

Effects of rare earth oxides additions on microstructure and properties of alumina-magnesia refractory castables

Wenjie Yuan*, Qingyou Zhu, Chengji Deng, Hongxi Zhu

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

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ABSTRACT

To accommodate the glaring requirements of castables with high performances for the iron and steel making industry, effects of rare earth oxides additions on microstructure and properties of alumina-magnesia refractory castables were investigated. The properties of castables including phase compositions, apparent porosity, permanent linear change and strength were compared. It was demonstrated that yttria and ceria presented varying degrees of influence on in-situ reactions and volumetric stability of castables. The solid-soluted content of spinel and calcium hexaluminate (CA₆) was both dependent on the additions.

1. Introduction

With the development of the novel technique for the metallurgy industry, the requirements of castables' performances are getting more rigorous. The prolonging service life and reducing consumption of castables attracted more and more attention. In recent years, alumina-magnesia castables have been widely used in the secondary refining and ladle below the slag line because they have better slag resistance than alumina-spinel castables [1]. There were a lot of alternatives for the design of high-performance alumina-magnesia refractory castables such as the sources of raw materials (alumina and magnesia) with different sizes, the microsilica content and the binders (calcium aluminate cement and hydratable or colloidal alumina) [2–5]. The addition of nano-scaled particle and adjustment of particle distribution in matrix for castables also were addressed [6–8]. However, the in situ reactions of the spinel (MgAl₂O₄) and calcium hexaluminate (CA₆) formation resulted in a certain amount of expansion [9], which was a key issue determining the properties of spinel forming alumina-magnesia castables. The use of mineralizing compounds could not only control the overall expansion but also have the marked impact on phase and microstructure evolution of castables [10–12]. All sorts of additives such as fluorides (AlF₃ and MgF₂), chlorides (MgCl₂), oxides (B₂O₃, V₂O₅, TiO₂, Cr₂O₃ and SnO₂), magnesium borate and borosilicate had been used as mineralizers for speeding up the spinel-forming reaction [11,13–15].

Rare earth as “vitamin” for modern industries has received more attention in the ceramic manufacturing since 1930's. The sintering behavior of alumina with rare earth oxides (REOs) was investigated systemically as early as 1979 [16]. Rare earth oxide could improve the

sintering and properties of alumina and magnesium aluminate spinel [17,18]. Thermal shock resistance of spinel refractories and hot modulus of rupture and coating adhering performance of magnesia refractories were boosted with REOs addition especially Y₂O₃ [19–22]. In previous study, rare earth chlorides as primary products in the process to produce rare earth oxides were added into alumina-magnesia refractory castables [23]. In spite of the effects of chloride ion on the setting of cement, a small amount of Y and Ce elements still played a certain role to castables. Therefore, yttria (Y₂O₃) and ceria (CeO₂) as the representative of heavy and light rare earth oxide respectively were selected as additives. The purpose of the present work was to evaluate effects of rare earth oxides additions on microstructure and properties of alumina-magnesia refractory castables. By comparing with corundum castables as the reference, the mechanism of rare earth oxides action in alumina-magnesia castables was discussed.

2. Experimental procedure

For the evaluation of Y₂O₃ and CeO₂ additions, alumina-magnesia castables consisted of tabular aluminas and reactive alumina (Almatis), calcined magnesia (95 wt%), microsilica (Elkem) and calcium aluminate cement (Kerneos). Corundum castables (without magnesia) containing rare earth oxides were also prepared for comparison. All compositions of castables were listed in Table 1. Yttria (d₅₀=3.6 μm) and ceria (d₅₀=15.1 μm) were used as the mineralizing additives. The dispersion was carried out by using 0.2 wt% of an electrosteric dispersant (BASF). The water in the range of 4.2–4.6 wt% was added for the vibratable casting.

* Corresponding author.

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Table 1
Composition of corundum and alumina-magnesia castables.

Raw materials	Content (wt%)									
	A	AY0.5	AY1	AC0.5	AC1	AM	AMY0.5	AMY1	AMC0.5	AMC1
Tabular alumina (≤ 6 mm)	61	61	61	61	61	61	61	61	61	61
Tabular alumina (≤ 0.2 mm)	25	24.5	24	24.5	24	19	18.5	18	18.5	18
Reactive alumina (CL370)	7	7	7	7	7	7	7	7	7	7
Calcined magnesia (180 mesh)	–	–	–	–	–	6	6	6	6	6
Calcium aluminate cement (Secar71)	6	6	6	6	6	6	6	6	6	6
Microsilica (951U)	1	1	1	1	1	1	1	1	1	1
Y ₂ O ₃	–	0.5	1	–	–	–	0.5	1	–	–
CeO ₂	–	–	–	0.5	1	–	–	–	0.5	1

After mixing, castables were molded to two types of outline dimensions. All samples were cured at 25 °C for 24 h in a climatic chamber with relative humidity of 100%, followed by drying at 110 °C for 24 h. The expansion curve of the bar sample (40 mm×40 mm×160 mm) was measured by heating up to 1450 °C at the rate of 5 °C/min using a self-made dilatometer. Other bar samples (25 mm×25 mm×150 mm) were calcined at 1450 °C for 5 h. The permanent linear change (PLC) of castables was measured according to GB/T 5988-2007. The apparent porosity of castables was tested according to the Archimedes technique. Cold modulus of rupture (CMOR) measurements for castables was conducted by three-point bending test according to GB/T 3001-2007. The phase compositions in castables were determined by X-ray diffraction (XRD, Philips, X'pert Pro MPD, Netherlands). The microstructure of castables was observed by scanning electron microscopy (SEM, JEOL JSM-6610, Japan) and energy dispersive spectrometer (EDS, Bruker QUANTAX200-30, Germany).

