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Crystallisation behaviour of blast furnace slag modified by adding fly ash

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ABSTRACT

Crystallisation of molten blast furnace (BF) slag can increase its viscosity, which can in turn affect the quality of slag fibres. Fly ash was added to BF slag to control its crystallisation and modify its chemical composition. FactSage simulation and analyses using X-ray diffraction (XRD), scanning electron microscope-backscattered electrons (SEM-BSE) coupled to an energy dispersive spectrometer (EDS), and single hot thermocouple technique (SHTT) were performed to explore the crystallisation behaviour of the modified BF slag. The relationship between temperature, mineral precipitation, and added fly ash content was investigated. The minerals contained in the modified BF were melilite, anorthite, clinopyroxene, and spinel. Variation in the fly ash content did not change the composition of the precipitate, but changed its content and the crystallisation temperature of the minerals, which affects the initial crystallisation temperature of the modified BF slag. It decreased as fly ash content increased, and was influenced by the crystallisation of melilite when the added fly ash content was between 5% and 20%. When the added fly ash content increased to 25%, the initial crystallisation temperature was influenced by the precipitation of anorthite. The initial crystallisation temperatures obtained by FactSage simulation, XRD analysis, and SHTT experiments differed due to kinetic effects. The modified BF slag with a fly ash content of 15% is considered suitable for manufacturing of slag fibres due to its low initial crystallisation temperature and cost.

1. Introduction

Blast furnace (BF) slag is a by-product of ironmaking processes, and approximately 220 million tons are generated in China annually [1–3]. Currently, most BF slag is used for low-added value products, such as cement manufacture, civil engineering materials, roadbed materials, and concrete admixture as it has good hydraulic activity and high calcium silicate content [4–6]. BF slag is discharged at temperatures ranging from 1450° to 1650°C and contains a large amount of latent heat, which cannot be utilized well when the slag is recycled into low-added value products. To build an environmentally and economically sustainable steel factory, new methods of utilising both BF slag and its latent heat are receiving increasing attention, such as slag fibre manufacture [7–9].

The production of slag fibre from molten BF slag can effectively utilize the latent heat and produce high-added value products [10,11]. However, during the cooling process, the initial crystallisation temperature of BF slag is relatively high, which leads to the inclusion of microcrystals in some fibres. The crystals will break the slag fibre, shorten the length and lower the strength, which affect the quality of the slag fibre. The crystallisation behaviour of BF slag is especially

significant, and some researchers have studied how its chemical composition influences it. According to Qin et al. [12], an increase in MgO content and ${\rm CaO/SiO_2}$ ratio is conducive to crystallisation, while an increase in ${\rm Al_2O_3}$ content is unfavourable for crystallisation. Esfahani et al. [13] investigated the effect of composition on the crystallisation of synthetic ${\rm CaO-SiO_2-Al_2O_3-MgO}$ slags. They showed that, with increasing basicity, the time temperature transformation (TTT) and continuous cooling transformation (CCT) diagrams of glassy to crystalline transformation shift to the left (shorter time) and higher temperatures. It can be concluded that the crystallisation behaviour of BF slag can be controlled by modifying its chemical composition.

Fly ash is one of the main solid wastes discharged from coal-fired power plants, and is mainly composed of SiO_2 and Al_2O_3 . The key aspect of slag fibre manufacture is that the crystallisation of BF slag should be inhibited to generate more amorphous phases during cooling. Fly ash may be a good additive for modifying BF slag as it has high SiO_2 and Al_2O_3 content [14]. Li et al. [11] demonstrated that the preparation of high-quality slag fibres using BF slag with fly ash as an additive is feasible. A similar result was obtained by Lin et al. [15]. BF slag modified with fly ash (ranging from 15.5% to 37.46%) is suitable for the manufacture of slag fibres. However, few reports have explored the

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Table 1
Chemical compositions of the blast furnace slag and fly ash (wt%).

Component	SiO_2	CaO	MgO	Al_2O_3	Fe_2O_3	${ m TiO_2}$	K_2O	Na_2O	MnO	S
Blast furnace slag	33.53	36.25	8.64	15.82	1.57	1.38	0.54	0.32	0.17	0.84
Fly ash	51.87	3.37	0.011	33.34	3.6	0.82	0.78	0.1	0.044	0.10

crystallisation behaviour of BF slag modified by the addition of fly ash.

This study aimed to explore the crystallisation behaviour of BF slag

This study aimed to explore the crystallisation behaviour of BF slag with added fly ash. The relationship between the fly ash content, temperature, and precipitation phases was established using FactSage simulation, and the results of this were used to determine the initial temperature-controlled crystallisation phase. To verify the validity of FactSage simulation and analyses using X-ray diffraction (XRD), a scanning electron microscope-backscattered electrons (SEM-BSE) coupled to an energy dispersive spectrometer (EDS), and the single hot thermocouple technique (SHTT) were conducted.

