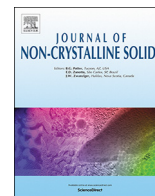




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Crystallization behavior of amorphous slag beads prepared by gas quenching of blast furnace slag

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ABSTRACT

Slag beads with different amorphous phase contents can be prepared by gas quenching of blast furnace slag (BFS). The amorphous phase content can affect the activity of these slag beads, with higher amorphous phase contents leading to higher activity. In this study, thermodynamic simulations with FactSage 7.1 and differential scanning calorimetry were used to investigate the crystallization behavior of BFS during continuous cooling. The influence of different cooling rates and basicities on the crystallization behavior of BFS was studied, and the activation energy for crystallization was calculated by the Kissinger and Ozawa methods. The results showed that the precipitation of crystals could be prevented at high cooling rates and low BFS basicity. Accordingly, no precipitation of BFS crystals was observed when basicity was less than 0.91 and the cooling rate was above 50 °C/min. In addition, the activation energy for crystallization of BFS decreased with increasing basicity

1. Introduction¹

Blast furnace slag (BFS) is the main byproduct of iron manufacturing, with approximately 300–350 kg of BFS produced per ton of iron. Traditional water quenching process wastes a considerable amount of water and pollute the environment, and, the resulting hot water is a low-quality heat source [1–3] that cannot be effectively recovered or recycled; additionally, the utility of the treated water slag is low. Dry granulation, on the other hand, can effectively recover sensible heat from BFS, without consuming large amounts of water nor producing harmful gases that pollute the environment. Further, the BFS obtained by granulation has high utility [4–6]. Gas quenching of BFS is a dry granulation process, where in liquid molten slag is granulated using a high-speed air jet and heat transfer between the slag particles and air is used to recover the sensible heat of BFS [7–9].

Gas quenching of BFS is primarily a process for cooling the BFS. Different injection flow rates produce different cooling rates, which in turn influence the BFS crystallization behavior. The precipitation of BFS crystals reduces the utility of BFS beads. Therefore, BFS crystallization should be avoided as much as possible. Thus, considerable research has been conducted on the crystallization mechanism in glass ceramics prepared by BFS and the crystallization behavior of BFS; however, research on the crystallization kinetics of BFS granulation by gas quenching has been scarce. Gan et al. [10] studied the influence of

different cooling rates on the BFS crystallization behavior at four different positions on a sample using differential scanning calorimetry (DSC). It was found that the cooling rate was the fastest at the surface, and that no crystal precipitation occurred at this position, while, the cooling rate was slowest at the center of the sample wherein complete precipitation of the crystals occurred. Wang et al. [11] studied the crystallization kinetics of the parent glass when Cr₂O₃ and Fe₂O₃ were added as nucleating agents, and found that when Cr₂O₃ was added, the stability of the parent glass weakened, the activation energy for crystallization decreased, and the crystal growth index increased. Because of the characteristics of BFS, fibers could easily form during gas quenching [12], so BFS was modified and the effect of different BFS basicities on its crystallization behavior was studied in this study.

The crystallization behavior of BFS was simulated by the thermodynamic software FactSage 7.1, and the crystallization temperatures of the initial phase and mineral phases of BFS with different basicities were studied. Further, the activation energies for crystallization with different basicities were obtained by DSC kinetics experiments. The influence of different cooling rates on the crystallization behavior was also studied, and the modifying direction and critical cooling rate of BFS slag beads during gas quenching were confirmed, thus providing a theoretical basis for the preparation BFS beads with high amorphous contents.

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E-mail address: kang-kai-yue@163.com (Y. Kang).¹ BFS: blast furnace slag, DSC: differential scanning calorimetry, JMA: Johnson–Mehl–Avrami, XRD: X-ray diffraction<https://doi.org/10.1016/j.jnoncrysol.2018.08.034>

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Table 1

The chemical composition of BFS and modifying agents(wt%).

Composition	SiO ₂	CaO	MgO	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	K ₂ O	Na ₂ O	MnO
BFS	33.53	36.25	8.64	15.82	1.57	1.38	0.54	0.32	0.17
Iron ore tailing	67.32	1.82	2.66	12.48	0.36	4.59	2.54	–	–
Limestone	2.08	52.04	2.85	1.22	–	–	–	–	–

Table 2

The content of tempering agent and BFS under each basicity.

Basicity (R)	Mass ratio (wt%)		
	BFS	Iron ore tailing	Limestone
0.7	83.0	17.0	–
0.8	91.6	8.4	–
0.91	100.0	–	–
1.0	92.0	–	8.0
1.1	84.5	–	15.5

Tips: R is defined as the mass ratio of the main basic oxides to the main acidic oxides in the BFS using the formula $R = \frac{\omega_{CaO} + \omega_{MgO}}{\omega_{SiO_2} + \omega_{Al_2O_3}}$.

