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Article

Effect of CaO/SiO_2 and MgO/Al_2O_3 on the Metallurgical Properties of Low Boron-Bearing High Alumina Slag

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Abstract

It is crucial for the efficiency and the productivity of operations that the viscous flow behavior of the $CaO-SiO_2-MgO-Al_2O_3-B_2O_3$ blast furnace slag systems. In this paper; the effects of CaO/SiO_2 and MgO/Al_2O_3 on the viscosity; the break point temperature (T_{Br}) and activation energy (E_η) of low boron-bearing high alumina slag were considered in detail. Meanwhile; the effect mechanisms of CaO/SiO_2 and MgO/Al_2O_3 on slag viscous behavior were expounded using the X-Ray Diffraction (XRD); Fourier transformation infrared spectroscopy (FTIR) and the Factsage. The results show that; with the increasing of CaO/SiO_2 from 1.10 to 1.30， the viscosity at 1773 K decrease from 0.316 Pa·s to 0.227 Pa·s ,the T_{Br} and E_η increase from 1534 K and 117.01 $kJ\cdot mol^{-1}$ to 1583 K and 182.86 $kJ\cdot mol^{-1}$ respectively. With the increasing of MgO/Al_2O_3 from 0.40 to 0.65， the viscosity at 1773 K and the T_{Br} decrease from 0.290 Pa·s and 1567 K to 0.208 Pa·s and 1542 K respectively. The deterioration of slag behaviors is due to the increase of polymerization degree of complex viscous units in the slag. Ultimately; when CaO/SiO_2 at 1.25 and MgO/Al_2O_3 at 0.55; the viscous behaviors of slag are better.

Keywords: high alumina slag; viscous behavior; activation energy; slag structure; MgO/Al_2O_3; CaO/SiO_2

1. Introduction

It is one of the important measures that ironmaking enterprises reduce cost and increase economic efficiency by utilization of cheap bentonite and high alumina ore in blast furnace smelting process. However, excessive Al_2O_3 in the slag can deteriorate viscous behavior of the slag, permeability of the blast furnace cohesive zone, and make it difficult to separate slag and iron. B_2O_3 in the blast furnace slag can improve its viscous behavior**Error! Reference source not found.**. Adding the appropriate amount of CaO and MgO to the slag will reduce the degree of slag polymerization and improve viscous behavior of the slag. However, excessive CaO and MgO will increase the slag amount and energy consumption. It is not conducive to energy conservation and emission reduction. Therefore, it is necessary to study the effect of CaO/SiO_2 and MgO/Al_2O_3 on the viscous flow behaviors of low boron-bearing high alumina blast furnace slag.**Error! Reference source not found.**

For recent years, some researches about the viscous flow behaviors of boron-containing slag had been reported. Li et al. [4] studied the roles of MgO and Al_2O_3 in the viscous and structural behavior of $CaO-MgO-Al_2O_3-SiO_2$-10 mass pct FeO with C/S = 1.4 slag. They found the MgO can prompt the depolymerization of the silicate network structure and reduce the viscosity of slag. The Al_2O_3 content to greater than 10 mass pct has the opposite effect on viscosity, as a result of the polymerization of the silicate network structure. Liang et al. [5] studied the effects of CaO/SiO_2 and MgO on the metallurgical properties of $CaO-SiO_2-MgO-Al_2O_3-TiO_2$ blast furnace slag system. The results showed when CaO/SiO_2 increased from 1.10 to 1.30, the polymerization degree of viscous units in the slag decreased, the η and E_η decreased, the T_{Br} increased; when the mass fraction of MgO increases from 6.0% to 12.0%, η decreased, the T_{Br} and E_η decreased first and then increased. Gao et al. [6] studied the effects of basicity and MgO content on the viscosity of SiO_2-CaO-MgO-9wt%Al_2O_3 slags with basicity from 0.4 to 1.0 and MgO content from 13wt% to 19wt%. They found the viscosity is strongly dependent on the combined action of basic oxide components in the slag. In their study, increasing the basicity is found to be more effective than increasing the MgO content in decreasing the viscosity of the slag. At higher temperatures, the increase of basicity or MgO content does not appreciably decrease the viscosity of the slag, as it does at lower temperatures. Huang et al. [7] studied the influence of B_2O_3 on the viscosity and degree of polymerization of SiO_2-30wt%Al_2O_3-B_2O_3-12 wt%Na_2O-CaO slag system. The results show that the degree of polymerization of slag decreases, the viscosity and break point temperature of slag decrease with the increase of B_2O_3. The above studies mainly focus on the influence of MgO、Al_2O_3、basicity and B_2O_3 et al. as single factors on the structure and viscosity in different types of slag systems. The effect of CaO/SiO_2 and MgO/Al_2O_3 on the viscosity behavior of low boron-bearing high alumina slag system has not been studied in detail.

In this paper, we verify the effect of CaO/SiO_2 and MgO/Al_2O_3 on the viscous behavior of low boron-bearing high alumina slag. The viscosity, the break temperature and the activation energy of the viscous flow of the slag are considered in detail. Firstly, a series of experiments were conducted to measure the viscosity of the slag. Subsequently, the effect mechanisms of CaO/SiO_2 and MgO/Al_2O_3 on low boron-bearing high alumina slag viscous behavior were expounded using the XRD and FTIR. Then the Factsage was adopted to demonstrate the liquidus temperature of the slags. Finally, the boron-bearing high alumina blast furnace slag with better performance was obtained, which provides guidance for the actual industrial production of the ironmaking company.

