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ORIGINAL PAPER

High-temperature modification and air-quenching granulation of steel slag

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Abstract

To solve the problem of difficult utilization of steel slag, the liquid steel slag was modified and the air-quenching granulation process was carried out to make steel slag into a value-added end product: air-quenching granulated steel slag. The granulated slag was tested to analyze the variation rule of slag properties under different modification conditions. Based on the phase diagram of CaO–Si_2O–FeO–MgO–Al_2O_3 slag system, the feasibility of blast furnace (BF) slag as modifier was determined. When the addition of BF slag was increased from 0% to 35%, following results were obtained. The slag fluidity was improved, and the air-quenching temperature range was expanded. Then, the yield of air-quenched steel slag increased, while the granulation rate, the degree of sphericity, the compactness were decreased. Furthermore, the air-quenching granulation process could substantially improve the stability and the amorphous content of steel slag. The maximum removal rate of free CaO was above 80% and the amorphous content was up to 95%. Taking the factors of yield and properties of granulated steel slag into full consideration, the optimum proportion of BF slag is around 15%.

Keywords Steel slag · Modification · Air-quenching granulation · Free CaO · Amorphous · Property

1 Introduction

Steel slag produced in the steelmaking process is one of typical industrial solid waste, which accounts for about 15% of the yield of crude steel [1]. In 2018, the yield of crude steel in China is 928 million tons [2], which is about 139.2 million tons if converted into the discharge amount of steel slag. However, the utilization rate of steel slag in China is less than 30%, which is much lower than that in developed countries. It is the outdated steel slag treatment process that is the root cause leading to the low utilization in China [3]. The steel slag has been accumulated over a long period of time and cannot be consumed, bringing about a great burden on China's land resources and environmental protection [4].

The discharging temperature of liquid steel slag is about 1450–1650 °C, and the heat produced is directly emitted into the atmosphere during the cooling process, resulting in heat waste of about 10^9 J per ton steel slag [5]. More and more attention has been paid to the efficient utilization and recovery of sensible heat of molten slag [6]. The slag treatment technology is advancing along the direction in taking full advantage of waste heat for dry granulation, which is considered as an ideal method of dealing with steel slag. Air-quenching granulation technology of molten slag is using high-pressure air jet to quench the liquid molten slag into granulated slag [7]. The crushing mechanism and crystallization behavior of air-quenched blast furnace (BF) slag have been studied [8, 9], which has a certain guiding significance for the air-quenching granulation of steel slag. Original molten steel slag has poor fluidity, but good fluidity of molten slag is the basic requirement of air-quenching process [10]. In order to improve the slag fluidity to meet the requirement of air-

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quenching, the high-temperature modification of molten slag is a common method [11]. Moreover, silicon-containing materials as modifier have an obvious effect on eliminating free CaO (f-CaO) in steel slag [12]. Ma'anshan Iron and Steel Co., Ltd. adopted the method of air-quenching and water-cooling to deal with steel slag and realized industrial production [13], through which the utilization rate of steel slag has been improved significantly. In addition, due to the advantages of high sphericity and good stability, air-quenched steel slag has been applied to abrasive [14] and concrete fine aggregate [15] with good results. Also, since the air-quenching process can lead to the rapid cooling of steel slag, the amorphous transition degree of beads can be improved [16], which is favorable for replacing Portland cement clinker [17].

Based on the principle of using waste to treat waste and easy to obtain, in this study, high silicon solid waste blast furnace slag was taken as the modifier of high-temperature modification, and compressed air was taken as a medium to conduct air-quenching process. Besides, the air-quenching effect, amorphous transition and partial properties of air-quenched slag were analyzed. It is an exploration of dry granulation of steel slag, aiming to provide a reference for the subsequent industrial application of air-quenching granulation of liquid steel slag.

2 Experimental

2.1 Materials

Lack of the conditions of direct air-quenching of liquid steel slag, this experiment uses cooled steel slag as the raw materials (as shown in Fig. 1), which are firstly re-melted in a small DC arc furnace and then used for the air-quenching process. The raw materials in this experiment are untreated converter steel slag and water-quenched BF slag provided by a local steel company. Steel slag is the main composition and BF slag is the modifier. The main chemical compositions of steel slag and BF slag are shown in Table 1.

Air-quenching granulation requires good fluidity and low melting temperature of molten slag. However, the liquidity of the original steel slag is generally poor. When the slag basicity is relatively high, the viscosity grows rapidly with the decrease in temperature [18]. In this experiment, the basicity of steel slag is 2.88, which is slightly high. The measured viscosity is larger, which is not conducive to the air-quenching granulation. BF slag is used as the modifier in this study, and the reasons are as follows. On one hand, it contains more SiO_2, which can effectively lower the basicity of slag and then change the fluidity and viscosity, in order to make the slag achieve a better air-quenching condition. On the other hand, it contains more Al_2O_3, which can eliminate f-CaO, reduce melting point and promote the transition of steel slag into amorphous state [19].

2.2 Modification

The poor fluidity of original steel slag will lead to incomplete air-quenching result and low granulation rate. Therefore, the original steel slag must be modified to improve the fluidity. The crystallization behavior of slag is analyzed by using FactSage thermodynamic software, which provides theoretical support for high-temperature modification.

