FULL PAPER

The adjustment of CA₆ morphology and its effect on the thermo-mechanical properties of high temperature composites

Feng WANG¹, Xiangcheng LI^{1,2,†}, Pingan CHEN^{1,‡}, Girish Madhav KALE² and Boquan ZHU¹¹The State Key Laboratory of Refractories and Metallurgy, Wuhan University of Science and Technology, Wuhan 430081, P. R. China²School of Chemical and Process Engineering, University of Leeds, Leeds LS2 9JT, UK

In this paper, the morphology of CA₆ was adjusted by changing the particle sizes of alumina powders ($D_{50} = 7.26, 5.33$ and $2.37 \mu\text{m}$), and the effect of this on the thermo-mechanical properties of corundum based high temperature composites was investigated. The results indicated that when the alumina particle size was decreased from 7.26 to $2.37 \mu\text{m}$, the morphology of calcium hexaluminate (CA₆) transformed from equiaxial to platy shape. The microstructure changes of CA₆ may be due to differences in CA₆ nuclei numbers and the contact area between alumina and calcium dialuminate. This transformation process can proceed without liquid. Because of the transformation of the CA₆ morphology, the hot modulus of rupture and residual cold modulus of rupture of corundum composites fired at 1600°C increased by 143 and 192% , respectively. The loop areas of in-situ elastic modulus also increased by 20% . The fracture ways of composites were transformed from intergranular to transgranular. Therefore, corundum-based composites with excellent properties including room temperature and high temperature properties can be obtained by adjusting the microstructure of CA₆.

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1. Introduction

Refractory castables as a kind of high-temperature composite, have the advantages of easy installation, energy saving and monolithic lining, and have been widely used in high temperature industries, including steel ladels, Ruhrstahl-Heraeus refining furnaces, and iron runners.^(1,2) The typical components of such materials comprise the aggregate, powder and binders. Amongst these, calcium aluminate cement (CAC), the most popular binding system, plays a vital role in the comprehensive properties of workability, mechanical strength and service performance.^(3–5) Furthermore, the phase and microstructure evolution of CAC at elevated temperatures greatly influences hot strength, slag resistance and thermal shock.^(2,6,7) For example, Pinto and Silva et al.⁽⁴⁾ found that the composition with $1 \text{ wt}\%$ CAC exhibited a dried flexural strength 98% higher than that of CAC free, suggesting that the presence of CAC could significantly improve the workability. Liu and Andreas⁽⁸⁾ found that calcium hexaluminate (CA₆) could be easily formed in the castable's matrix and increased its mechanical strength at 1450°C . Auvray and

Huger et al.⁽⁹⁾ further concluded that the bonding phases between the CA₆ and in-situ formation of spinel grains were responsible for enhancing the castable's elastic properties at high temperature.

Recently, researchers have found that besides the different CA₆ phases, their distribution could also significantly impact the thermo-mechanical properties of refractory castables.⁽¹⁰⁾ Sako and Pandolfelli et al.⁽¹¹⁾ found that adding MgAl₂O₄ spinels in-situ or pre-formed in the castables would determine whether the CA₆ was in the aggregate region. The in-situ formation of MgAl₂O₄ spinels could contribute to the CA₆ phases located in the both matrix and aggregates borders, which would improve the slag resistance of the castables. However, the pre-formed MgAl₂O₄ spinels could lead to CA₆ distribution in the matrix, which would result in the high dissolution of aggregates into the molten slag⁽¹¹⁾ and inversely give rise to poor slag resistance. Martinez and Luz et al.⁽¹²⁾ also reported the excellent high temperature properties of alumina-magnesia castables in the formation of CA₆ crystals mainly located at the interface of coarse tabular alumina grains.