2. Experimental

2.1. Raw Material

Based on the on-site BF slag compositions, the slag samples for experiments were synthesized with the analytical reagent oxides of CaO, SiO_2, MgO and Al_2O_3. The basicity of the BF slag is 1.25, the Al_2O_3 is 17.00 %, the B_2O_3 is 0.47 %, and the MgO/Al_2O_3 is 0.47, which belongs to the boron-bearing high alumina blast furnace slag. The chemical compositions of slag samples are listed in Table 1. The experimental slag samples were synthesized by adding the analytical-grade oxides with the site blast furnace slag as the reference slag. In order to improve the accuracy of the experiment, the furnace slag and the analytical-grade oxides were roasted, mixed evenly and put into the molybdenum crucible, then pre-melted under the Argon atmosphere to stabilize the temperature and homogenize the compositions. The pre-melted slag samples were used for the determination of viscous flow behaviors**Error! Reference source not found.**. The experimental scheme shown in Table 2. In the series-1, keeping the MgO 7.98%、the Al_2O_3 17.00% and B_2O_3 3.83% in the slag and increase the CaO/SiO_2 from 1.10 to 1.30. In the series-2, keeping the CaO/SiO_2 radio 1.25 in the slag and increase the MgO/Al_2O_3 from 0.40 to 0.65.

Table 1. Chemical composition of base blast furnace slag (mass fraction/%).

CaO	SiO_2	MgO	Al_2O_3	B_2O_3
38.01	30.37	7.98	17.00	0.47

Table 2. Experimental scheme (mass fraction/%).

NO.	CaO	SiO_2	MgO	Al_2O_3	B_2O_3	CaO/SiO_2	MgO/Al_2O_3
1	35.03	31.85	7.98	17.00	0.47	1.10	0.47
2	35.77	31.11	7.98	17.00	0.47	1.15	0.47

3	36.48	30.40	7.98	17.00	0.47	1.20	0.47
4	37.15	29.72	7.98	17.00	0.47	1.25	0.47
5	37.80	29.08	7.98	17.00	047	1.30	0.47
6	37.81	30.25	6.80	17.00	0.47	1.25	0.40
7	37.74	29.87	7.65	17.00	0.47	1.25	0.45
8	36.87	29.49	8.50	17.00	0.47	1.25	0.50
9	36.39	29.11	9.35	17.00	0.47	1.25	0.55
10	35.92	28.74	10.20	17.00	0.47	1.25	0.60
11	35.45	28.36	11.05	17.00	0.47	1.25	0.65

2.2. Experimental Procedure

The viscosity-temperature (η-T) curves of slag were acquired on the RTW-10 melt property tester by the rotating cylinder method. Figure 1 shown the schematic diagram of the experimental device, which consisted of a heating system, a rotating system, a measuring system, a control system and an atmosphere system.

The crucible containing the experimental slag sample was placed on the graphite base and heated to 1500 °C with the furnace temperature, and then the temperature was held for 30 min. During the constant temperature, the molybdenum probe was used to stir the slag to homogenize the chemical compositions. When the temperature stabilized, the viscosity was measured at a speed of 200r/min. In the process of viscosity measurement, Argon gas was injected into the furnace tube at the flow rate of 1.5 L/min to maintain an inert atmosphere. The measurement results were recorded by the control system. When the viscosity reached about 3.5 Pa·s, the measurements were ended. The experiment was repeated twice. The quenched experimental slags and the natural cooling slags were crushed and grinded to facilitate the analysis by FTIR and XRD.

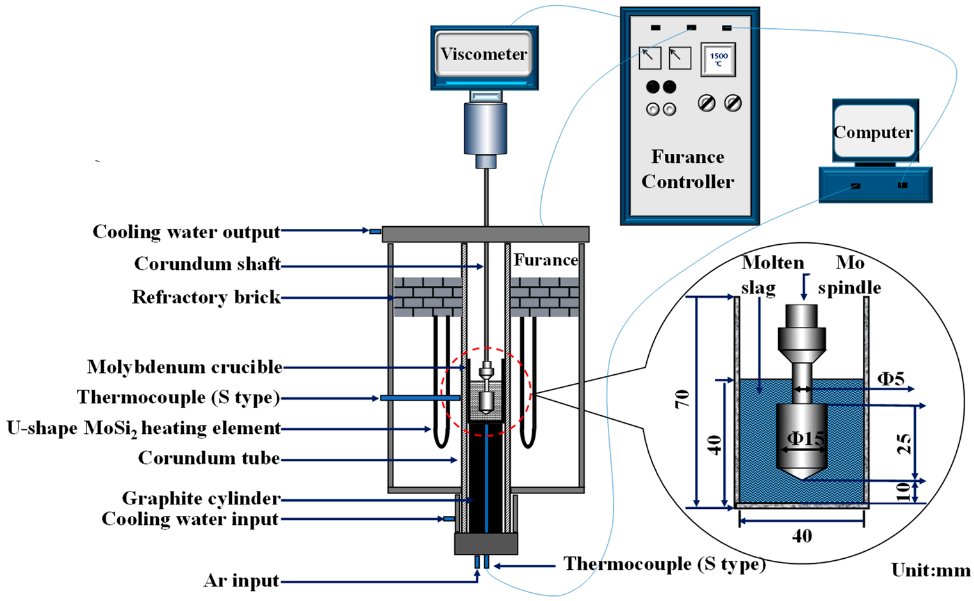

Figure 1. Schematic diagram of the experimental apparatus.

