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Desulfurization Ability of Blast Furnace Slag Containing High Al_2O_3 at 1773 K

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Abstract: With the consumption of high-quality iron ore resources, the grade of iron ore raw used in the iron making process has gradually decreased. The high aluminum raw materials has led to an increase in the Al_2O_3 content in the blast furnace slag, which has affected the normal operation of the blast furnace. The activity of the components involved in the desulfurization reaction is an important factor affecting the desulfurization process. In this paper, the effects of $B(w(\text{CaO})/w(\text{SiO}_2))$ and $w(\text{MgO})/w(\text{Al}_2\text{O}_3)$ on the desulfurization ability of a $\text{CaO-SiO}_2\text{-MgO-Al}_2\text{O}_3$ quaternary blast furnace slag system were studied by using a double-layer graphite crucible to simulate the process of molten iron dropping through the slag at 1773 K. The desulfurization reaction mechanism of high alumina blast furnace slag is explained from the aspects of slag structure and component activity, which provides a theoretical basis for the reasonable selection of a slag making system under the condition of a high alumina content. The effects of different B and $w(\text{MgO})/w(\text{Al}_2\text{O}_3)$ on the slag structure 2343 analyzed by Fourier transform infrared spectroscopy (FT-IR). The results show that when $w(\text{MgO})/w(\text{Al}_2\text{O}_3) = 0.50$ and $w(\text{Al}_2\text{O}_3) = 20\%$, the desulfurization ability and MgO activity of slag increased with the increase of B ; when $w(\text{Al}_2\text{O}_3) = 20\%$ and $B = 1.30$, the desulfurization ability and MgO activity of the slag increased with the increase of $w(\text{MgO})/w(\text{Al}_2\text{O}_3)$. The FT-IR analysis showed that with the increase of B or $w(\text{MgO})/w(\text{Al}_2\text{O}_3)$, the dissociated free oxygen ions (O^{2-}) in the slag increased, and the dissociated free oxygen ions (O^{2-}) interacted with the bridging oxygen (O^0) of silicate, which made the complex Si-O structure in the slag gradually depolymerize and increase the desulfurization ability.

Keywords: slag; $B(w(\text{CaO})/w(\text{SiO}_2))$; $w(\text{MgO})/w(\text{Al}_2\text{O}_3)$; desulfurization ability; sulfur distribution ratio(L_s); FT-IR

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1. Introduction

In recent years, with the extensive use of high alumina iron ore, the content of Al_2O_3 in slag has increased greatly, which inevitably affects the metallurgical properties of blast furnace slag [1–10]. Among these metallurgical properties, the desulfurization ability of blast furnace slag plays an important role in the quality of hot metal. Many scholars have done a lot of research on the metallurgical properties of blast furnace slag. Shankar A's study on the desulfurization ability of a $\text{CaO-SiO}_2\text{-MgO-Al}_2\text{O}_3$ blast furnace slag system shows that the desulfurization ability of slag increases with the increase of slag $B(w(\text{CaO})/w(\text{SiO}_2))$ or $w(\text{MgO})$ content [11]. The desulfurization ability of the $\text{CaO-SiO}_2\text{-MgO-Al}_2\text{O}_3$ quaternary slag system at 1773~1823 K was measured by Ma X D et al. [12,13]. In their study, the desulfurization ability of slag increased with the increase of $B(w(\text{CaO})/w(\text{SiO}_2))$ or $w(\text{MgO})$ content. The desulfurization ability of $\text{CaO-SiO}_2\text{-MgO-Al}_2\text{O}_3$ quaternary high MgO blast furnace slag system was studied by Condo et al. [14]. Many researchers such as Xin H, Wang Y, and Nzotta M M [15–17] have also studied the desulfurization ability of slag. Wang L J [18] and others analyzed the structure of chromium containing slag using Raman spectroscopy. The results show that the spectral results are consistent with the results

of the sulfide capacity measurements. Lee S [19] studied the viscosity behavior of FeO containing slag melt and a slag structure. Choi J S [20] studied the cationic effect of charge compensation on the sulfide capacity of aluminosilicate slag. Choi J S [21] studied the structure–property relationship amphoteric oxide systems via phase stability and ionic structural analysis. However, for the high alumina CaO-SiO₂-MgO-Al₂O₃ slag system, there are few reports on the desulfurization reaction mechanism of high alumina blast furnace slag from the perspective of the slag structure. Therefore, it is necessary to explain the desulfurization reaction mechanism from the perspective of the slag structure, so as to provide a scientific basis and theoretical guidance for the application of high alumina iron ore in the blast furnace.