Fig. 1 Morphological characteristics of converter steel slag. **a** Hot steel slag; **b** cooled steel slag

Table 1 Main chemical composition of raw materials (%)

Raw material	CaO	SiO_2	MgO	Al_2O_3	TiO_2	FeO	f-CaO
Steel slag	31.42	12.49	14.33	3.39	0.64	19.72	3.07
BF slag	37.38	32.30	8.48	14.18	1.56	–	–

2.3 Air-quenching granulation process

The air-quenching granulation process belongs to the category of dry granulation of metallurgical slag. High-speed air jet impingement on the liquid steel slag can form beads with stable performance and uniform particle size by adjusting the parameters like addition amount of modifier, nozzle type, air-quenching pressure, slag temperature, etc.

Figure 2 shows the change of slag flow from the beginning of pouring hot slag to the end of air-quenching. The process is as follows. Firstly, the steel slag was heated until melting in the arc furnace. Then, BF slag was added to modify the steel slag and continue heat until the temperature exceeded 1600 °C, so as to fully melt the mixed slag. Next, molten slag was poured. The liquid slag flew out from the outlet and then fell into the chute, at the bottom of which there exists a flow hole. When the liquid slag flew along the slag chute to the hole, it started to free fall in the vertical direction. Then, after pushed by the aerodynamic force of high-speed compressed air jet in the horizontal direction, the slag flow was smashed by the high-speed jet and flew along the direction. Finally, it was cooled into beads that fell into the beads collection room.

2.4 Preparation and characterization of air-quenched granulated steel slag

In the experiment of air-quenching granulation of steel slag, the following essential parameters are fixed: the temperature of slag is 1600 °C; the nozzle is Laval nozzle (the Mach number is 1.4); the air-quenching pressure is 0.3 MPa; the slag flow rate is 12.5 kg/min; and the distance between nozzle and slag flow is 10 cm. The addition amount of BF slag is the only variable, which is 0%, 5%, 15%, 25% and 35%, respectively. The air-quenched granulated steel slag obtained is named A0, A5, A15, A25 and

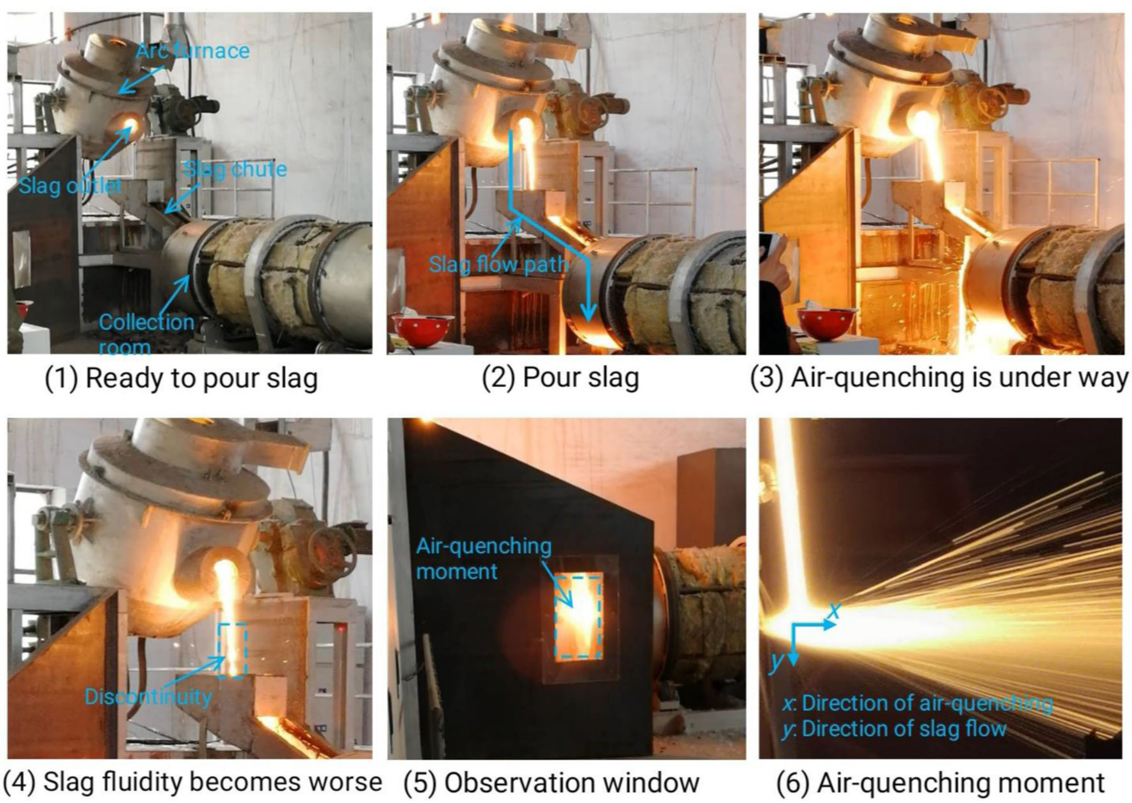

Fig. 2 Change of slag flow during air-quenching process of steel slag

A35, respectively. Then, the morphological characteristics, degree of sphericity, compactness and stability of air-quenched steel slag were analyzed.