This article defines the viscosity of slag at 1773 K as high-temperature viscosity (η_{1773K}). Making 135° straight line is tangent to the viscosity-temperature curve(η-T). The temperature corresponding to the tangent point is defined as the T_{Br} of the slag, as shown in Figure 2.

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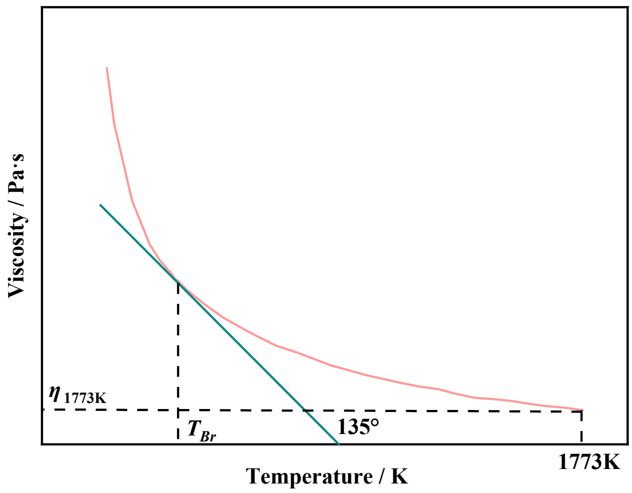

Figure 2. Slag viscosity curve and T_{Br} determination diagram.

3. Results and Discussion

Different CaO/SiO_2 and MgO/Al_2O_3 slag were obtained through experiments. The *η*-*T* curve is shown in Figure 3. The viscosity increases with decreasing of temperature and there is a clear turning point on every *η*-*T* curve.

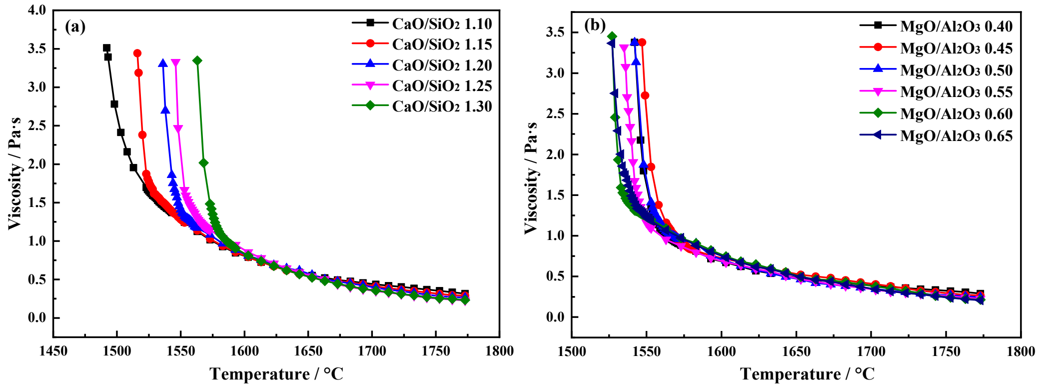

Figure 3. *η*-*T* curves of experimental slags with different CaO/SiO_2 and MgO/Al_2O_3.(a) - CaO/SiO_2; (b) - MgO/Al_2O_3.

3.1. Effects of CaO/SiO_2 and MgO/Al_2O_3 on the Viscous Behaviors of the Slag

3.1.1. Effects of CaO/SiO_2 on the Viscous Behaviors of the Slag

The effects of different CaO/SiO_2 on slag viscosity are shown in Figure 4(a). When the temperature increases from 1733 to 1773 K, with the increasing of CaO/SiO_2 from 1.10 to 1.30, the *η* of the slag decreases significantly first and then slows down. When CaO/SiO_2 is 1.25, the minimum of η_{1773K} is 0.227 Pa·s.

The viscosity of slag is mainly affected by the internal network structure. The $Si_xO_y^{z-}$ and $Al_xO_y^{z-}$ tetrahedral structures are the main structural units of the slag. The complex network structure inside the slag can be depolymerized to reduce viscosity. As CaO/SiO_2 increases in the slag, the viscosity will decrease. The reason is that the free oxygen ion O^{2-} dissociated from basic oxide CaO can interact with bridging oxygen O in the network structure of aluminosilicate to form non-bridging oxygen O^- [9–11], resulting in the aluminate silicate network structure being depolymerized into smaller network units. By the viscosity module of FactSage prediction, the theoretical viscosity at 1773 K are 0.314, 0.296, 0.281, 0.268, and 0.256 Pa·s respectively. The trend of change is the same as that of the experiment, providing theoretical support for the experimental results.

