



Microstructure and mechanical properties of ceramics prepared under simulated oxygen-enriched or oxy-fuel atmosphere

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Abstract

Different simulated sintering atmospheres, such as conventional air, oxygen-enriched air, and oxy-fuel atmosphere were utilized to prepare ceramic specimens. The effects of sintering atmosphere and Al_2O_3 content on the microstructure and properties of the ceramic samples were investigated. Increasing the O_2 concentration in the simulated sintering atmosphere enhanced the crystallization. An optimized microstructure, resulting in improved bending strength, Vickers hardness, and bulk density was obtained through an increased O_2 concentration in the simulated sintering atmosphere. Compared to ambient air, oxygen-enriched air or oxy-fuel atmosphere showed superior sintering performances, leading to ceramics with better mechanical properties and denser microstructures. However, an increase in the Al_2O_3 content led to the degradation in the mechanical properties of the ceramics, mainly due to the blocking effect of AlO_6 groups.

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1. Introduction

The carbon dioxide-induced climatic changes observed in recent years highlight the necessity to reduce CO_2 emissions from fossil fuel combustion [1]. Oxy-fuel combustion [2,3] and oxygen-enriched combustion [4–7] can greatly reduce the energy consumption and environmental pollution, and thus represent particularly useful technologies for combustion systems. Oxygen-enriched combustion, using an oxidizer with O_2 concentrations higher than 20.93%, is one of the most useful energy-saving and environmental-protecting combustion technologies [8–10]. Oxy-fuel combustion is the removal of nitrogen from the oxidizer to carry out the combustion process in oxygen [11]. The advantages attributed to oxygen-enriched or oxy-fuel combustion include increased productivity, energy efficiency, turndown ratio, and flame stability, with reduced exhaust gas volume and pollutant emissions. The oxygen-enriched or oxy-fuel technology is also beneficial for

the capture of CO_2 and SO_x , with a consequent reduction in pollutant emissions [12]. Generally, oxygen-enriched combustion is applied in high temperature processes, for example, metal melting, ceramic sintering, incinerators, glass-melting furnaces, and ladle furnaces [13,14].

The traditional ceramic industry is typically associated with high production, high energy consumption, low energy efficiency, and high levels of pollution. Another problem is represented by the production of large amounts of lower-grade ceramics. All these issues seriously limit the sustainable development of ceramic industry [15]. Technologies like oxygen-enriched and oxy-fuel combustion have been developed to solve these problems [4,11,16]. Up to now, research on these technologies has mostly focused on numerical simulations of the temperature distribution in the kiln, environmental problems, thermal characteristics, and so on [17–21]. Substantial fuel savings were also reported by Kimura and Browall [22]. However, when oxygen-enriched or oxy-fuel combustion is employed in ceramic sintering, the fume gas composition in the furnace atmosphere may also change, which in turn affects the sintering properties of the ceramics. A key question is then whether the utilization of

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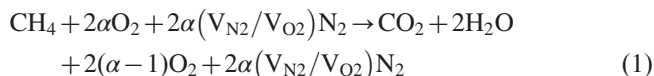
oxygen-enriched or oxy-fuel technology can also affect the structure and mechanical properties of ceramics, and then the final products. An investigation on the microstructure and mechanical properties of ceramics sintered in an oxygen-enriched or oxy-fuel atmosphere would thus be highly valuable.

In this study, the actual micro-environment of a ceramic kiln was simulated by an atmosphere oven, and the ceramics were exposed to different atmospheres. Phase microstructure and mechanical properties of the ceramics were investigated. The obtained results are meaningful for supporting practical applications of the oxygen-enriched combustion technology in ceramic manufacturing. The large-scale application of oxygen-enriched or oxy-fuel sintering in the ceramic industry could allow recycling of large amounts of CO₂ and SO_x, as well as increasing the qualification rate of the sintered ceramics, also with substantial energy savings.

2. Experiments

2.1. Simulation of different sintering atmospheres

Natural gas is currently the most widely used fuel in the ceramic industry. According to equation (1) [23], the combustion products of natural gas are CO₂, H₂O, and N₂. In this study, oxygen-enriched air with 25 vol. % O₂ and oxy-fuel atmosphere with 100 vol. % O₂ were simulated by changing the volume proportion of CO₂, H₂O and N₂. In the process of ceramic sintering, the concentration of CO₂, H₂O and N₂ were measured with a MGA5 infrared gas analyzer.