Additional to the method of adding spinels to the castables, the existence of liquid could accelerate the formation of CA₆ phases in the matrix and affect the morphology of CA₆ at elevated temperatures, which would ultimately influence the high temperature properties.⁽¹³⁾ For

[†] Corresponding author: X. Li; E-mail: lixiangcheng@wust.edu.cn

[‡] Corresponding author: P. Chen; E-mail: pinganchen@wust.edu.cn

example, CA₆ grains with equiaxial shape can be formed in the castables without microsilica. However, when microsilica was introduced to the castables, CA₆ phases did not take on equiaxial shape but rather a platy or needle-like shape.^{14)–16)} Braulio and Bittencourt¹⁴⁾ explained that the equiaxial CA₆ phases was generated by solid state reaction while the non-equiaxial CA₆ was synthesized by solution precipitation reaction with assistance of liquid at high temperature. Furthermore, Domí Nguéz and Chevalier et al.¹⁷⁾ investigated the effect of CA₆ morphology on the mechanical properties of castables and found that equiaxial CA₆ caused the planar cracks while platy CA₆ generated tortuous cracks. An and Chan¹⁸⁾ also revealed that platy CA₆ phases with non-equiaxial morphology possessed more pronounced R-curve behavior than the equiaxial CA₆ crystals. Besides the existence of liquid, the types of raw materials (calcium carbonate or calcium oxide) could also adjust the CA₆ grain morphology by in-situ reaction sintering.¹⁹⁾ additionally, the alumina particle size influences phase composition, microstructure and hot properties of the refractory castables.^{20)–22)} Therefore, the CA₆ morphology has an important influence on the high temperature mechanical properties of castables. Additionally, the CA₆ phase with non-equiaxial shapes such as platy structure can enhance the hot properties, creep resistance, and mechanical strength because of their toughening and reinforcement mechanism of deflection, shielding, and bridging for microcracks.

In this paper, the morphology of CA₆ phases in the castables was adjusted through the particle size of alumina powders and non-equiaxial CA₆ phases were obtained. The systematic experimental data, including the microstructure and formation mechanism of platelet-shaped CA₆ phases, room temperature physical properties, and thermal elastic modulus (EM) change during the thermal shock cycles were determined. It is believed that this research could be helpful to understand the relationship between properties and CA₆ microstructure. Moreover, based on the experimental data, the improvement mechanism of the comprehensive performance of the refractory castables could also be revealed.

2. Experimental

2.1 Preparation of corundum castable specimens

The refractory castable compositions contained tabular alumina ($d \leq 5$ mm, Almatiss, Qingdao, China) as aggregates and 5 wt.% CAC (Secar71, Kerneos, Tianjing, China) as the binder. The photo of tabular alumina can be seen in Fig. 1. Three different types of micro alumina powders (denoted as A, B and C) were used as filler, and their particle size distributions were analyzed by laser particle size analyzer (Mastersizer 2000, Malvern Instruments Ltd., UK). The properties of the alumina powders are depicted in Table 1. The typical mean diameter (D_{50}) of A–C decreased from 7.26 to 2.37 μm while the specific surface area (SSA) is improved from 3.28 to 5.88 $\text{m}^2\cdot\text{g}^{-1}$. The content of micro alumina was 14% and the prepared

Fig. 1. Photo of tabular alumina.

Table 1. The properties of alumina powders

Samples	A	B	C
D_{50} (μm)	7.26	5.33	2.37
SSA ($\text{m}^2\cdot\text{g}^{-1}$)	3.28	4.73	5.88

castables were labeled as sample RA, RB and RC, which were corresponded to the specimen containing micro alumina powder of A, B and C, respectively.

The raw materials were dry-mixed for 5 min; and then, 4.8 wt % of water was added gradually. After being mixed for another 5 min, the mixture was vibro-casted into the specimens with a dimension of 40 mm $\times$ 40 mm $\times$ 160 mm rectangular blocks with the help of a table vibrator. All specimens were cured at room temperature for 24 h and were subsequently dried at 110°C for another 24 h. Finally, the specimens were fired at 1100 and 1600°C for 3 h in an air atmosphere at the heating rate of 5 °C min^{−1}.