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In order to elucidate the relationship between slag viscosity and internal structure, FTIR is employed to analyse the slag of different CaO/SiO_2. The FTIR of silicate aluminate slag is generally divided into three regions, and the wave number range is 400-600 cm^{-1}, 600-800 cm^{-1} and 800-1200 cm^{-1}, which respectively corresponds to T-O-T (T represents Si or Al) bending vibration, $[AlO_4]^{5-}$ tetrahedral asymmetric tensile vibration and $[SiO_4]^{4-}$ tetrahedral symmetric tensile vibration [12–14]. The FTIR analysis results of different CaO/SiO_2 are shown in Figure 5(a). As CaO/SiO_2 increases from 1.10 to 1.30, the transmitted wave valley of the $[SiO_4]^{4-}$ tetrahedral symmetric stretching vibration band shifts towards lower wave numbers, and the bandwidth widens, indicating the simplification of the aluminosilicate network structure. Gaussian deconvolution was performed on Si-O axisymmetric vibration bands with CaO/SiO_2 ratios of 1.10 and 1.30, and the corresponding areas of each peak are used to characterize the corresponding amount of Q_i (i=0~3). Q_0, Q_1, Q_2, and Q_3 represent the structures of SiO_4^{4-}, $Si_2O_7^{6-}$, $Si_2O_6^{4-}$, and $Si_2O_5^{2-}$[15–17]. The smaller the value of i, the simpler the slag structure and the higher the degree of polymerization. In brief, when CaO/SiO_2 increases from 1.10 to 1.30, the silicate and aluminate network structure in the slag are depolymerized, resulting in the reduction of the η in the slag.

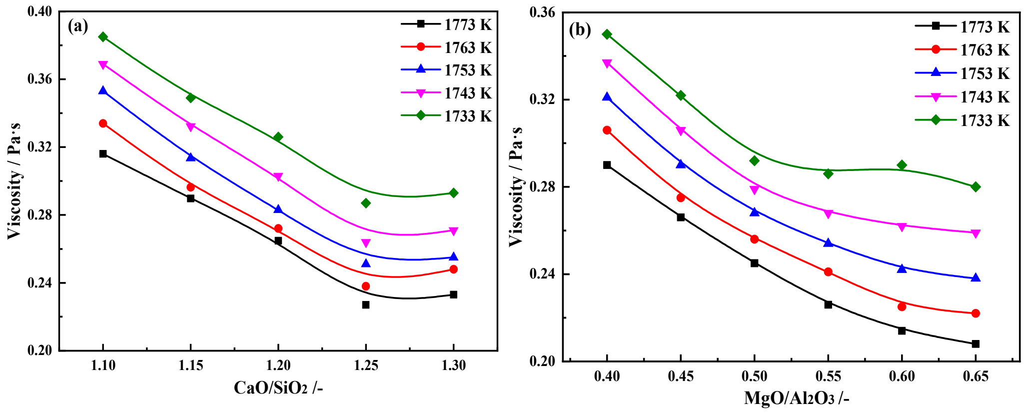

Figure 4. Viscosity of different CaO/SiO_2 and MgO/Al_2O_3 experimental slags at 1733-1773 K.(a) - CaO/SiO_2; (b) - MgO/Al_2O_3.

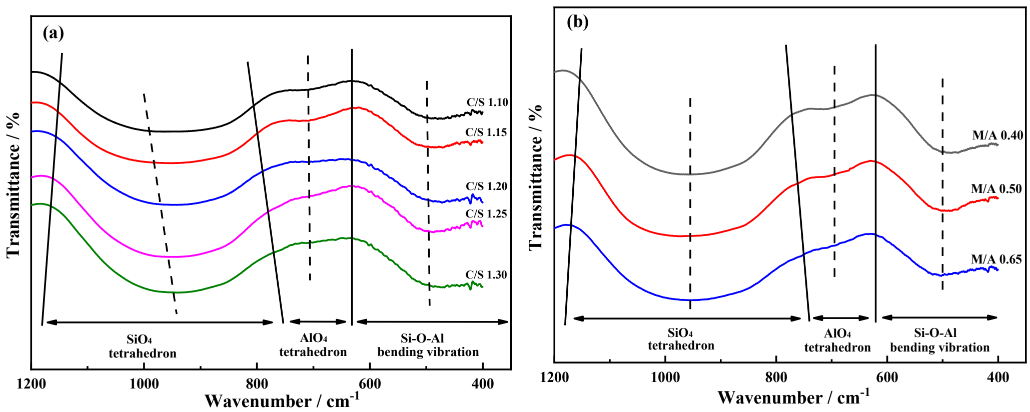

Figure 5. FTIR of experimental slag with different CaO/SiO_2 and MgO/Al_2O_3 (a) - CaO/SiO_2; (b) - MgO/Al_2O_3.

3.1.2. Effects of MgO/Al_2O_3 on the Viscous Behaviors of the Slag

The effect of different MgO/Al_2O_3 on slag viscosity are shown in Figure 4(b). When the temperature increases from 1733 to 1773 K, with the increasing of CaO/SiO_2 from 0.40 to 0.65, the η of the slag decreases significantly first and then slows down. When MgO/Al_2O_3 is 0.55, the η_{1773K} is 0.226 Pa·s.