In Eq. (1), α is the air excess coefficient, while V_{N_2} and V_{O_2} are the volume of N₂ and O₂, respectively. α ranges from 1 to 1.2 for the oxygen-enriched combustion, and, was set to 1 in the present work.

2.2. Preparation of ceramics

K₂O-Al₂O₃-SiO₂ ceramics, with nominal chemical compositions tabulated in Table 1, were prepared by liquid sintering method. The compression molding technique was utilized to prepare specimens by pressing powdery materials, previously ball-milled for 5 h and screened through a 200-mesh screen. The samples were sintered in a controlled atmosphere furnace using simulated conventional air, oxygen-enriched air with 25 vol. % O₂, and oxy-fuel atmosphere of 100 vol. % O₂,

Table 1
Chemical Composition of the Ceramics (wt. %).

Composition (wt. %)	SiO ₂	Al ₂ O ₃	K ₂ O	Na ₂ O	MgO	CaO	TiO ₂	Fe ₂ O ₃
A ₁	68.80	19.50	2.29	0.96	0.89	0.72	0.43	0.41
A ₂	67.38	20.92	2.29	0.96	0.89	0.72	0.43	0.41
A ₃	65.50	22.80	2.29	0.96	0.89	0.72	0.43	0.41
A ₄	64.00	24.30	2.29	0.96	0.89	0.72	0.43	0.41

respectively. The different sintering atmospheres are detailed in Table 2. In order to prepare the ceramics specimens, green compacts were sintered with different atmospheres at a maximum of 1180 °C for 1h.

2.3. Ceramic characterizations

X-ray diffraction (Rigaku D/MAX-RB) was employed to identify the crystals generated in the powdery samples, after grinding and screening through a 200-mesh sieve. The microstructure of samples etched in HF solution (5 vol. %) for 45 s was examined by a JSM 5610LV microscope operating at 25 kV. IR analysis of powdery specimens was carried out using a Nexus FT-IR. The bending strength of 40 × 5 × 5 mm rectangular samples was measured by the three-point bending method using an AG-IC universal testing machine. Measurements of Vickers hardness were acquired by a HVS-1000 type micro-hardness tester. Bulk density was determined by the Archimedes method refers to GB/T 3810.3- 2006.

3. Results and discussion

3.1. Effects of Al₂O₃ content on microstructure and mechanical properties

Fig. 1 shows the XRD profiles of ceramics with Al₂O₃ content ranging from 19.50 to 24.30 wt. %. All the XRD profiles were identical, indicating the presence of similar crystalline phases. Mullite and quartz were indexed. Fig. 1 denotes the complex effect of the Al₂O₃ content on the relative amounts of the crystalline phases. An increase in Al₂O₃ content favors the formation of primary mullite. However, the crystallization of secondary mullite that precipitates at a higher temperature than primary mullite is hindered, as the sintering temperature of the ceramic sample also increases for higher Al₂O₃ contents. SEM micrographs of A-series ceramics with different Al₂O₃ content are shown in Fig. 2. Grainy premullite and Quartz, and fine acicular secondary mullite precipitated from the glass phase were observed. The number and size of crystals seemed to increase for higher Al₂O₃ contents. Fig. 3 displays the IR spectra of ceramic specimens with different Al₂O₃ content. Cheng et al. previously found that a single band at 740 cm⁻¹ is observed when only AlO₄ groups are present, but 700 cm⁻¹ and 760 cm⁻¹ bands form when both AlO₄ and AlO₆ species coexist in the ceramic structure [24]. The bands at 695 cm⁻¹ and 779 cm⁻¹ in Fig. 3 are assigned to Al-O stretching vibrations, and denote the coexistence of AlO₄ and AlO₆ groups. The bands observed at 460 cm⁻¹, 779 cm⁻¹ and 800 cm⁻¹, 1090 cm⁻¹ are associated with Si-O bending, O-Si-O symmetric

Table 2
Simulated sintering atmospheres.

Experimental	B ₁	B ₂	B ₃
Atmosphere	Air	Oxygen-enriched	Oxy-fuel
O ₂ concentration	21 vol.%	25 vol.%	100 vol.%
CO ₂ :H ₂ O:N ₂	1:2:7.52	1:2:6	1:2:0

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