The main factors affecting slag desulfurization are the furnace temperature and slag chemical composition. The hearth temperature should not be changed excessively. Therefore, improving the slag composition is the main means to improve the desulfurization ability of slag. In this paper, the effects of B and $w(\text{MgO})/w(\text{Al}_2\text{O}_3)$ on the desulfurization ability of a CaO-SiO₂-MgO-Al₂O₃ quaternary blast furnace slag system were studied using a double-layer graphite crucible to simulate the process of molten iron dropping through slag. According to the experimental results, the effects of B and $w(\text{MgO})/w(\text{Al}_2\text{O}_3)$ on the desulfurization ability of blast furnace slag were discussed. FT-IR was used to analyze the effect of different B and $w(\text{MgO})/w(\text{Al}_2\text{O}_3)$ on the slag structure. Further study on the microstructure of blast furnace slag can better understand the relationship between the slag chemical composition and metallurgical properties, and can provide a theoretical basis for the reasonable selection of a slag making system under the condition of a high Al₂O₃ content.

2. Experimental

2.1. Desulfurization Experiment

2.1.1. Experimental Principle

The desulfurization reaction in the blast furnace starts after the initial slag is formed. It is mainly carried out in the hearth or the drip zone, and is finally completed in the hearth. The desulfurization reaction is a process of ion migration at the interface between the slag and iron. For the desulfurization reaction, it can be considered that atomic sulfur, which was originally neutral in molten iron, was obtained at the interface between slag and iron, and the electrons in slag became S^{2−}. The oxygen anion (O^{2−}) in the slag loses electrons at the interface, becomes neutral atomic oxygen, and enters into molten iron. The schematic diagram of the desulfurization reaction of slag is shown in Figure 1. The desulfurization of the blast furnace slag mainly makes sulfur enter the slag in the form of sulfide through the reaction of basic oxide in slag and sulfur in molten iron. The theory of molecular ion coexistence holds that only free (Ca²⁺ + O^{2−}) and (Mg²⁺ + O^{2−}) ions have a desulfurization ability [22,23]. S^{2−} exists in slag in the form of (Ca²⁺ + S^{2−}) and (Mg²⁺ + S^{2−}) ion pairs, and participates in the desulfurization reaction [22,23].

According to the ion theory, the reaction equation of slag desulfurization can be expressed as follows [22,23]:

The sulfur distribution ratio (L_S) is a common evaluation index of the desulfurization ability, and its definition is shown in Equation (4):

$$L_S = (\%S) / [\%S] \quad (4)$$

where $(\%S)$ and $[\%S]$ are the sulfur content of the slag and hot metal, respectively.

Figure 1. The schematic diagram of the desulfurization reaction.

2.1.2. Experimental Procedure

The desulfurization experiment was carried out in the high temperature tubular furnace (RTW-10, Northeastern University, China), and the temperature of the tubular furnace was controlled by a PID controller. The special double-layer graphite sleeve crucible was used as the container in the experiment. The outer diameter of the upper and lower graphite crucibles was 60 mm, the inner diameter of the upper and lower graphite crucibles was 44 mm, the height of the lower graphite crucible was 40 mm, and the height of the upper graphite crucible was 80 mm. The bottom of the upper crucible was plugged with a 120 mm high graphite plug. The experimental device is shown in Figure 2.

