



Full Length Article

Phase evolution of blast furnace slags with variation in the binary basicity in a variable cooling process

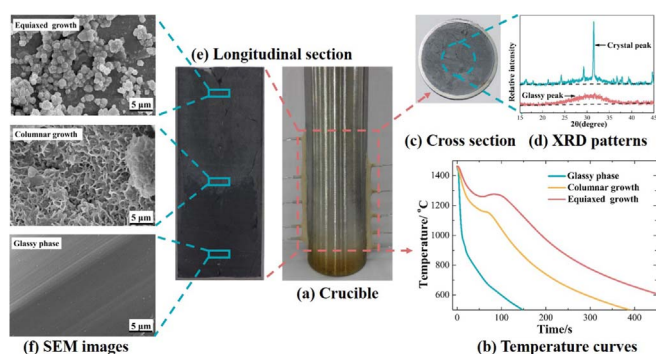


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GRAPHICAL ABSTRACT



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ABSTRACT

Understanding the crystallization behaviour of a blast furnace (BF) slag under a phase change cooling process and determining a suitable cooling rate are of tremendous importance for the double-wins centrifugal granulation and heat recovery technology. Moreover, the binary basicity shows a significant effect on the crystallization behaviour. In the study presented here, four synthetic BF slags with a binary basicity ranging from 1.0 to 1.4 were prepared. Moreover, the liquidus and glass transformation temperatures of the BF slags were obtained by differential scanning calorimetry (DSC). Furthermore, an improved directional solidification technique and X-ray diffractometer (XRD) were combined to explore the effect of the binary basicity on the crystallization region, critical supercooling degree and cooling rate, and crystal phase type and content. The results indicate that the crystallization region narrows gradually with a decrease in the average cooling rate. Moreover, the increase in the binary basicity results in a gradual decrease in the critical supercooling degree and a linear increase in the critical cooling rate. In contrast, the primary phase transforms from akermanite to merwinite as the binary basicity increases from 1.3 to 1.4, which leads to a sharp decrease in the critical supercooling degree and a rapid increase in the critical cooling rate. In addition, the critical cooling rate of the BF slag in the cooling process with a variable cooling rate is approximately 50% of that when the cooling process is conducted with a constant cooling rate.

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1. Introduction

A blast furnace (BF) slag is formed as a result of the refining reactions, remaining gangue of iron ore, erosion of a BF refractory, and added fluxes. Melting BF slag is trapped at a temperature of approximately 1500 °C [1], carrying a plenty amount of high-grade thermal energy. In 2015, the global output of pig iron was more than 1153 million tons and accompanied with approximately 384 million tons of BF slag production [2]. This implies that the total heat was equivalent to 22.3 million tons of standard coal [3]. Moreover, a BF slag is rich in lime (CaO), silica (SiO₂), alumina (Al₂O₃), and magnesia (MgO), which is a potential cement auxiliary material [4]. The atomic structure of a solid BF slag is governed by the cooling rate [5]. Specifically, a rapid cooling rate results in an open atomic structure (glassy phase), which has a good hydration activity. Therefore, a glassy slag is usually used as a cement clinker and has a high economic value. In contrast, a slow cooling rate produces low value-added crystal slag because the hydration activity is limited by the compact structure. At present, water quenching with a high cooling rate is the most well-known treatment to acquire a BF slag with a high glassy phase content. However, this treatment suffers from numerous shortcomings such as thermal energy waste, water consumption, and toxic gases (SO₂ and H₂S) emission [6,7].

At present, the centrifugal granulation and heat recovery is the best practical technology to solve the above-mentioned problems. It is characterized by slag granulation and air cooling, i.e. centrifugal granulation and waste heat recovery devices are its core components. In a centrifugal granulation device, a high-speed rotating atomizer (disk/cup) is adopted to break the melting slag into small droplets of diameter 1–5 mm [8–10]. Concurrently, the droplet is rapidly cooled to a semi-melting state by air during the flying process. Subsequently, the particles are further cooled to complete the solidification in the waste heat recovery device. However, the bottleneck of this technology lies in the rather low heat transfer capability of air. Therefore, this technology is faced with two contradictory problems: the demand for a rapid cooling rate to obtain a glassy slag and the requirement to harvest a high-quality thermal energy under a low cooling rate. Thus, understanding the crystallization behaviour of a BF slag under a phase change cooling process and determining a suitable cooling rate are of tremendous important to the double-wins technology.