The effect mechanism of MgO/Al_2O_3 on slag viscosity is similar to the CaO/SiO_2. Both MgO and CaO are basic oxide, and the dissociated O^{2-} ions promote the depolymerization of complex structures. By the viscosity module of FactSage prediction, when MgO/Al_2O_3 increases from 0.40 to 0.65, the theoretical viscosity at 1773 K are 0.281, 0.272, 0.263, 0.254, 0.247 and 0.239 Pa·s respectively. The trend of change is the same as that of the experiment, providing theoretical support for the experimental results.

In order to elucidate the relationship between slag viscosity and internal structure, FTIR is employed to analyse the slag of different MgO/Al_2O_3. The FTIR analysis results of different MgO/Al_2O_3 slag are shown in Figure 5(b). When MgO/Al_2O_3 increases from 0.40 to 0.65, the depth of $[SiO_4]^{4-}$ tetrahedral symmetric tensile vibration becomes shallower and the bandwidth becomes wider, indicating an increase in the distance between Si-O bonds and the disintegration of the slag silicate network structure into smaller network units; The depth of $[AlO_4]^{5-}$ tetrahedron asymmetric tensile vibration band gradually becomes shallow, and finally almost disappears, indicating the aluminate network structure in the slag is depolymerized; The groove depth of the Si-O-Al bending vibration band is slightly weakened, indicating a decrease in the number of Si-O-Al structures used to connect $[AlO_4]^{5-}$ and $[SiO_4]^{4-}$ tetrahedra [18–20]. In conclusion, the silicon Aluminate network structure in the slag is depolymerized, resulting in the reduction of the η in the slag.

3.2. Effects of CaO/SiO_2 and MgO/Al_2O_3 on the Break Point Temperature of Slag

3.2.1. Effects of CaO/SiO_2 on the Break Point Temperature of Slag

The effect of different CaO/SiO_2 on the break point temperature is shown in Figure 8(a). When CaO/SiO_2 increases from 1.10 to 1.30, the T_{Br} shows an uptrend, increasing from 1534 K to 1583 K.

The phase diagram of the five-component slag system CaO-SiO_2-MgO-17.00%Al_2O_3-3.83%B_2O_3 as plotted by the phase diagram module in the FactSage is shown in Figure 9. The different CaO/SiO_2 components are located in the crystalline region of the pyrochlore. With the increasing of CaO/SiO_2, the liquid temperature of the slag increases and the ability to crystallize at high temperatures becomes stronger, leading to an increase in T_{Br}. The liquid temperatures were 1613.92, 1623.05, 1631.00, 1637.96 and 1643.76 K respectively. The liquidus temperature of slag increased. Thus, the crystallization capacity of slag is enhanced and the T_{Br} also increases. These results are agreement with the trend of the measurements of T_{Br}.

The XRD analysis results of different CaO/SiO_2 slag are shown in Figure 11(a). The basic phase in different CaO/SiO_2 slag is melilite. When CaO/SiO_2 increases from 1.10 to 1.30, the diffraction peak intensity of melilite, spinel, and $Ca_2B_2O_5$ phases increases. When CaO/SiO_2 is 1.15, the $Mg_3(BO_3)_2$ phase disappears and pyroxene phase appears in the slag. The number of high melting point phases in the slag increase relatively and the crystallization ability of the slag increases under high temperature conditions, resulting in an increase in the T_{Br} and a decrease in fluidity.

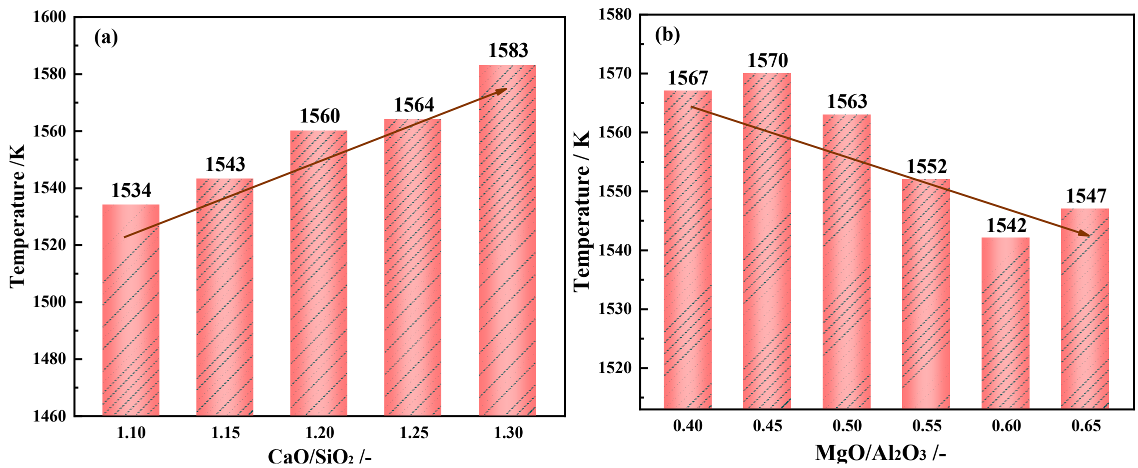

Figure 8. T_{Br} of experimental slags with different CaO/SiO_2 and MgO/Al_2O_3 (a) - CaO/SiO_2; (b) - MgO/Al_2O_3.