3 Results and discussion

3.1 Thermodynamic analysis of crystallization

The phase diagram module in thermodynamic simulation software FactSage 8.0 was employed to simulate the crystallization behavior of slag after modification. Based on the air-quenching experimental setting, the calculation conditions were as follows: FToxid database, pressure $P = 100$ kPa, temperature $T = 1300$–1650 °C, and temperature gradient $\Delta T = 50$ °C. CaO, SiO_2, FeO, MgO and Al_2O_3 account for more than 80% in steel slag and the five oxides are the main components of steel slag. It is assumed that the contents of MgO and Al_2O_3 in slag are constant that is approaching the actual slag composition ($w_{MgO} = 14\%, w_{Al_2O_3} = 4\%$), and then, the phase diagram of CaO–SiO_2–FeO–MgO–Al_2O_3 system is calculated to analyze the influence of the change of CaO–SiO_2–FeO component on the slag performance after adding BF slag, as shown in Fig. 3.

Figure 3 shows that FeO reacts with Si and Ca oxides in slag to form eutectic with low melting point, and almost no iron-containing crystal exists in the phase diagram above 1300 °C. The basicity of original steel slag is 2.88 and the FeO content is 19.72%, as shown of point B in Fig. 3. The precipitated phase of the original slag at high temperature is periclase and the precipitated temperature is above 1650 °C. The original steel slag is already solid–liquid coexistence before air-quenching, which results in high crystal content in slag after air-quenching. After adding BF slag to steel slag gradually, both the basicity (R_2) and content of FeO decrease (R_2: A → A′, w_{FeO}: C → C′), and the composition point of molten slag moves along the direction of BB'. The liquidus temperature of slag decreases gradually according to the temperature isotherm change, and the precipitated crystals are melilite and pyroxene with lower melting point. Air-quenching process can realize rapid cooling of molten slag in high-temperature region, and the ions in slag do not have enough time to rearrange and form crystal, and finally form amorphous phase. Therefore, the addition of BF slag can reduce the liquidus temperature of molten slag and contribute to the formation of amorphous vitreous body after air-quenching.

The phase diagram is of guiding significance to the crystal precipitation behavior during the cooling process of molten slag; however, the composition of steel slag is actually extremely complex, and the crystallization behavior is difficult to be controlled accurately. For example, some tiny changes of certain substances that occupy a small amount may bring about a huge change of slag property. Therefore, the actual detection of steel slag mineral phase is indispensable in the research process.

3.2 Viscosity of molten slag

Viscosity is one of the main physical properties of metallurgical melts, which characterizes the geometry and degree of polymerization of anion groups in molten slag. Due to the simple operation, high precision and good repeatability, rotating cylinder method [20] is widely used in the measurement of slag viscosity. The RTW-13 melt viscosity tester, based on the rotating cylinder method, was employed to measure the viscosity data, as shown in Fig. 4, and castor oil was used to calibrate the viscosity tester before the experiment to ensure the accuracy of the experimental results. Also, a comprehensive analysis was conducted on the viscosity and fluidity of molten slag combined with the changes of liquid slag surface in the electric arc furnace melting pool (as shown in Fig. 5). The accurate measurement of slag temperature is achieved by the combined use of the thermocouple and temperature measuring gun in this study.

In the study of steel slag viscosity, FactSage software is generally used to calculate the viscosity and there are few measured values [21]. However, the liquidus temperature of the steel slag is pretty high, and the calculation with FactSage software is far away from the real value. Figure 4 shows the viscosity–temperature curve of actual slag; it can be concluded that the viscosity of steel slag decreases after it is modified by BF slag, and with the increase in proportion of BF slag, the viscosity decreases gradually. The reasons for the decrease in viscosity are as follows. First, the decrease in slag liquidus temperature by adjusting the CaO–SiO_2–FeO component after adding BF slag is the direct cause of the decrease in viscosity. Second, the formation of substances like FeO·SiO_2 with low melting point and relative decrease of substances like MgO with high melting point in slag promote the decrease in viscosity [22]. Third, the increase in Al_2O_3 in slag destroys the complex $[SiO_4]^{4-}$ tetrahedron structure and releases more free oxygen ions (O^{2-}), which is beneficial to reduce the viscosity of slag [23].

Generally, when the viscosity of slag is greater than 1 Pa s, it is easy to be blown into slag fibers [24]. Therefore, the viscosity required in air-quenching granulation of slag must be less than 1 Pa s, so as to ensure a good effect. It can be seen from Fig. 4 that when the viscosity is 1 Pa s, the temperature of original steel slag is 1450 °C, while the temperature of all modified slags is less than 1450 °C, which is the lowest temperature of air-quenching. After