3. Results and discussion

3.1. Expansion behavior

The thermal expansion of castables was dominated by micro factors. In order to evaluate the effect of additions on the expansion, alumina-magnesia refractory castables containing different amount of Y₂O₃ and CeO₂ as well as the reference samples A and AM were tested. The expansion values for all samples increased with the temperature during the continuous heating stage (Fig. 1). There was an obvious difference of the expansion curves above 1300 °C. Due to the spinel formation, the expansion of the reference AM was greater than corundum castables (sample A). The expansion values of samples AMY0.5, AMY1 and AMC0.5 were in between two reference samples, which was associated with the enhanced sintering by rare earth oxides. While the expansion of sample AMC1 presented a sharply increase at the temperature over 1400 °C. Permanent linear change (PLC) stand-

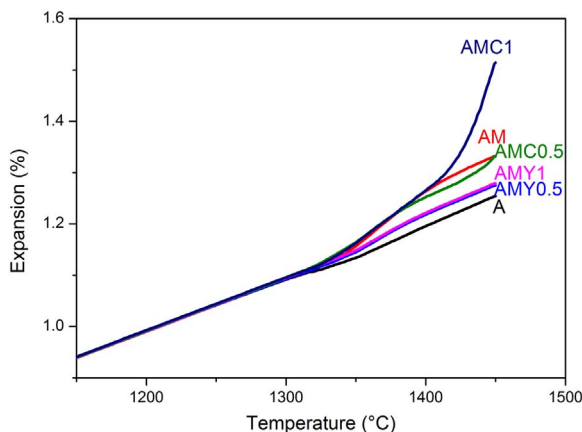


Fig. 1. Linear expansion curves of alumina-magnesia castables.

ing for the volume stability of refractories measured after the cooling was shown in Fig. 2. The sequence of PLC for alumina-magnesia castables containing rare earth oxides was same with the expansion value at 1450 °C in Fig. 1. PLC of reference sample AM was just 0.97%. In contrast, PLC of sample AMC1 reached 2.7%. The increasing scope of PLC for samples AMY0.5 and AMY1 was smaller than castables with CeO₂ addition. Only considering the CA₆ formation, the variation of PLC for corundum castables with rare earth oxides was not so much, which demonstrated that the overall expansion of alumina-magnesia castables was more dominated by the spinel formation. It was worth noting that PLC of AY1 was little less than AY0.5. The addition of CeO₂ was more effective than Y₂O₃ in both systems. To sum up, it was demonstrated that the contribution of spinel formation to the expansion of castables with CeO₂ was more significant than that of CA₆ formation according to the difference between two series of castables. The effect of Y₂O₃ addition on the expansion of castables was probably counterbalanced by the promoted sintering.

3.2. Phase composition and microstructure

The patterns of X-ray diffraction (XRD) for all castables are given in Fig. 3. According to the phase identification results, the major phases were corundum, CA₆ and spinel (for corundum castables). Y₂O₃ reacted with alumina to produce yttrium aluminum garnet (YAG, Al₅Y₃O₁₂) in samples AY0.5, AY1 and AMY1, which was consistent with the equilibrium phase diagram [24]. The amount of Y₂O₃ playing effective role on expansive reactions was reduced because the formation of YAG consumed a part of it. Unreacted CeO₂ only existed in samples AC0.5 and AC1, while there was not any diffraction peak of CeO₂ in XRD patterns of samples AMC0.5 and AMC1. Because the most amount of addition was 1 wt%, rare earth oxides would probably dissolve into spinel and CA₆. The changes of the selected diffraction peak's position for spinel and CA₆ than corundum (104) peak as the reference are

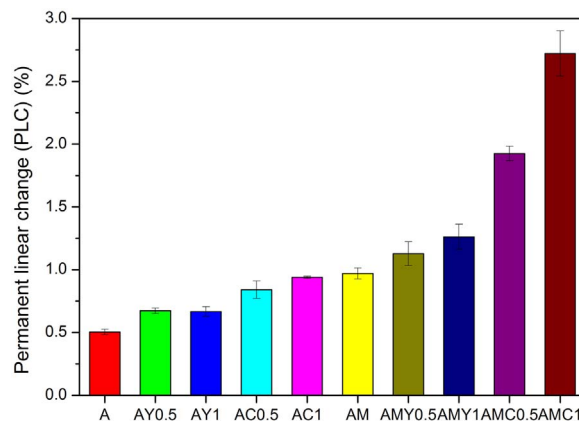


Fig. 2. Permanent linear changes for alumina-magnesia refractory castables after calcined at 1450 °C for 5 h.

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