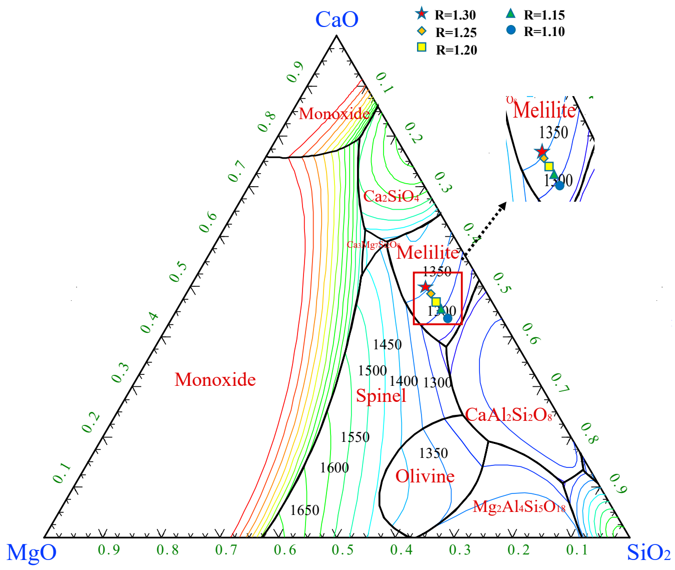

Figure 9. Phase diagram of CaO-SiO_2-MgO-17.00%Al_2O_3-3.83%B_2O_3 blast furnace slag with different CaO/SiO_2.

3.2.2. Effects of MgO/Al_2O_3 on the Break Point Temperature of Slag

The effect of different MgO/Al_2O_3 on the break point temperature is shown in Figure 8(b). With the increasing of MgO/Al_2O_3 from 0.40 to 0.65, the T_{Br} of the slag shows a downtrend, decreasing from 1570 K to 1542 K.

The phase diagram of CaO-SiO2-7.98%MgO-Al_2O_3-3.83%B_2O_3 slag system is caculated by the Phase Diagram module in FactSage. As shown in the Figure 10, with the continuous decreasing of MgO/Al_2O_3, the composition of the slags is located in the area of melilite phase, and the liquidus temperature of melilite is relatively sparse, which demonstrates the phase is stable. According to FactSage, the liquidus temperature of slag with different MgO/Al_2O_3 was calculated as 1345.83, 1349.95, 1354.95, 1360.93, 1367.64, 1374.78 °C, and the liquidus temperature of slag increased. Thus, the crystallization capacity of slag is enhanced and the T_{Br} also increases. These results are agreement with the trend of the measurements of T_{Br}.

The XRD analysis results of different MgO/Al_2O_3 slag are shown in Figure 11(b). There are melilite, spinel, pyroxene, $Ca_2B_2O_5$ and $Mg_3(BO_3)_2$ in the slag and the melilite is the basic phase. When MgO/Al_2O_3 increases from 0.40 to 0.65, the diffraction peak intensity of melilite, spinel, pyroxene and $Mg_3(BO_3)_2$ gradually weakens, while the diffraction peak intensity of $Ca_2B_2O_5$ and pyroxene slightly increases. This indicates that the $Ca_2B_2O_5$ and pyroxene in the slag are relatively increased, while the number of high melting point phases is relatively reduced, resulting in a decrease in the crystallization ability of the slag, a decrease in the T_{Br} and an improvement in fluidity under high temperature conditions.

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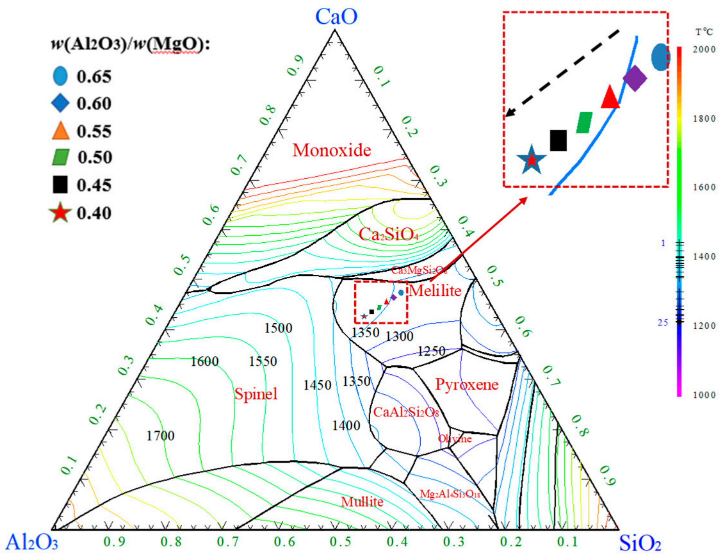

Figure 10. Phase diagram of five-element slag system CaO-SiO_2-7.98%MgO-Al_2O_3-3.83%B_2O_3 with different MgO/Al_2O_3.