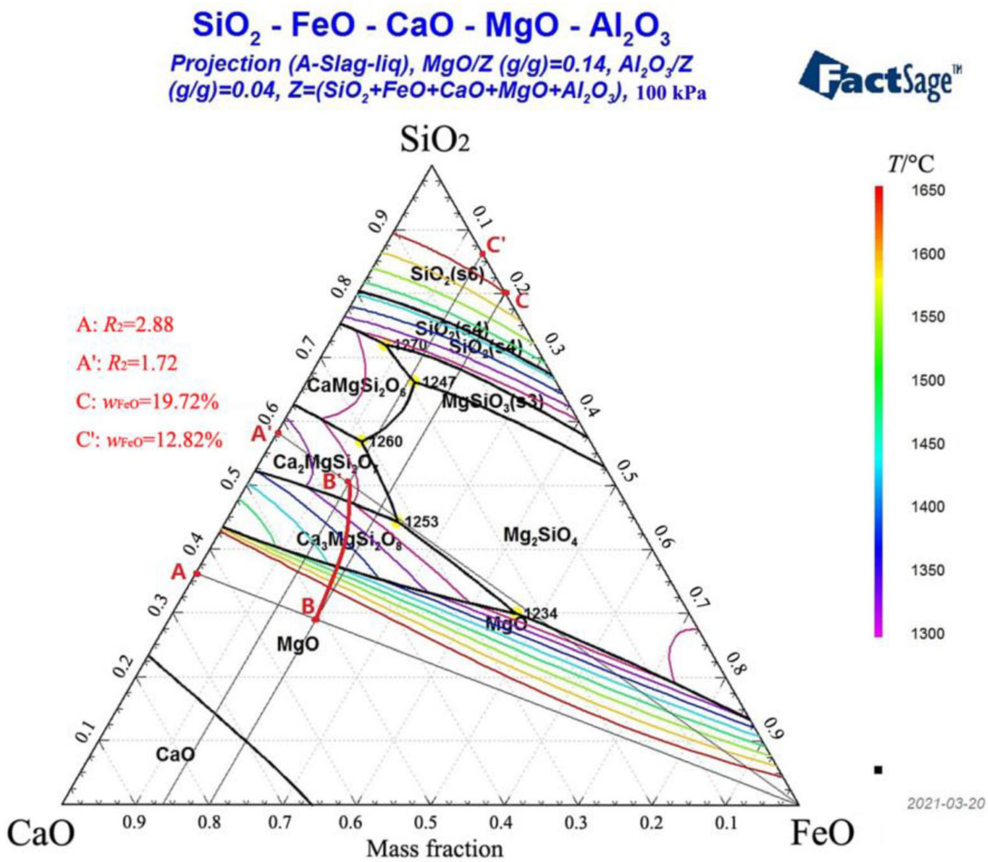

Fig. 3 Phase diagram of CaO–SiO$_2$–FeO–MgO–Al$_2$O$_3$ system ($w_{MgO} = 14\%, w_{Al_2O_3} = 4\%$)

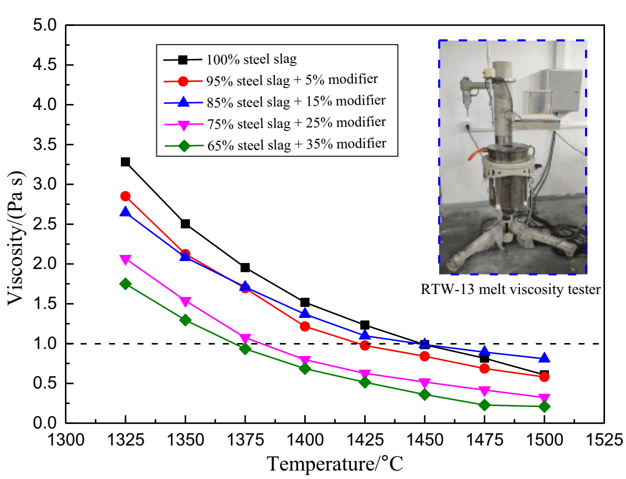

Fig. 4 Effect of addition amount of modifier on viscosity of steel slag

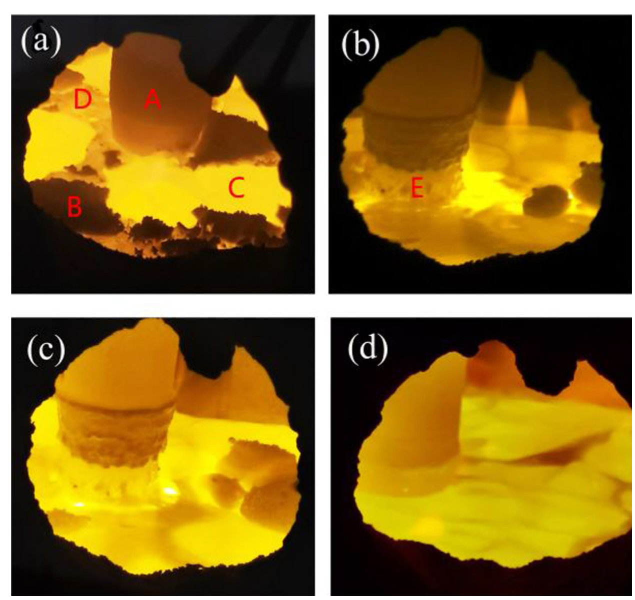

Fig. 5 Change of liquid slag in arc furnace melting tank (5% BF slag). **a** Slag begins to melt at about 1350 °C; **b** slag basically melts at about 1450 °C; **c** slag is in free flow state at about 1550 °C; **d** slag at 1600 °C. Point A is electrode; Point B is solid slag; Point C is liquid slag; Point D is foam slag; Point E is electric arc

modification, the viscosity curve moves downward. At the same temperature below 1450 °C, the greater the degree of modification, the lower the viscosity of slag, which greatly widens the temperature range in accordance with air-quenching process.