Figure 3 shows the quaternary phase diagram of $\text{CaO-SiO}_2\text{-MgO-Al}_2\text{O}_3$ [24]. It can be seen from Figure 3 that the suitable slag composition for the blast furnace iron smelting should be in the primary crystal region of plagioclase ($2\text{CaO}\cdot\text{MgO}\cdot 2\text{SiO}_2$) [25]. In this region, the melting temperature is low and the isotherm distribution is sparse. Therefore, in this paper, an isothermal single variable desulfurization experiment was carried out for the contents of B and $w(\text{MgO})/w(\text{Al}_2\text{O}_3)$. The chemical reagents (CaO , CAS no. 1305-78-8, AR, 98.0% purity; SiO_2 , CAS no. 7631-86-9, AR, 98.0% purity; MgO , CAS no. 1309-48-4, AR, 98.0% purity; Al_2O_3 , CAS no. 1344-28-1, AR, 98.0% purity) employed as the collectors were procured from Sinopharm Reagent Group (Ningbo, China). The slag composition of the desulfurization experiment is shown in Table 1. The eight points in Figure 3 represent the eight groups of blast furnace slag systems in Table 1. In the preparation stage of the experiment, when the furnace temperature of the muffle furnace was raised to 1523 K, the graphite crucible containing the chemical reagents CaO , SiO_2 , MgO , and Al_2O_3 were put into the muffle furnace for pre-melting treatment to prepare the pre-melted slag. The heating process of the muffle furnace is shown in Figure 4.

Figure 2. Experiment set-up in a MoSi₂ resistance furnace: (1) furnace shell; (2) Si-Mo bar; (3) argon; (4) alumina reaction tube; (5) iron; (6) graphite crucible containing the samples; (7) pre-melted slag; (8) double Pt-Rh thermocouple; (9) temperature-controlled cabinet.

Figure 3. Quaternary phase diagram of CaO-SiO₂-MgO-Al₂O₃ [24].

Table 1. Composition of slag (mass fraction).

No.	Slag Compositions				B, -	$w(\text{MgO})/w(\text{Al}_2\text{O}_3)$, -
	CaO	SiO ₂	MgO	Al ₂ O ₃		
1	35.85	34.14	10.0	20	1.05	0.50
2	37.51	32.49	10.0	20	1.15	0.50
3	38.89	31.11	10.0	20	1.25	0.50
4	40.21	29.78	10.0	20	1.35	0.50
5	42.39	32.60	5.0	20	1.30	0.25
6	41.26	31.73	7.0	20	1.30	0.35
7	40.13	30.86	9.0	20	1.30	0.45
8	39.00	30.00	11.0	20	1.30	0.55

Figure 4. The heating process of the muffle furnace.

In the experiment, the iron in a graphite crucible came from a steel plant. The composition of iron before the experiment is shown in Table 2. Before the start of the experiment, iron and pre-melted slag were taken as samples with a mass ratio of 3:1. The slag sample was placed in the lower graphite crucible, while the iron sample was placed in the upper graphite crucible. The special double-layer graphite crucible was put into the high temperature tube furnace one by one. The switch was tightened with a graphite stopper. After the slag sample and iron sample were completely dissolved, when the temperature in the high temperature tubular furnace rose to 1773 K, the graphite stopper switch was turned on to make the molten iron flow in from the gap between the graphite stopper and the crucible, pass through the slag layer, and then flow to the bottom of the lower crucible for deposition, so as to simulate the process of molten iron dropping through the slag. The content of the slag composition of the equilibrium experiment was as follows: $w(\text{CaO}) = 39\%$, $w(\text{SiO}_2) = 30\%$, $w(\text{MgO}) = 11\%$, and $w(\text{Al}_2\text{O}_3) = 20\%$. The equilibrium times were set at 30, 60, 90, and 120 min, respectively. The relationship between the sulfur content in molten iron and the desulfurization time is shown in Figure 5. It can be seen from Figure 5 that after 80 min, the sulfur content in iron basically did not change. From 90–120 min, the sulfur content in iron decreased by 0.8%, which can be considered as the reaction basically reaching equilibrium. After 90 min of reaction, the crucible was taken out quickly and immersed in oil for rapid cooling to preserve the original state of the slag iron at the end of the reaction. During the experiment, high purity argon was introduced for protection, and then the samples were taken out to separate the slag and iron, and the sulfur content was analyzed to calculate the sulfur distribution ratio. The content of sulfur in the iron alloy was subjected to a chemical analysis using a PE Avio 500 Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES).