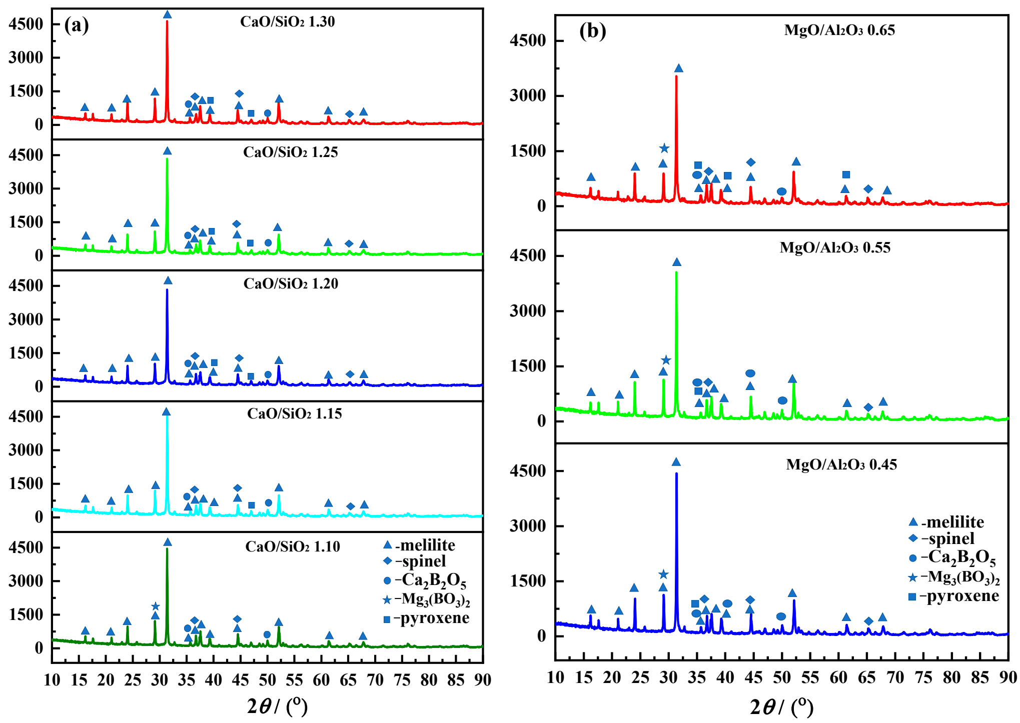

Figure 11. XRD analysis of different CaO/SiO_2 and MgO/Al_2O_3 experimental slags. (a) - CaO/SiO_2; (b) - MgO/Al_2O_3.

3.3. Effects of CaO/SiO_2 and MgO/Al_2O_3 on Activation Energy of Slag Viscous Flow

Viscous flow activation energy is a crucial viscosity characteristic of slag. The E_η reflects the sensitivity of slag viscosity to temperature, representing the thermostability of slag [21–23]. The calculation of viscous flow activation energy in this article adopts the modified Weymann-Frenkel equation by Urban, as shown in formula (1). Formula (2) can be obtained by taking the logarithm of both sides formula (1). The viscosity data measured in the experiment are calculated using linear regression method, and the slope is E_η. Linear fitting results and the trend of E_η with different CaO/SiO_2 and MgO/Al_2O_3 are shown in Figures 12 and 13. The results indicate that there is a fine

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linear relationship between ln(η/T) and $1/T$. The linear correlation coefficients are all greater than 0.99.[24,25]

$$\eta = ATexp\left(\frac{E_\eta}{RT}\right) \tag{1}$$

$$\ln\left(\frac{\eta}{T}\right) = \ln A + \frac{E_\eta}{R} \times \frac{1}{T} \tag{2}$$

where, η is the viscosity, Pa·s; A is the proportionality constant; T is the temperature, K; R is the gas constant, 8.314 J·(mol·K)$^{-1}$.

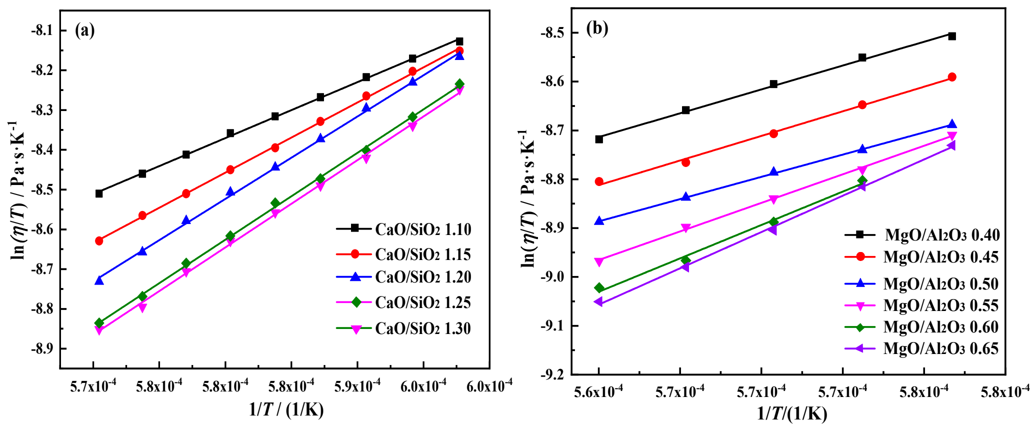

Figure 12. Fitting results of experimental slag ln(η/T) and $1/T$ for different CaO/SiO_2 and MgO/Al_2O_3 (a) - CaO/SiO_2; (b) - MgO/Al_2O_3.