2.2 Testing and characterization methods

The phase composition and microstructural characteristics of specimens were studied using X-ray diffraction (XRD, X'pert Pro MPD, Philips, Netherlands) and field emission scanning electron microscopy (SEM, JSM-6610, JEOL, Japan). The apparent porosity (AP) and bulk density (BD) of the sintered castable specimens was tested according to ASTM C0020-00R05 standard based on Archimedes' Principle. The permanent linear change (PLC) was calculated according to the length change of specimens before and after firing. Mechanical properties including cold modulus of rupture (CMOR, fracture strength at room temperature) and EM were measured using the three-point bending test at ambient temperature according to ASTM C-1161 standard. The measurement was conducted using an electronic digital control system (EDC 120, DOLI Company, Germany). The force span was 100 mm and the loading rate was 0.5 mm·min^{−1}. The force-displacement curve of the fired specimens was simultaneously recorded during the three-pointing bending test. The hot modulus of rupture (HMOR) of the specimens was measured in air using the three-point bending test method based on ASTM C583-8 Standard. The test temperature was 1400°C, and the soaking time was 0.5 h.

The dynamic pulse excitation method was used to determine the resonance frequencies of the specimens (RFDA-HTVP 1600, IMCE, Belgium) according to ASTM C1198

standard. The thermal modulus of elasticity was calculated using the following equation.^{23),24)}

$$E_t = 0.9465 \times \frac{mf^2}{b} \times \frac{L^3}{d^3} \times k \quad (1)$$

where f , m , L , b , d , and k are the resonance frequencies, mass, length, width, thickness of the specimens and correction factor, respectively. The test was conducted with the temperature range of 30–1400°C in air ($p_{O_2} = 0.21$ atm) with heating and cooling rates of 3 °C·min⁻¹.

The thermal shock resistance of the specimens fired at 1600°C was performed in air using the water quenching method according to ASTM C1171-16. The specimens were heated at 1100°C for 30 min and put into water at room temperature for 5 min. The specimens were dried, and their residual CMOR_{st} was determined by the three-point bending test after three thermal-shock cycles.

3. Results and discussion

3.1 Physical properties of corundum refractory castables with different alumina powders

The effect of particle size of micro alumina on the AP and BD of refractory castables is presented in **Table 2**. With the particle size of alumina powders decreasing from 7.26 to 2.37 μm, all the AP values are decreased by about 3% and the BD values increased at 110, 1100 and 1600°C.

Table 2 also shows the PLC values of refractory castables with different particle sizes of alumina powders fired at 1100 and 1600°C. At 1100°C, the PLC of three kinds of castables is almost 0.25%. When the temperature rises to 1600°C, the PLC values of all specimens are increased, and the different PLC values appear. The PLC value decreased from 0.55 to 0.3% with decreasing particle size of alumina powders from 7.26 to 2.37 μm. This and the AP and BD results show that the decreasing particle size of alumina powders do not affect AP and BD but improve the volume stability of castables according to PLC variations.

Mechanical properties of CMOR for refractory castables containing three kinds of micro alumina powders are also depicted in Table 2. The CMOR values increased with increasing firing temperature. This is due to the sintering and densification process. It is noteworthy that the fired temperature has greater influence on the CMOR of refractory castables with small particle size of alumina. When the heating temperature is increased from 110 to 1600°C,

the CMOR of specimen RA increases from 2.58 to 11.48 MPa, while the value of specimen RC increases from 3.78 to 27.77 MPa. The increasing amplitude is about 400 to 700%. Although the values of AP, BD and PLC for specimen RB and RC in Table 2 are similar after firing at the same temperature, the particle size of alumina influences the mechanical properties of castables. At the same temperature of 1600°C, the CMOR value increased by 142% as the particle size of alumina is decreased from 7.26 to 2.37 μm, as shown in Table 2.