Table 2. Chemical compositions of the iron (mass fraction).

C	Si	P	S	Mn	Fe
4.63	0.260	0.104	0.105	0.229	94.672

Figure 5. Relationship between the sulfur content in the molten iron and the desulfurization time.

2.2. Activity Experiment

2.2.1. Experimental Procedure

In this paper, slag/metal equilibrium experiments were carried out for the determination of the activity of MgO in CaO-SiO₂-MgO-Al₂O₃ melts for blast furnace slag at T = 1773 K, using Sn as the metal solvent and CaF₂-MgO slag as reference slag. At equilibrium, the principal reactions between the molten Sn and reference slag or measured slag can be expressed as reactions (5) and (6), respectively:

where $p_{(\text{CO})}$ and p_{CO}^θ are the pressures of CO and standard atmosphere, respectively, and a_{C} is the activity of the carbon relative to the pure solid. When reaction (5) and reaction (6) are at the same conditions, equation $K_5 = K_6$ can be written as follows:

$$a_{(\text{MgO})} = a_{\text{ref}(\text{MgO})} \cdot \frac{a_{[\text{Mg}]}}{a_{\text{ref}[\text{Mg}]}} \quad (7)$$

where $a_{(\text{MgO})}$ and $a_{\text{ref}(\text{MgO})}$ are the MgO activities of the measured slag and reference slag relative to the pure solid, respectively. $a_{[\text{Mg}]}$ and $a_{\text{ref}[\text{Mg}]}$ are the activities of Mg in molten Sn at equilibrium with the measured slag and the reference slag relative to the infinite dilute solution, respectively.

The concentration of Mg in Sn is small enough to make the Henry's law suitable, so $f_{[Mg]} = 1$. The schematic diagram of the experimental principle is shown in Figure 6. From the CaF_2 - MgO phase diagram, when the mass fraction of MgO is greater than 48%, it is the saturated region of MgO . Therefore, the composition of point S ($w(\text{CaF}_2) = 45\%$ and $w(\text{MgO}) = 55\%$) was chosen as the reference slag in this study, so $a_{\text{ref}(\text{MgO})} = 1$. Thus, Equation (7) can be simply derived from Equation (8):

$$a_{(\text{MgO})} = \frac{x_{[\text{Mg}]}}{x_{\text{ref}[\text{Mg}]}} \quad (8)$$

where $x_{(\text{Mg})}$ and $x_{\text{ref}(\text{Mg})}$ denote the concentration of Mg in Sn at equilibrium with the measured slag and the reference slag, respectively.

Figure 6. The schematic diagram of the activity determination principle: (a) the phase diagram of the CaF_2 - MgO binary [24]; (b) schematic diagram of the slag sample placement.

2.2.2. Experimental Principle

In order to measure the activity of MgO in the silicate melt, solvent metal ($\text{Sn} \geq 99.9\%$) and the slag (measure slag or reference slag) were equilibrated in a MoSi_2 resistance furnace. The chemical reagents (CaO , CAS no. 1305-78-8, 99.99% purity; SiO_2 , CAS no. 7631-86-9, 99.99% purity; MgO , CAS no. 1309-48-4, 99.99% purity; Al_2O_3 , CAS no. 1344-28-1, 99.99% purity; CaF_2 , CAS no. 7789-75-5, 99.99% purity) employed as the collectors were procured from Sinopharm Reagent Group (Ningbo, China). The slag composition of the activity experiment is shown in Table 1. A six-hole graphite crucibles was used in the experiment. About 5 g of solvent metal ($\text{Sn} \geq 99.9\%$) and 10 g of the slag (measure slag or reference slag) were filled in a graphite crucible. When the temperature of a tubular furnace sample rose to 1500°C , CO was introduced. After the balance time was 24 h, the slag sample was oil cooled. Thereafter, the metal and the slag were carefully separated for chemical analyses using Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES).

