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Effect of zirconia particle size on the properties of alumina-spinel castables

Yanshan Liu *, Bingqiang Han *, Ting Zhang, Hui Yu, Wen Yan, Yaowu Wei, Nan Li

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

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ABSTRACT

The industrial application of alumina-spinel refractory castables has crucial requirements on the service performance. Thus, the effects of different sized desilicated zirconia particles on the castables microstructure, thermal-mechanical properties and high temperature elastic modulus have been investigated. The zirconia particle sizes were varied from 1000 μm to 2.5 μm (d_{50}). It was observed that the finer (below 88 μm) zirconia particles were beneficial to improve the cold modulus of rupture (CMOR) and the hot modulus of rupture (HMOR), but could not effectively enhance the thermal shock resistance. Fine zirconia particles can homogeneously disperse in the matrix and significantly promote the sintering process. Accompanied with the phase transformation of zirconia, both the high density of matrix cracks and the strong ceramic bonding (between the coarse grains and the matrix) were found in the refractory castables, which was responsible for an increase of CMOR. However, the binding characteristic could also give rise to the high stored elastic energy that was adverse to the thermal shock resistance, and the excessive amount of preexisting matrix cracks could induce more microdamage during the thermal shock. Additionally, it was proposed that the second-phase dispersion reinforcement and the highly ceramics bonding resulted in the superior HMOR when introducing fine ZrO_2 particles.

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1. Introduction

The increasing demand and application of refractory castables suitable for quality steel making process encourage researchers to investigate their physical and thermal-mechanical properties [1,2]. Alumina-spinel castables are widely used as steel ladle linings below the slag line owing to the excellent properties, such as high resistance to structural spalling, high corrosion resistance to basic slag and good thermal shock resistance [3–6]. Generally, refractory castables are comprised by matrix fraction (bonding phase) and reinforced by coarse aggregates. The reactions between constituents and microstructural evolutions in the matrix strongly affect the properties both at ambient and elevated temperatures [7]. To further enhance the performance of the castables based on alumina-spinel system, many studies have been taken by researchers. On the one hand, adding additives such as ZrO_2, Y_2O_3, TiO_2 and Cr_2O_3 were reported to obtain the beneficial effects on the sintered properties of refractory samples [8–11]. Moreover, it is advisable to select the desired particle sizes of these additives to make them far more reactive in the refractories. Introduction of nano size materials in fired matrix is of great importance in recent years [12–16]. However, the inhomogenity in dispersion and higher price of nano scaled powders limits the use of these additives.

Zircona (ZrO_2) with highly refractory nature and good resistance to aggressive environment can be added into the castables [17,18]. It is well known that the toughening mechanisms of ZrO_2 is mainly based on the tetragonal to monoclinic martensitic transformation and microcracks formation [19,20]. During the cooling stage, the local intrinsic stress fields, resulting from the difference in thermal expansion coefficient of constituents, can be relaxed by propagating cracks. Accompanied by a volume expansion (4–5%), the phase transformation of ZrO_2 can absorb the propagating energy and thus avoid the microdamage of ceramics. Furthermore, with the applied load, the principal cracks will propagate and finally lead to gradual rupture of refractory materials. Such propagation can be inhibited by the existed microcracks that derived from the reversible transformation of ZrO_2 [21,22]. Rasim et al. [23] incorporated ZrO_2 into MgO-spinel composite refractories and found that both the mechanical properties and thermal shock resistance were improved. They related the microcracks interlinking to each other and the inhibition of cracks propagation by the pores and ZrO_2 were two of the decisive factors. In addition to mica glass-ceramic composites toughened by Y-PSZ particles, Montazerian et al. [24] also pointed out that the

* Corresponding author. Permanent address: 947# Heping Road, Qingshan District, Wuhan City, Hubei Province 430081, PR China.
E-mail addresses: mylyss@163.com (Y. Liu), kedawulys@163.com (B. Han).

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crack deflection by dispersion ZrO_2 particles was a prevalent mechanism for improving mechanical properties, while transformation toughening had a little contribution to the increase of fracture toughness.

Generally, fine additive particles are found to have advantageous sintering characteristics: 1) the high surface area to volume ratio that increases the driving force for sintering; 2) the better distribution throughout the compact [25,26]. However, the properties of materials are also depended on other process variables, especially for the crystal transformation of ZrO_2, which can lead to great changes in the microstructure. M. Ghaffari et al. [27] investigated the mica-diopside glass-ceramic composites reinforced by nano sized and micro sized zirconia particles and found that the nano sized zirconia agglomerated in the microstructures and improved fracture toughness more effectively, but the improvement of mechanical properties was more influenced by micro sized zirconia.

From above mentioned summary, the different sized ZrO_2 particles have been of interest as additives to promote the materials performance. However, the effects of different sized ZrO_2 particles on the alumina-spinel castables such as sintering behavior and thermal-mechanical properties have not been studied sufficiently. In this study, we have examined both the room temperature properties and high temperature properties of the castables with different sized m-ZrO_2 particles, and the related mechanism was discussed in detail.