2. Materials and methods

2.1. Materials

The BF slag was obtained from a steel plant, and the fly ash was obtained from the boiler of a thermal power plant in China. The chemical compositions of the BF slag and fly ash were determined following the chemical analysis method YB/T140–2009. As shown in Table 1, the major chemical components of the BF slag used in this work were SiO₂, CaO, Al₂O₃, and MgO. The measured S is sulphur in the form of sulphide, which was present in the BF slag. The chemical components of the fly ash used in this work were mainly SiO₂ and Al₂O₃. The modified BF slag was prepared by mixing the BF slag with the fly ash. The chemical composition of the modified BF slag is shown in Table 2.

2.2. Sample preparation and mineralogical measurements

One hundred grams of modified BF slag was placed in an alumina crucible and melted using an experimental tube furnace. The melting temperature was increased from 25° to 1500 °C in 250 min, and held at 1500 °C for 1 h to completely homogenize the slag. The melting experiments were conducted under an air atmosphere. The melted modified BF slag was then cooled slowly to the specified temperature at a rate of $10\,^{\circ}\mathrm{C\,min}^{-1}$ in the experimental tube furnace. Following the slow cooling process, the samples were then quenched in water to determine their high-temperature mineralogical composition. The cooled samples with grain sizes below 74 μm were measured using XRD and SEM-BSE coupled to EDS. The XRD analysis was conducted using Cu-K α radiation at 40 kV and 80 mA, with a 2θ range of 10–80° and scanning velocity of $10\,^{\circ}/\mathrm{min}$.

2.3. Thermodynamic simulation

Thermodynamic simulation was conducted using FactSage 7.0 software, which provides theoretical support for the complex

 Table 2

 Main chemical components of the modified blast furnace slag.

Mass ratio (wt%)	Content of component (wt%)					
Blast furnace slag	Fly ash	SiO_2	CaO	MgO	Al ₂ O ₃	
95	5	34.45	34.61	8.21	16.70	
90	10	35.36	32.96	7.78	17.57	
85	15	36.28	31.32	7.35	18.45	
80	20	37.20	29.67	6.91	19.32	
75	25	38.12	28.03	6.48	20.20	
	Blast furnace slag 95 90 85 80	Blast furnace slag Fly ash 95 5 90 10 85 15 80 20	Blast furnace slag Fly ash SiO ₂ 95 5 34.45 90 10 35.36 85 15 36.28 80 20 37.20	Blast furnace slag Fly ash SiO ₂ CaO 95 5 34.45 34.61 90 10 35.36 32.96 85 15 36.28 31.32 80 20 37.20 29.67	Blast furnace slag Fly ash SiO ₂ CaO MgO 95 5 34.45 34.61 8.21 90 10 35.36 32.96 7.78 85 15 36.28 31.32 7.35 80 20 37.20 29.67 6.91	

calculation and simulation of thermodynamic processes. The initial chemical components of the modified BF slag were selected based on the results shown in Table 2. The Scheil-Gulliver cooling approach, which is included in the Equilib module of FactSage, was used to establish the relationship between the added fly ash content, temperature, and mineral precipitation. The cooling temperatures used in this simulation ranged from 1000° to 1500°C. The FToxid database was used in this simulation.

2.4. Single hot thermocouple experiments

SHTT is an experimental procedure that explores the mineral precipitation of modified BF slag during cooling. A hot thermocouple welded into a U shape was used to simultaneously heat the samples and measure their temperature, and a microscope equipped with a video camera was used to capture images. A small amount of the powdery modified BF slag was placed on top of the thermocouple with ethanol. The sample was heated from 200° to 1500°C in 260 s and maintained at 1500°C for 60 s to eliminate any existing bubbles. The melted slag was cooled to a specific temperature at a rate of 10°C/s. Isothermal experiments were then conducted at this temperature over a period of 1000 s to observe the crystallisation behaviour of the modified BF slag. The temperature profiles are shown in Fig. 1.

3. Results and discussion

3.1. FactSage simulation of crystallisation behaviour

Fig. 2 shows the relationship between temperature, mineral precipitation, and added fly ash content. As shown in Fig. 2(a), for the modified BF slag with 5% fly ash, the quantity of liquid slag decreased gradually as the temperature decreased. As the temperature decreased to $1410.1\,^{\circ}$ C, the solid-phase melilite was crystallised first. The initial crystallisation temperatures of other phases, such as anorthite, clinopyroxene, and spinel, were 1267.2, 1246.0, and $1272.5\,^{\circ}$ C, respectively. The liquid slag was completely transformed into the solid phase at $1245.9\,^{\circ}$ C. At this temperature, the mass ratios of melilite, anorthite, clinopyroxene, and spinel were 77.5%, 7.5%, 14.0%, and 1.0%, respectively. These mass ratios were maintained as the temperature continued to decline.

As shown in Fig. 2(b)-(e), when the added fly ash content increased

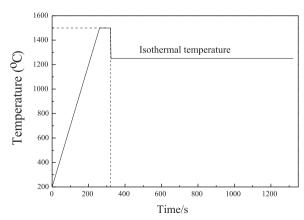


Fig. 1. Temperature profiles of the SHTT experimental conditions.

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