Figure 5a shows that there still exists solid slag at 1350 °C in the furnace melting pool (Point B). The steel slag around electric arc melts first, which indicates that the temperature here is the highest and then the slag melts around gradually. In Fig. 5b, the solid slag almost disappears and the steel slag is basically melted. There floats a layer of foam scum on the surface of the slag, which is produced through the carbothermal reduction reaction of both iron oxides in the steel slag and carbon (coke and electrode in the furnace, etc.) that produces gases. As the temperature continues to rise, the foam scum decreases and slag is homogenized gradually. The slag in Fig. 5c, d has good fluidity and is suitable for air-quenching. It can be predicted from the viscosity–temperature curve of slag and molten pool level changes that the temperature of steel slag should not be lower than 1450 °C until the end of slag pouring. Besides, based on experience and considering the temperature drop during slag pouring, slag should have at least 150 °C degree of superheat, which means that the initial slag temperature should not be lower than 1600 °C. Only in this way will slag not be influenced by the temperature drop to become viscous, and thus will the air-quenching effect not be influenced.

3.3 Morphological characteristics of air-quenched steel slag

The morphology characteristics of granulated steel slag have great differences with various addition proportions of modifier. The accumulation state, appearance, surface and microstructure of five types of granulated steel slags are observed with camera, field emission scanning electron microscope and Zeiss Axio Scope A1 Polarizing/Reflective microscope, as shown in Fig. 6. The yield (the mass ratio of air-quenched steel slag and total materials), the granulation rate (the mass ratio of granulated steel slag and total air-quenched steel slag) and the amorphous content are shown in Table 2. The amorphous content of sample can be directly measured when the microstructure is observed under the Zeiss Axio Scope A1 Polarizing/Reflective microscope.

3.3.1 Appearance

It can be concluded from the accumulation state of air-quenched granulated steel slag in Fig. 6 that, with the increase in proportion of modifier, the granulated steel slag decreases gradually and the flakes increase. Especially when it comes to the accumulation state of A35, the granulated slag can hardly be seen. In view of the appearance in Fig. 6, various shape compositions of different air-quenched steel slags are shown in high definition. A0 and A5 have uniform particle size and are gray. In A15, a few light green flakes appear, while in A25 and A35, light green flakes increase dramatically and even become the main part. It shows that, with the increase in the addition proportion of modifier, the color of air-quenched granulated steel slag gradually turns to light green from gray, and the shape gradually changes from sphere to flake with the granulation rate decreasing. According to the literature [23], under the condition of appropriate basicity, slag will even be blown into slag fibers. From the surface features of A0 to A35 in Fig. 6, the surface smoothness has a gradual increase with impurities decreasing, which may be caused by the increase in amorphous phase content in slag.

From Table 2, the yield of air-quenched steel slag increases while the granulation rate reduces gradually with the increase in BF slag addition amount. The granulation rate of original steel slag is high, but the viscosity of it grows too fast, which leads to incomplete air-quenching, and solidification of steel slag in the furnace and slag chute leads to low yield of air-quenched slag in collection room. Instead, the modified steel slag can be treated completely by air-quenching due to good fluidity. Based on the appearance of the air-quenched steel slag in Fig. 6, it can be concluded that the reduction in granulation rate after air-quenching is mainly caused by a large number of flakes produced. The granulated steel slag, after simple magnetic separation and sieving, can be applied directly to fine aggregate or abrasive without treatment, which can increase the additional value of steel slag and broaden the utilization field of steel slag.

3.3.2 Microstructure

It can be seen from the amorphous content in Table 2, when the steel slag is not modified, only a small amount of amorphous phase is detected in the air-quenched steel slag, most of which still exists in the form of crystal. Much amorphous phase, even up to 95%, is obtained when BF slag is used to modify steel slag. Amorphous content in the air-quenched steel slag decreases first and then increases with the addition amount of BF slag, which should be due to temperature drop caused by the time difference in temperature measurement and air-quenching during slag pouring. At this point, the slag reaches the condition of crystal precipitation during the air-quenching process. More crystal precipitation leads to less amorphous content in air-quenched steel slag. From the microstructure in Fig. 6, A0 contains magnetite, limonite, melilite, etc., while the opalescent parts of A5 to A35 are amorphous vitreous body. Meanwhile, a small amount of Ca_2SiO_4, MgO and metallic iron exist. Therefore, it can be inferred that when the addition amount of modifier is 25%, amorphous content becomes less due to the large precipitation of Ca_2SiO_4, MgO, etc. Above phenomena indicate that air-