The force-displacement curves of three kinds of castable specimens fired at 1600°C are illustrated in **Fig. 2**. Both the maximum values of force and displacement increased when the alumina particle sizes decreased from 7.26 to 2.37 μm. The maximum force increased from 1.3 kN(RA) to 2.9 kN(RC); this increase in amplitude is more than 2.2-fold. The maximum displacements for RA-RC also increase from 0.61 to 1.2 mm, which indicates that the toughness of castables is also enhanced by decreasing particle sizes. The force-displacement change of the specimens RA-RC is in agreement with that of CMOR, which proves that the reduction of alumina particle size to 2.37 μm (D50) plays a key role in the strength and toughness improvement.

To further evaluate the performance of refractory castables with different particle size of alumina powders at high temperature, the HMOR and CMOR_{st} of castables after fired at 1600°C are evaluated. The results, shown in **Fig. 3**, show that both HMOR and CMOR_{st} (after three thermal cycles) significantly increased with alumina parti-

Fig. 2. Force-displacement curves of castable specimens treated at 1600°C.

Table 2. Physical properties of refractory castables heated at different temperatures

	RA	RB	RC	RA	RB	RC
	Apparent porosity (%)			Bulk density (g/cm ³)		
110°C	18.6 ± 0.93	17.5 ± 0.88	15.7 ± 0.79	2.97 ± 0.15	3.03 ± 0.15	3.08 ± 0.15
1100°C	21.6 ± 1.08	18.2 ± 0.91	18.2 ± 0.91	2.91 ± 0.14	3.01 ± 0.15	3.04 ± 0.14
1600°C	22.6 ± 1.13	19.3 ± 0.97	19.3 ± 0.97	2.92 ± 0.15	3.01 ± 0.15	3.05 ± 0.16
	Permanent linear change (%)			Cold modulus of rupture (MPa)		
110°C	—	—	—	2.58 ± 0.13	2.98 ± 0.15	3.78 ± 0.19
1100°C	0.25 ± 0.013	0.25 ± 0.013	0.21 ± 0.01	4.65 ± 0.23	6.41 ± 0.32	8.57 ± 0.43
1600°C	0.55 ± 0.028	0.37 ± 0.019	0.30 ± 0.015	11.48 ± 0.57	17.24 ± 0.86	27.77 ± 1.39

Fig. 3. Thermal performances of RA-RC specimens treated at 1600°C: (a) HMOR at 1400°C; (b) CMOR_{st}.

Fig. 4. Thermal EM evolution of specimens RA-RC as a function of the temperature (loop area of three specimens is 8006, 9685 and 9102 respectively).

cle size decreasing. The HMOR values increased from 10.45 to 25.37 MPa and the values of CMOR_{st} increased from 1.95 to 5.70 MPa when alumina particle sizes decreased from 7.26 to 2.37 μm . The magnitudes of HMOR and CMOR_{st} increased by 143 and 192%, respectively. The toughness of refractory castables is the ability to resist thermal stress, which could be denoted by CMOR_{st}. Therefore, this represents that the finer alumina powders in the refractory castables could greatly improve the hot strength and toughness of castables. The results are consistent with the force-displacement curves.

Thermal EM at high temperature can be used to evaluate the thermo-mechanical behavior of refractory castables during the servicing process. Therefore, the thermal elastic moduli of specimens RA-RC fired at 1600°C were measured from 30 to 1400°C (Fig. 4). All evaluated specimens show similar variation trends under the same testing condition. During the heating process, specimens present a decrease in EM from room temperature to 800°C. The reduction range is about 10 GPa. Then a small increase is shown at 1400°C. During the cooling period, the thermal EM markedly rises when the temperature decreases from 1400 to 300°C. Finally, the EM behaves a slow decrease as the temperature decreases from 300°C to room temper-

ature. Therefore, the finer the adding alumina particle, the higher the EM values. At the same temperature, there is always an approximately 50 GPa increment in the thermal EM for RA and RC specimens.