It should be noted that the crystallization behaviour of a slag is governed by its main component [11,12]. In an alkaline melting slag, the acidic components (SiO₂, Al₂O₃) usually act as network formers, whereas the alkaline components (CaO, MgO) play the role of network modifiers [13,14]. Therefore, the binary basicity (CaO/SiO₂) and ratio of MgO/Al₂O₃ are usually adopted to investigate the effects of the main components on the crystallization behaviour of a slag [15]. Furthermore, the binary basicity plays a dominant role in the crystallization behaviour of a BF slag owing to the unique content of the main components: CaO (30–45%), SiO₂ (28–38%), Al₂O₃ (8–24%), and MgO (3–18%) [16]. At present, differential scanning calorimetry (DSC) [17], X-ray diffraction (XRD) [18–20], single/double hot thermocouple technique (S/DHTT), [21–23] and confocal laser-scanning microscope (CLSM) [24,25] are the commonly employed techniques to investigate the crystallization behaviour of a slag at a constant temperature/cooling rate. Specifically, DSC [17] is mainly used to obtain the liquidus, glassy transition, and crystallization onset and ending temperatures and latent heat of the glassy and crystal phases. XRD [18–20] is adopted to confirm the phase type and content of the precipitated crystal. S/DHTT [21–23] are the common approaches to explore the crystallization behaviour of a slag at an isothermal temperature or a constant cooling rate. For instance, SHTT was adopted by Qin et al. [26] to explore the crystallization behaviour of a BF slag at various binary basicities. The results indicated that the crystallization capability of a BF slag was enhanced with an increase in the binary basicity, producing to a higher critical cooling rate. CLSM [24,25] is a visualization method

to observe the growth process of the crystal phase at the micrometre scale. However, these methods fail to explore the phase change cooling process as well as the interplay between the phase change heat transfer and crystal phase growth owing to the limitation of a small amount of the testing sample. In our previous work [27], the interplay between the phase change heat transfer and crystal phase growth of a BF slag was thoroughly investigated by the directional solidification technique in combination with XRD and DSC. The results showed that a slow cooling rate resulted in the precipitation of the crystal phase, and thus, a more latent heat release that resulted in an obvious decrease in the cooling rate. However, until now, a further step of the effect of the binary basicity on the crystallization behaviour of BF slags in a phase change cooling process has not been reported.

In this study, four BF slag samples with binary basicity ranging from 1.0 to 1.4 were prepared to explore the effect of the binary basicity on the crystallization behaviour. Moreover, the liquidus and glass transformation temperatures of the BF slags were obtained by DSC. Furthermore, an improved directional solidification technique and XRD were combined to explore the effect of the binary basicity on crystallization region, critical supercooling degree and cooling rate, and crystal phase type and content. These investigations may provide a theoretical guidance for choosing the operating conditions for the dry graduation and waste heat recovery technique.

2. Experimental section

2.1. Samples preparation and analysis

To explore the effect of binary basicity on the crystallization behaviour of a BF slag, four synthetic BF slags were prepared by mixing the BF slag from Chongqing Iron & Steel (Group) Co., Ltd. (China) and the reagent-grade powders of CaO, MgO, Al₂O₃, and SiO₂. As listed in Table 1, the binary basicity (CaO/SiO₂) was 1.0, 1.2, 1.3, and 1.4, respectively. Moreover, the content of MgO, Al₂O₃, and other components was set at 10.81%, 10.81%, and 6.14%, respectively. To obtain the liquidus temperature (T_l) and glass transition temperature (T_g) of the slags, purely glassy slag samples were essential. The four samples were heated to 1550 °C in a heating furnace (Model SX-G08163, TJZH, China) at a heating rate of 10 °C·min⁻¹ to obtain a purely glassy slag. Subsequently, the temperature (1550 °C) was maintained for at least 1 h to completely melt the slag. Thereafter, the melting slag was removed from the heating furnace and quenched by water. Finally, the T_l and T_g of the four samples were detected by DSC (Q20 DSC, TA, USA). Moreover, the heating and cooling rates of the samples during the DSC tests were set at 10 °C·min⁻¹. As listed in Table 2, T_l gradually increases from 1370.3 ± 5.5 °C to 1408.3 ± 2.0 °C as the binary basicity increases from 1.0 to 1.4. In contrast, the T_g fluctuated in a temperature range between 740.1 ± 4.0 °C and 758.9 ± 1.5 °C. In addition, the slag samples with various crystal phase contents (for example, 10%, 30%, etc.) were adopted as the standard samples in the XRD test. The standard samples were usually obtained by mixing the purely glassy and crystal slag samples. Therefore, the melting slag (1550 °C) was slowly cooled to 1100 °C and kept warm for approximately 12 h to obtain a purely crystal slag.

Table 1
Major chemical components of the slag samples.

Sample		S1	S2	S3	S4
Chemical composition (wt%)	CaO	36.12	39.40	40.83	42.14
	SiO ₂	36.12	32.83	31.41	30.10
	MgO	10.81	10.81	10.81	10.81
	Al ₂ O ₃	10.81	10.81	10.81	10.81
	Other	6.14	6.14	6.14	6.14
Binary basicity	R	1.0	1.2	1.3	1.4

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