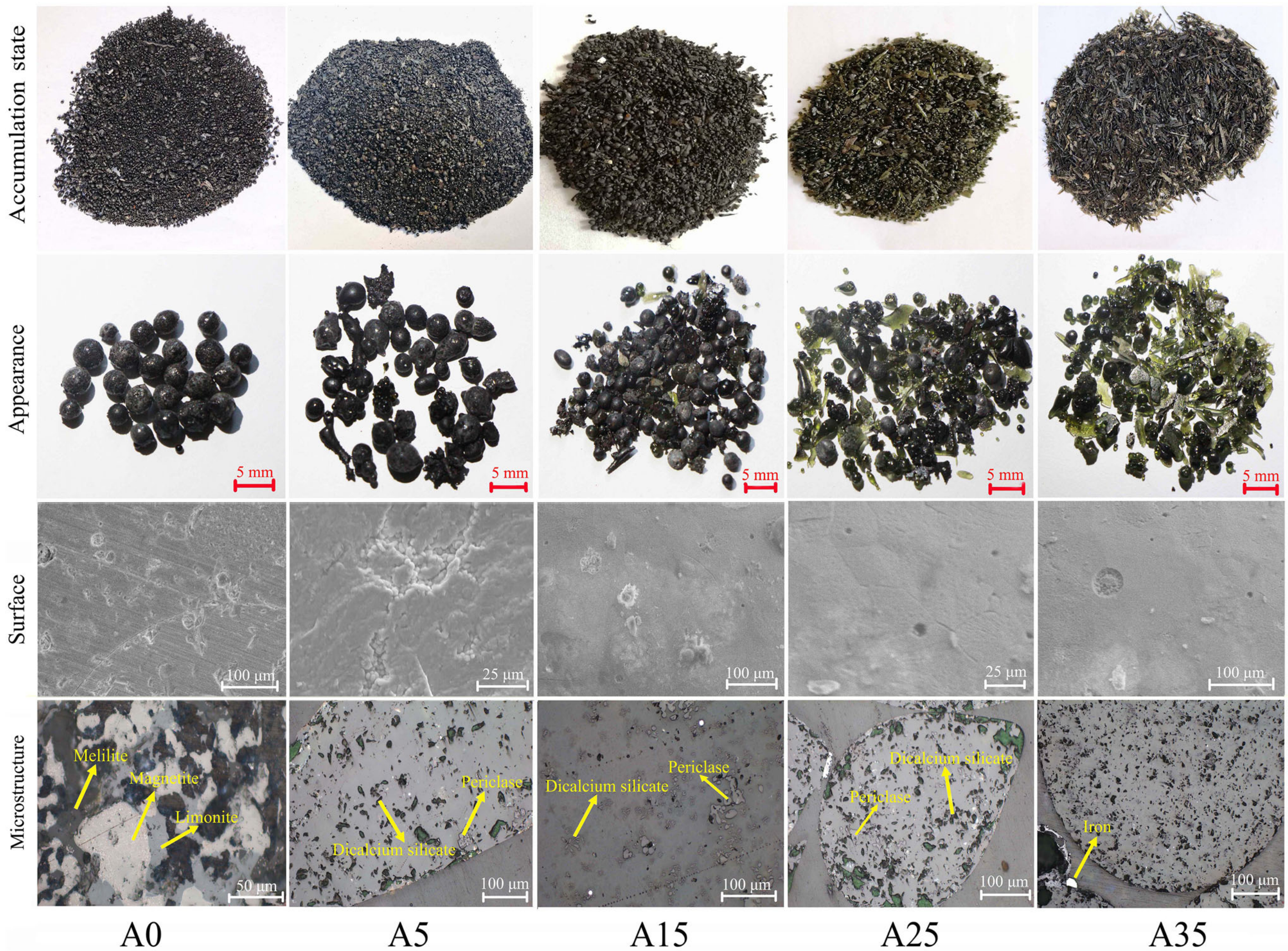

Fig. 6 Macro- and micro-photographs of air-quenched granulated steel slag

Table 2 Yield, granulation rate and amorphous content of air-quenched steel slag

Granulated slag No.	A0	A5	A15	A25	A35
Yield/wt.%	33.5	55.3	76.3	82.9	89.3
Granulation rate/wt.%	95	91	85	79	< 50
Amorphous content/vol.%	Little	90–95	85–90	70–80	93–95

quenching process can promote the transformation of steel slag to amorphous state, but the transformation degree is very low. After being modified by BF slag, the transformation degree of air-quenching steel slag to amorphous state is improved.

According to the amorphous content and the change of the morphological characteristics in Fig. 6, it can be further inferred that when the amorphous content is high, the surface of the air-quenched steel slag is smoother and more luminous. On the contrary, with low amorphous content and more crystal precipitation, the surface is dull and lackluster. Meanwhile, according to the smooth, transparent and glossy features of flakes in Fig. 6, it can be concluded that the flakes belong to the amorphous structure. The high amorphous content in air-quenched steel slag is beneficial to be used as cement clinker.

3.4 Sphericity

Based on the granulation rate, the sphericity degree of four kinds of granulated beads is analyzed (the granulation rate of A35 is low, so there is no analysis of it). The sphericity analysis method is as follows. Firstly, four kinds of granulated steel slags are sampled by quartering, and 50 pieces of each are randomly detected. Then, the maximum length and minimum length of middle section of a single bead are measured, respectively, which can be obtained by Eq. (1).

$$K = \frac{D_{\max}}{D_{\min}} \quad (1)$$

where K is the particle size coefficient of bead; $D_{\max}$ is the maximum length of bead, mm; and $D_{\min}$ is the minimum length of middle section of bead, mm. If the K value is close to 1, it proves high sphericity. The analysis results are shown in Fig. 6.