3. Results and Discussion

In this paper, a double-layer graphite crucible was used to simulate the process of melting iron dropping through the slag. Isothermal single variable desulfurization experiments were carried out for B and $w(\text{MgO})/w(\text{Al}_2\text{O}_3)$. The desulfurization results of each slag sample are listed in Table 3. Previous researchers have done a lot of research on silicate melts [26–28], and divided the oxygen ions in the silicate melts into three types: bridging oxygen, non-bridging oxygen, and free oxygen. The silicate melt is a

tetrahedral structure, and the different tetrahedrons are connected by bridging oxygen (O^0) and non-bridging oxygen (O^-) to form a three-dimensional continuous space network structure. Basic oxides can break the three-dimensional network structure by releasing free oxygen ions (O^{2-}) and bridging oxygen to form non-bridging oxygen. The degree of polymerization of slags can be expressed by Equation (9).

$$O^{2-} + O^0 = 2O^- K_9 = \frac{a_{(O^{1-})}^2}{a_{(O^{2-})} \cdot a_{(O^0)}} \quad (9)$$

Table 3. Experimental results.

No.	$w(Al_2O_3)$, -	B , -	$w(MgO)/w(Al_2O_3)$, -	$w(S)$, -	$w[S]$, -	L_S , -	$a_{(MgO)}$, -
1	20	1.05	0.50	0.302	0.02	15.10	0.1917
2	20	1.15	0.50	0.332	0.016	20.75	0.2032
3	20	1.25	0.50	0.347	0.014	24.79	0.2531
4	20	1.35	0.50	0.348	0.011	31.64	0.2914
5	20	1.30	0.25	0.291	0.018	16.17	0.1304
6	20	1.30	0.35	0.319	0.016	19.94	0.1994
7	20	1.30	0.45	0.309	0.014	22.07	0.2454
8	20	1.30	0.55	0.334	0.012	27.83	0.3259

3.1. Effect of B on Desulfurization Ability of Slag

Figure 7 indicates the effect of B on the desulfurization ability and MgO activity of slag when $w(MgO)/w(Al_2O_3) = 0.50$ and $w(Al_2O_3) = 20\%$. It can be seen from Figure 7 that when $w(MgO)/w(Al_2O_3) = 0.50$, $w(Al_2O_3) = 20\%$, as B increases from 1.05 to 1.35, the desulfurization ability and MgO activity of the slag increases with the increase of B . With the increase of B , the content of CaO increases, the content of SiO_2 decreases, and the content of MgO and Al_2O_3 remains unchanged. With the increase of B in the slag, the content of CaO and the number of free oxygen ions (O^{2-}) in slag increase gradually, which makes the reaction of Equation (1) ($Ca^{2+} + O^{2-} + [S] = (Ca^{2+} + S^{2-}) + [O]$) go forward, and the desulfurization ability of the slag increases. Figure 8 indicates the oil cooled FT-IR of slag samples with different B at 1773 K when $w(MgO)/w(Al_2O_3) = 0.50$ and $w(Al_2O_3) = 20\%$. From the results of the FT-IR in Figure 8, it can be found that with the increase of B , the grooves of the $[SiO_4]$ tetrahedron structure at the wavenumber of $800\text{--}1200\text{ cm}^{-1}$ gradually become shallower and flattened, which indicates that the complex silicate structure polymerized by SiO_2 is reduced. With the increase of B , the number of free oxygen ions (O^{2-}) in the slag increases, which promotes the forward progress of reaction (3), resulting in the increase of the desulfurization ability of the slag. At a certain temperature, the equilibrium constant (K_9) is constant. As the content of free oxygen ions (O^{2-}) in the slag increases, the activity of free oxygen ions (O^{2-}) in the slag increases, the activity of bridging oxygen (O^0) decreases, and the activity of non-bridging oxygen (O^-) increases. The interaction between the free oxygen ions (O^{2-}) and bridging oxygen (O^0) of silicate breaks the complex Si-O-Si bond, and the complex silicate structure of the slag disintegrates into a simple silicate structure. The desulfurization ability of the slag is gradually enhanced.