It is noticeable from three curves in Fig. 4 that the EM during the cooling stage is higher than that of the heating period, which was also reported by Huger and Pandolfelli.^{9),23)} After the heating period, the specimen density would increase because of the sintering accompanied by the phenomena of crystal growth, decreasing microcracks, and strong bonding interface. All these microstructural changes during elevated temperature lead to the high EM in the cooling stage. The other point in Fig. 4 that should be highlighted is that the loop area of thermal EM for specimen RA-RC differs, which could also denote the variation amplitude of thermal EM during the thermal cycles.²⁵⁾ The detailed value could be obtained by integrating the loop curves shown in Fig. 4. The loop areas of specimen RA, RB and RC are 8006, 9685 and 9102 respectively. This means that the fine alumina powders contribute to the microstructure optimization in the aspects of microcrack, densification and bonding, which ultimately lead to the increase in EM and tolerance to the thermal shock [Fig. 3(b)].

3.2 Phase and microstructure evolution of corundum refractory castables with different alumina powders

To understand the mechanism of mechanical behavior improvement of refractory castables, the phase and microstructure evolution of the specimen RA-RC are investigated. The main phase composition in calcium aluminate is mono-aluminate of calcium (CA) and calcium dialuminate (CA₂) is the minor phase. Usually, CA phases in CAC reacts with alumina (A) to form CA₂ in the temperature range of 1000–1200°C. When the temperature rises, the alumina further reacts with calcium-aluminate and the new-born CA₂ phase to generate CaO·6Al₂O₃ crystals at temperatures above 1450°C.^{8),16),26)} As Auvray⁹⁾ illustrated, the reactions can be expressed by the following equations,

The specimens fired above 1600°C mainly comprise corundum and CA_6 . The phase diagram of $\text{CaO-Al}_2\text{O}_3$ ¹⁹⁾ suggests that there is no low melting phase formation and the formation of CA_6 could increase the slag-resistance of castables to above 1700°C.

The XRD patterns of specimen RC after firing at 1100 and 1600°C are depicted in **Fig. 5**. The $\text{CaO} \cdot 2\text{Al}_2\text{O}_3$ (CA_2) and corundum phase are found in the specimen after being fired at 1100°C. Compared with reactions 2 and 3 and the phase diagram of $\text{CaO-Al}_2\text{O}_3$, the PLC variation of specimens fired at 1100°C (depicted in Table 2) is attributed to

Fig. 5. XRD patterns of specimen RC fired at 1100 and 1600°C for 3 h.

the formation of CA_2 . The reduction of PLC at 1600°C from 0.55 to 0.30% can be explained as follows. On the one hand, the formation of CA_6 causes the expansion of refractory castables; on the other hand, the densification can be accelerated in the sintering process because of the high surface area (as shown in Table 1) with decreasing particle size,^{27),28)} which indicates that high volume stability of refractory castables can be obtained by adding fine alumina particles (Table 2).

The microstructures of refractory castables containing different particle sizes of alumina powders fired at 1600°C are presented in **Fig. 6**. The formed CA_6 is interlinked with the alumina aggregates and powders in the matrix. With decreasing particle size of alumina powders, the microstructure between aggregate and matrix tends to be densely packed, as shown in Figs. 6(a), 6(c) and 6(e). In detail, there are significant differences for the morphology of CA_6 formed in matrixes. As shown in Fig. 6(b), the CA_6 grains take on a round shape. However, the amount of CA_6 grains is greater and the morphology becomes hexagonal plate-like [Fig. 6(d)]. As shown in Fig. 6(f), the amount of CA_6 with non-equiaxial shape reaches the maximum in the matrix. Notably, this non-equiaxial shape morphology is believed to improve physical and chemical bonding strength between particles, which ultimately enhances room temperature and high temperature properties, as seen in Table 2 and Figs. 2–4. Therefore, combined with the phase diagram of $\text{Al}_2\text{O}_3\text{-CaO}$, XRD in Fig. 5 and SEM in Fig. 6, it is deduced that CA_6 with non-equiaxial shape of platelet could be formed in this system

Fig. 6. SEM images of refractory castables containing different alumina powders fired at 1600°C: (a, b) RA, (c, d) RB, (e, f) RC.

Fig. 7. SEM images of fracture surfaces of the specimen fired at 1600°C: intergranular fracture of RA (a) and transgranular fracture of RC (b, c).

without any liquid phase, which subsequently enhance the comprehensive performances of castables.