According to the results of sphericity detection of different types of granulated slags, the sphericity and dispersion of grain size distribution can be observed. The black diagonal line is the base line, and the K value is 1, which means spherosome. The closer the data point is to the base line, the better the sphericity. Besides, the red line is the fitting curve. The larger the correlation coefficient (the value of "Adj. R-square" in Fig. 7) is, the closer the fitting data are to the actual data. Meanwhile, when the slope of the fitting curve is larger, the sphericity of the granulated slag is worse. Based on these, it is presented in Fig. 7 that the grain size distribution of A0 is wide and its sphericity is high, while the other three kinds of granulated slags show a general trend of smaller particle size, closer to the base line, and higher sphericity. Also, the particle size of four types of granulated slags is mainly centered in the range of 1–3 mm. Their correlation coefficients which fit curves are 0.98774, 0.97012, 0.95656 and 0.88545, respectively, and the slopes are 1.02373, 1.12913, 1.15409 and 1.19457, respectively. It indicates that the fitting curve of granulated slag A0 is the best and its spherical degree is the highest, while the fitting degree of the other three kinds of fitting curves of granulated slag gradually decreases with the slope increasing. Furthermore, it infers that the sphericity of granulated slag gradually decreases. The results of sphericity are basically consistent with the appearance characteristic of air-quenched slag shown in Fig. 6.

3.5 Compactness

The compactness is an important index to measure the performance of steel slag. With good compactness, it can expand the utilization range of steel slag (such as fine aggregate and abrasive). The compactness of steel slag

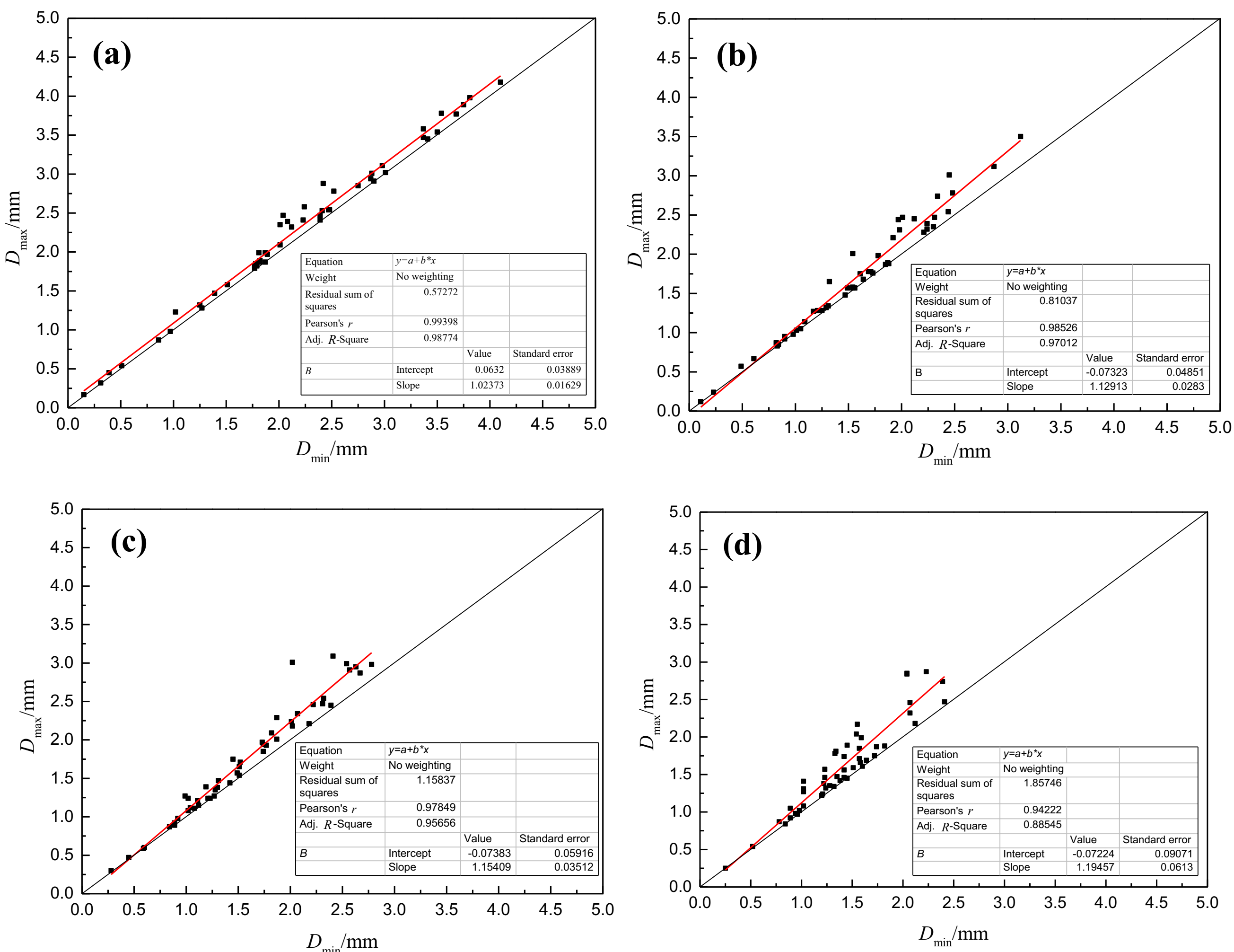

Fig. 7 Analysis of sphericity of four types of granulated steel slags. **a** Granulated slag A0; **b** granulated slag A5; **c** granulated slag A15; **d** granulated slag A25

beads can be characterized by sand crushing value of the Chinese standard GB/T14684-2011. The smaller the crushing value, the more compact the sample. After the granulated steel slags with different grain sizes were loaded to 25 kN as required, they were screened separately, and then, the crushing value is calculated according to Eq. (2). The calculation results are shown in Table 3.

