 14.45 |
| Pyrolusite powder | 4.52 | 18.41 | 13.43 | 1.82 | 0.67 | 0.05 | 56.82 | 4.28 |

Table 2
Design and formulation of experiment.

| Sample | Pyrolusite powder (wt%) | Bauxite (wt%) | Kaolin (wt%) |
|---|---|---|---|
| A | 0 | 41 | 59 |
| B | 2 | 40.2 | 57.8 |
| C | 4 | 39.3 | 56.7 |
| D | 6 | 38.5 | 55.5 |

### 2.3. Sample characterization

The density and open porosity of the sintered samples were measured by the standard Archimedes immersion method using water as medium. Linear shrinkage was obtained by comparing diameters of the specimens before and after sintered. The compressive strength was measured on a Hydraulic universal testing machine (WE-1000 A, Jinan Machinery Factory, China). The final testing results were obtained from the average value of 3 measurements. The microstructures of the specimens were characterized by an field emission scanning electron microscope (FESEM, S-4800). Particle size of the raw material powders was obtained by a laser diffraction method (Mastersizer 2000E, Malvern Instruments Ltd, England). Phase identification was investigated by X-ray diffraction (XRD, Philips Co. Ltd, Holland) using Ni filtered Cu Kα radiation with scanning speed of 0.02°/step. The structural parameters of the crystal phase were analyzed by X'Pert plus software.

## 3. Experimental results

### 3.1. Effect of sintering temperatures on the properties of the samples without additive

The relative density and compressive strength of the samples as a function of sintering temperatures are shown in Fig. 1. It can be seen that the relative density and compressive strength of the samples are enhanced with the temperature increasing, and then become steady from 1350 °C to 1450 °C. The relative density reaches approximately 91%, and compressive strength reaches 20 MPa. According to the above results, the optimum sintering temperature can be established at 1350 °C for the following work.

### 3.2. Effect of additive on the properties of the samples

Fig. 2 presents the effect of additive content on the apparent porosity and bulk density of the samples fabricated at 1350 °C. It indicates that the apparent porosity gets lower and the bulk

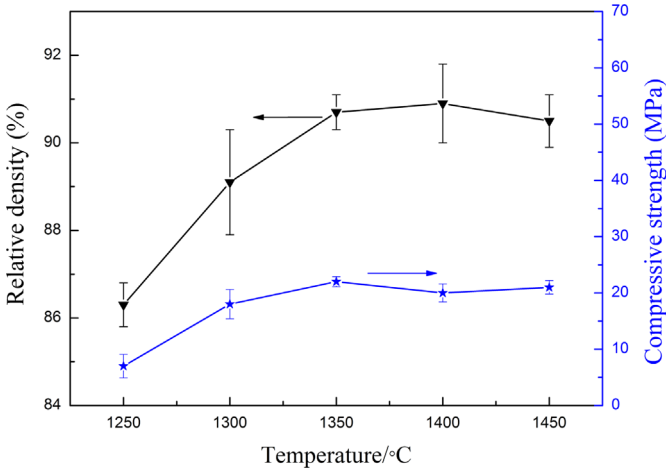


Fig. 1. Effects of sintering temperature on relative density and compressive strength of samples.