Based on the physical properties of castable specimens in Table 2, there is little effect of alumina particle sizes on the AP and BD when the fired temperature is the same. However, their CMOR values differ greatly (Table 2 and Figs. 2–4). This phenomenon is not due to the porosity but the morphology of CA₆ depicted in Fig. 6. Because of the formation of irregular platy CA₆, the ceramic bonding strength between particles is significantly enhanced. The most powerful evidence is the fracture mechanism transformation from intergranular fractures [Fig. 7(a)] to transgranular fractures [Fig. 7(b)]. The strong interlocking structure between the CA₆ and alumina grains contributes to the high bonding strength such as high CMOR, HMOR, and thermal EM (Table 2 and Fig. 2), which is consistent with the results of Dudezig,²⁶⁾ Chan²⁹⁾ and Ko.³⁰⁾ The plate-like shape of CA₆ crystals shown in Fig. 6(f) acts as in-situ toughness phases.³¹⁾ As previously mentioned, the thermal-shock resistance of specimen RC fired at 1600°C is significantly improved [Fig. 3(b)], which can be attributed to the bridging mechanisms of the platelet shape CA₆ phases.^{17),32)}

According to the above results and analysis, the morphology of platelet shaped CA₆ grains plays a vital role in improving the thermo-mechanical properties of refractory castables. The morphology of CA₆ grains is thought to be controlled by the coupled diffusion of Ca²⁺ ions and O²⁻ ions from calcium-rich phases to alumina-rich phases.³³⁾ The growth model of platelet CA₆ grains from differently sized powders is schematically depicted in Fig. 8. During the early stages of sintering from 1100 to 1250°C, CA₂ is formed from the reaction of CA-alumina. When the temperature rises to about 1450°C, the CA₆ nucleation process is controlled by solid state diffusion at the interfaces of CA₂-alumina. Owing to the difference in contact areas of alumina-CA₂, the number of CA₆ nuclei formed per unit volume varies with the particle size of alumina. When the

Fig. 8. Schematic representation of different mechanisms for the reaction of CA₂ and Al₂O₃ to form CA₆ with particle and platelet shapes: (a) coarse alumina powder, (b) fine alumina powder.

reaction is proceeding, CA₆ grains grow along their basal plane; this plane is oriented perpendicularly to the reaction surface. This growth process continues until the platelet grains impinge on each other. When the alumina particle size is large, the closely-packed particles are loose, so the contact area is smaller, so CA₆ grains develop with a high aspect ratio. The large number of CA₆ nuclei induces greater CA₆ grain growth. Similar results have been reported,³⁴⁾ in which the crystal size of CA₆ with fine raw materials was finer than that of the sample with coarse raw materials because of the differences of CA₆ nuclei numbers. However, when the alumina particle size is small, the high contact areas between CA-alumina lead to the growth of CA₆ with a low aspect ratio. Additionally, the amount of CA₆ grains decreases, as seen in Fig. 6(b). Moreover, because the particle size directly affects the driving force and the reaction rate of grain growth, more plate-like grains in specimen RC can be generated from smaller particle sizes. Song³⁵⁾ also observed a similar behavior during the growth of plate-like alumina grains with different starting crystallite sizes. Therefore, the fine alumina powders with large surface area are helpful to form

platelet-shaped CA₆ grains in refractory castables at elevated temperatures.

4. Conclusions

1) When the particle sizes of micro alumina powders decreased from 7.56 to 2.37 μm , the cold modulus of rupture and HMOR of the refractory castables fired at 1600°C increased by 142 and 143%, respectively. The thermal-shock resistance increased by approximately 192%.

2) The improvement of thermo-mechanical properties was attributed to the formation of platelet-shaped CA₆ phases. The platy CA₆ tended to form stronger bonding linkages with alumina grains than did equiaxial CA₆.

3) When the alumina particle size was fine, the low contact areas of alumina-CA₂ and the large number of CA₆ nuclei enabled the growth of plate-like CA₆ grains.

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