Figure 7. Effect of B on the desulfurization ability and MgO activity.

3.2. Effect of $w(MgO)/w(Al_2O_3)$ on Desulfurization Ability of Slag

Figure 9 shows the effect of $w(MgO)/w(Al_2O_3)$ on the desulfurization ability and MgO activity of slag when $w(Al_2O_3) = 20\%$ and $B = 1.30$. It can be seen from Figure 9 that when $w(Al_2O_3) = 20\%$, $B = 1.30$, as $w(MgO)/w(Al_2O_3)$ increases from 0.25 to 0.55, the desulfurization ability and MgO activity of slag increases with the increase of $w(MgO)/w(Al_2O_3)$. With the increase of $w(MgO)/w(Al_2O_3)$ in the slag, the content of MgO and the number of free oxygen ions (O^{2-}) in the slag also increase gradually, which promotes the forward reaction formula (2): $(Mg^{2+} + O^{2-}) + [S] = (Mg^{2+} + S^{2-}) + [O]$, and increases the desulfurization ability of the slag. Figure 10 shows the oil cooled FT-IR of different $w(MgO)/w(Al_2O_3)$ slag samples at 1773K when $w(Al_2O_3) = 20\%$ and $B = 1.30$. From the results of the FT-IR in Figure 10, it can be found that with the increase of $w(MgO)/w(Al_2O_3)$, the grooves of the $[SiO_4]$ tetrahedron structure at the wavenumber of $800\text{--}1200\text{ cm}^{-1}$ gradually become shallower and flattened, which indicates that the complex silicate structure polymerized by SiO_2 is reduced. With the increase of $w(MgO)/w(Al_2O_3)$, the content of MgO in the slag increases, and the free oxygen ion (O^{2-}) is increased. At a certain temperature, the equilibrium constant (K_9) is constant. When the free oxygen ions (O^{2-}) in the slag increase, the activity of the free oxygen ions (O^{2-}) increases, so the activity of the bridge oxygen

(O^0) decreases and the activity of the non-bridge oxygen (O^-) increases. The interaction between free oxygen ions (O^{2-}) and silicate bridge oxygen (O^0) breaks the complex Si-O-Si bond, decomposes the complex silicate structure of the slag into a simple silicate structure, and improves the desulfurization ability of the slag.

Figure 8. The FT-IR results of different B slag samples at 1773 K.

Figure 9. Effect of $w(\text{MgO})/w(\text{Al}_2\text{O}_3)$ on the desulfurization ability and MgO activity.

Figure 10. The FT-IR results of different $w(\text{MgO})/w(\text{Al}_2\text{O}_3)$ slag samples at 1773 K.

4. Conclusions

In this paper, a double-layer crucible was used to simulate the process of iron droplets passing through the slag. The desulfurization effect of $B(w(\text{CaO})/w(\text{SiO}_2))$ and $w(\text{MgO})/w(\text{Al}_2\text{O}_3)$ on high alumina blast furnace slag was studied. The conclusions are as follows: (1) when $w(\text{MgO})/w(\text{Al}_2\text{O}_3) = 0.50$ and $w(\text{Al}_2\text{O}_3) = 20\%$, the desulfurization ability and MgO activity of the slag increased with the increase of B ; (2) when $w(\text{Al}_2\text{O}_3) = 20\%$ and $B = 1.30$, the desulfurization ability and MgO activity of the slag increase with the increase of $w(\text{MgO})/w(\text{Al}_2\text{O}_3)$. (3) FT-IR analysis shows that with the increase of B or $w(\text{MgO})/w(\text{Al}_2\text{O}_3)$, the dissociated free oxygen ions (O^{2-}) in the slag increase, and the dissociated free oxygen ions (O^{2-}) interact with the bridging oxygen (O^0) of silicate, which makes the complex Si-O structure in the slag gradually depolymerize and increase the desulfurization ability.

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