2. Materials and experimental procedures

In the present work, commercial tabular alumina (Al_2O_3 99.42 wt%, Zhejiang Zili Co., Ltd, China), white fused alumina (Al_2O_3 98.51 wt%, Zhengzhou Yufa Abrasives Group Co., Ltd, China), synthesized spinel AR78 (Al_2O_3 73.4 wt% and MgO 25.28 wt%, Qingdao Almatis Aluminum Co., Ltd, China), α-Al_2O_3 micropowder (d_{50}=3.5 μm and Al_2O_3 97.69 wt%, Qingdao Almatis Aluminum Co., Ltd, China), microsilica 940 U (SiO_2 94.18 wt%, Elkem, Norway) and calcium aluminate cement Secar71 (Al_2O_3 69.74 wt% and CaO 29.55 wt%, Kerneos, France) were used as the raw materials. The balls (I) and the powders (II) of desilicated zirconia (Luoyang Lier Refractory Material Co., Ltd, China) were selected for the sources of ZrO_2. Their overall chemical compositions are presented in Table 1. In addition, BASF CASTMENT®FS20 was added as the water reducing agent of the castables.

The desilicated zirconia (I) particles with different sizes were prepared by screening after milling. Table 2 presents the details of the formulated castables without and with different sized desilicated zirconia particles, named Y0, Y1, Y2, Y3, Y4 and Y5, respectively. At first, all the raw materials were weighted according to the compositions and mixed for 3 min, then 5 wt% of water with dispersant dissolved was gradually added and mixed for another 2–3 min to obtain the proper flowability (i.e., the flow value in the range of 140–170 mm). Flow measurements were carried out by filling the castable into an inverted cone (ASTMC-230) placed on a vibration table. The cone was then removed to allow the castable to flow under vibration, and after 30 s the castable diameter was measured. After that, the mixtures were casted into samples with a dimension of 40 mm × 40 mm × 160 mm. The green samples were air dried for 24 h, and subsequently dried in an oven at 110 °C for 24 h. Next the samples were fired at 1100 °C and 1600 °C with 3 h soaking time.

Table 1
Chemical composition of two kinds of desilicated zirconia, wt%.

Type	SiO_2	Al_2O_3	MgO	Fe_2O_3	CaO	K_2O	Na_2O	TiO_2	ZrO_2	I.L.[a]
I	1.63	4.54	0.11	0.44	0.13	0.002	0.047	0.21	92.52	0.27
II	0.021	0.2	0.24	0.03	0.21	0.01	0.38	0.16	98.48	0.23

[a] Ignition loss.

The linear change of samples was determined by the length measurements before and after the firing process. The apparent porosity (AP) and bulk density (BD) were measured by the Archimedes' principle with water as the medium. The cold crushing strength (CCS) of the samples was obtained according to Chinese standard GB/T 5072-2008 (equivalent to ISO 10059-2: 2003). The cold modulus of rupture (CMOR) was measured using the three-point bending test following GB/T 3001-2007 (equivalent to ISO 5014:1997). The thermal shock resistance of samples after firing at 1600 °C for 3 h was evaluated using a water quenching method. The dried samples were heated at 1100 °C for 0.5 h and then put into the water at room temperature for 5 min. Afterwards, the samples were dried and their residual CMOR values were determined after three thermal shock cycles. The thermal shock resistance was evaluated by calculating the residual strength ratio of the CMOR. The hot modulus of rupture (HMOR) of samples fired at 1600 °C for 3 h was measured following GB/T 3002-2004 (equivalent to ISO 5013:1985). The testing temperature was 1400 °C and the soaking time was 30 min.

The elastic modulus is calculated based on the resulted vibration spectrum applying Pickett equations, which correlates the elastic modulus, the natural vibration frequencies and the sample dimensions. For the fundamental flexural frequency of a rectangular bar, the Young's modulus is given by [28]:

$$E = 0.9465\frac{mf_f^2}{b} \times \frac{L^3}{t^3} \times T_1 \quad (1)$$

where E is the Young's modulus (Pa), m is the mass (g), b is the width (mm), L is the length (mm), t is the thickness (mm), f_f is the fundamental resonance frequency of the bar in flexure (Hz), and T_1 is the correction factor for fundamental flexural mode to account for the finite thickness of the bar. The rectangular bar-shaped samples of nominal dimensions 130 mm × 30 mm × 25 mm were prepared by casting. The measurements of the Young's modulus were performed in air up to 1400 °C using dedicated equipment (RFDA-HTVP1600, IMCE, Diepenbeek, Belgium). During the thermal treatments, the samples were supported by Pt-Rh wires that located at the nodes of the first bending mode, and periodically excited by the impact of a small ceramic probe. A microphone located outside the furnace picked up the vibration signal. With an alumina tube being used as acoustic waveguide, the signal was analyzed by the software of RFDA. The samples were heated up and cooled down at a constant rate of 3 °C/min.

Simultaneously, the relative mineralogical phase contents of compositions Y2 and Y5 from 600 °C up to 1600 °C were also calculated by Factsage version 6.4 thermal-chemical software. FToxid database and Equilib modules were selected for this evaluation. The simulations were performed considering the castables' overall composition as the MgO-Al_2O_3-SiO_2-CaO-Fe_2O_3-TiO_2-K_2O-Na_2O-ZrO_2 system.

The fired samples were crushed and then the sections without coarse corundum aggregates were subjected to X-ray diffraction (XRD) analysis using a powder diffractometer (X'pert Pro MPD, PAnalytical B.V., Netherlands) with a Ni-filtered Cu Kα radiation. The microstructures of samples were obtained using a scanning electron microscopy (JSM-6610, JEOL Ltd., Japan) coupled with EDS (QUANTAX200-30, BRUKER, Germany).

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