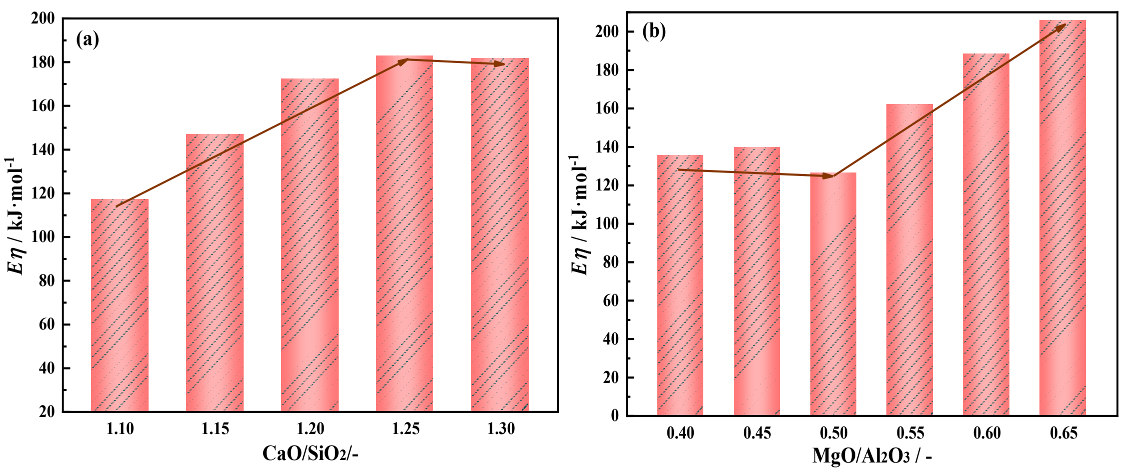

Figure 13. Variation of E_η for different CaO/SiO_2 and MgO/Al_2O_3 experimental slags (a) - CaO/SiO_2; (b) - MgO/Al_2O_3.

3.3.1. Effects of CaO/SiO_2 on Activation Energy of Slag Viscous Flow

As shown in Figure 13(a), when CaO/SiO_2 increases from 1.10 to 1.30，the E_η increase from 117.01 to 182.86 kJ·mol^{-1}. This indicates that the sensitivity of slag viscosity to temperature is weakened. On the premise of ensuring better slag stability, the CaO/SiO_2 value of 1.25 is reasonable. From the perspective of slag structure, the complex slag structure is decomposed into simpler structure, the activation energy of slag is increased, and the stability is improved[26–28]. The stability of the slag can also be characterized by the density of the isotherm in the phase diagram. The thinner the contour lines temperature and related subjects, the less the temperature affects the slag composition and the better the slag stability.

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3.3.2. Effects of MgO/Al_2O_3 on Activation Energy of Slag Viscous Flow

As shown in Figure 13(b), when MgO/Al_2O_3 increases from 0.40 to 0.50, the E_η chang inconspicuously. The E_η increase from126.20 to 205.86 $kJ \cdot mol^{-1}$. When MgO/Al_2O_3 increases from 0.50 to 0.65, th the E_η significant increase. This indicates that the thermostability of slag to temperature is enhanced. The complex slag structure is decomposed into simpler structure, the activation energy of slag is increased.

As shown in Figures 9 and 10, within the experimental value range, the isotherm becomes sparse with the increase of CaO/SiO_2 and MgO/Al_2O_3, indicating a better stability of the slag, which is consistent with the experimental fitting results [29,30].

Briefly, when CaO/SiO_2 is 1.25, η_{1773K} has a minimum of 0.227 Pa·s, a lower T_{Br} is 1570 K, E_η is stable at a lower level, the slag has a good thermal stability performance. When MgO/Al_2O_3 is 0.55, the decreasing trend of η_{1773K} begins to slow down to 0.226 Pa·s, T_{Br} and E_η are 1570 K and 161.99 $KJ \cdot mol^{-1}$ respectively. Overall, when CaO/SiO_2 is 1.25 and MgO/Al_2O_3 is 0.55, a good metallurgical properties of low boron-bearing high alumina slag system can be obtained, providing a good reference basis for blast furnace operation.

4. Conclusions

(1) With CaO/SiO_2 increasing from 1.10 to 1.30, viscosity first decrease significantly and then slowed down. When CaO/SiO_2 is 1.25, η_{1773K} is 0.227 Pa·s. T_{Br} shows an increasing trend, increasing from 1534 K to 1583 K. E_η increase from 117.01 to 182.86 $kJ \cdot mol^{-1}$ and the thermal stability of the slag deteriorates first and then improves. At this point, the slag system has a better performance.

(2) With MgO/Al_2O_3 increasing from 0.40 to 0.65, viscosity first decrease significantly and then slowed down. When MgO/Al_2O_3 is 0.55, η_{1773K} is 0.226 Pa·s. T_{Br} decrease from 1570 K to 1542 K. The E_η increase from 126.20 to 205.86 $kJ \cdot mol^{-1}$ and the thermal stability of the slag first improves and then deteriorates. At this point, the slag system has a better performance.

(3) Comprehensive considerations, when CaO/SiO_2 is 1.25 and MgO/Al_2O_3 is 0.55, the η1773K, T_{Br} and E_η are at a reasonable value. The low boron-bearing high alumina slag systems has the best metallurgical performance at this value.

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