2. Materials and methods

2.1. Materials

BFS was obtained from the #3 blast furnace (2500 m³) of the HBIS iron manufacturing plant of Tangsteel in Tangshan, China. The modifying agents, iron ore tailings and limestone, were also obtained from the HBIS iron plant in Tangshan, China. The chemical compositions of the BFS and modifying agents, as determined by the Chinese industry standard YB/T140–2009, were summarized in Table 1. Modified BFS was prepared by mixing BFS with the modifying agent. The modifying agent and BFS contents at each basicity value were shown in Table 2.

2.2. Methods

2.2.1. Thermodynamic simulation

Thermodynamic simulations were performed using FactSage 7.1, which simulates thermodynamic processes and provides theoretical support for complex calculations and simulations of. The FactSage software is based on the minimum Gibbs principle. Among the various modules of this software, the “Equilib” module can be used to calculate parameters for slag systems in thermal equilibrium at different temperatures. The “Equilib” module was selected to simulate BFS crystallization behavior at temperatures ranging from 1600 °C to 1000 °C, with a temperature gradient of 50 °C. The FToxide database was selected for this study, and all calculations were performed under a constant pressure of 1 atm. The thermodynamic simulation results assisted in observing the crystallization phases and their contents in modified BFS during cooling [13].

2.2.2. DSC analysis

The crystallization behavior of BFS was investigated by DSC (STA449F3 Jupiter, NETZSCH, Germany). For each DSC experiment, BFS samples of approximately 30 mg were ground to a size smaller than 200 mesh and were placed in an Al₂O₃ crucible in air. Samples were heated from 25 to 1500 °C at rates of 10–50 °C/min, and then maintained at 1500 °C for 30 min to homogenize the composition. Finally, samples were cooled to 25 °C at cooling rates of 20–60 °C/min.

The classical Johnson–Mehl–Avrami (JMA) theoretical equation is widely used in crystallization kinetics, providing the theoretical basis for interpreting DSC results. The equation can be simplified as follows [14]:

$$X = 1 - \exp[-Kt^n] \quad (1)$$

where X is the volume fraction crystallized at the specified temperature, t is the reaction time, n is the Avrami exponent, and K is the effective overall reaction rate given by the Arrhenius equation:

$$K = K_0 \exp(-E/RT) \quad (2)$$

where K₀ is the frequency factor, E is the activation energy for crystallization, R is the gas constant, and T is the temperature.

Differentiating Eq. (1) gives the following result:

$$dX/dt = Kn(Kt)^{n-1} \exp[-(Kt)^n] \quad (3)$$

Substituting Eq. (1) into Eq. (3) gives.

$$dX/dt = nK(1-X)[\ln(1-X)^{-1}]^{(n-1)/n} \quad (4)$$

The Kissinger method [15,16] is the method most commonly used method to analyze crystallization kinetics based on the JMA equation, treating crystallization as a first-order reaction which implies that $n = 1$ [15]. Thus, Eq. (4) can be simplified as follows:

$$dX/dt = K_0(1-X) \exp(-E/RT) \quad (5)$$

Taking the logarithm of Eq. (5), we obtain the following equation is obtained:

$$\ln\left(\frac{\alpha}{T_p^2}\right) = -\frac{E}{R} \cdot \frac{1}{T_p} + \ln\left(\frac{K_0 R}{E}\right) \quad (6)$$

where α is the heating rate for the DSC experiment, T_p is the peak temperature of the exothermic peak temperature of crystallization on the DSC curve, and R is the gas constant. By plotting $\ln(\alpha/T_p^2)$ vs. $10000/T_p$, E is obtained from the slopes of the straight lines.

The other widely used method to calculate the activation energy for crystallization during heating is the Ozawa method, which is expressed as follows:

$$\ln \alpha = -E/(RT_p) + C \quad (7)$$

where C is a constant. By plotting $\ln \alpha$ vs. $10,000/T_p$, E is obtained from the slopes of the straight lines.

2.2.3. XRD analysis

X-ray diffraction (XRD) analysis was performed on powder samples (ground by ZHM-1-P03061808). XRD spectra were collected at 298 K using an X-ray diffractometer equipped with CuK α radiation ($\lambda = 0.15405$ nm). The voltage and current were 40.0 kV and 100.0 mA, respectively. All diffraction profiles were obtained in the continuous mode in a 2θ range of 10°–80° at a scan speed of 2°/min.

3. Results and discussion

3.1. Influence of basicity on mineral crystallization

The relationship between mineral phases, crystallization temperature, and basicity during cooling was simulated by FactSage 7.1 according to the composition of the modified BFS. The mineral phases of the BFS are shown in Fig. 1 (a), which indicates that the crystallized content increased and the liquid slag content decreased gradually with decreasing temperature. The initial phase was melilite (Ca₂(MgAl)(AlSi)SiO₇), and the melilite phase began to crystallize at 1425.3 °C, which was slightly below the gas quenching temperature (1450 °C). The small temperature difference led to a smaller operational space,

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