$$Y_i = \frac{G_2}{G_1 + G_2} \times 100 \tag{2}$$

where Y_i is the crushing value of air-quenched steel slag, %; G_1 is the mass of screen residue, g; and G_2 is the screen throughput, g. The maximum crushing value of slags with different grain sizes is considered as the crushing value of this type of air-quenched granulated steel slag.

It can be seen from Table 3 that, with the increase in the proportion of modifier, the crushing value of air-quenched granulated steel slag increases gradually while the compactness decreases. The crushing value of A0 is 13.0%, the strongest, while the crushing value of A35 is the highest and the bead is the most easily broken. The change of the slag compactness should be related to the content of amorphous vitreous body and flakes. The more the content of amorphous vitreous body and flakes are, the more easily the granulated slag is broken. In the same type of granulated slag, the crushing value ascends with the increase in particle sizes. The smaller the particle size is, the tighter the granulated slag accumulation is, and the more compact the granulated slag is.

3.6 Stability

The content of f-CaO in steel slag is the key factor affecting the stability. According to the Chinese standard YB/T 4328-2012, the f-CaO content in different air-quenched granulated steel slags was obtained with Sucrose—EDTA complexometric titration method, as shown in Fig. 8.

As shown in Fig. 8, the initial value of f-CaO content in the original steel slag is 3.07%, which is relatively high. The expansion and pulverization of the steel slag will lead to poor stability. The f-CaO content in the five kinds of granulated slags is lower than 3.07%, indicating that the air-quenching method can reduce f-CaO content and stabilize free calcium. With the increase in the addition amount of modifier, f-CaO content of granulated slag declines gradually. When the addition proportion is 35%, f-CaO content reaches the least value (0.60%), and the eliminating rate of f-CaO exceeds 80%. It is concluded that the air-quenching and modification of steel slag have played the role in eliminating f-CaO. The reduction in f-CaO content has two main reasons. First, due to the rapid cooling during the droplet flight, there is no time for f-CaO and other mineral phases to crystallize, only to form amorphous vitreous body. Second, f-CaO is captured by SiO_2 in BF slag to form stable $2CaO{\cdot}SiO_2$ products [25].

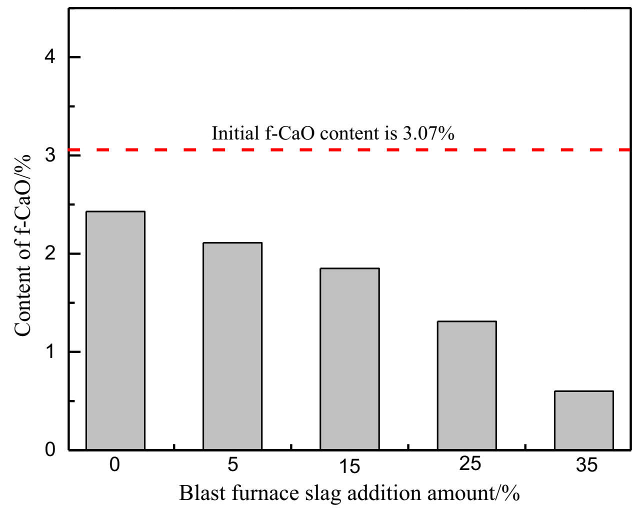

Fig. 8 f-CaO content in granulated steel slag

4 Conclusions

1. With the increase in the proportion of BF slag, the fluidity of molten slag increases. When the viscosity is less than 1 Pa s, and the superheat of molten slag is at

Table 3 Crushing value of air-quenched granulated steel slag (%)

Granulated slag No.	Crushing value of various grain sizes				Crushing value of granulated slag
	0.30–0.63 mm	0.63–1.25 mm	1.25–2.50 mm	2.5–5.00 mm	
A0	10.6	11.2	12.5	13.0	13.0
A5	14.9	16.3	17.5	18.2	18.2
A15	16.5	17.1	19.8	21.3	21.3
A25	30.2	33.7	35.2	35.9	35.9
A35	33.3	35.9	38.1	36.5	38.1

least 150 °C, better air-quenching effect can be obtained.

2. After modification, the shape varies from sphere to flake, and the color changes from gray to light green. In addition, the yield increases while the granulation rate decreases, and the surface becomes smoother with the gradual drop of impurities. The modification and air-quenching increase the degree of slag amorphous state.
3. The compactness and sphericity of granulated slag decrease gradually. After modification and air-quenching, the content of f-CaO is greatly reduced, and the maximum eliminating rate of f-CaO is over 80%.
4. Considering production rate, properties and application of air-quenched granulated steel slag, the optimal addition amount of BF slag is around 15%.

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