

Article

The Desulfurization Ability of a High Al_2O_3 Type $\text{CaO-SiO}_2\text{-Al}_2\text{O}_3\text{-MgO-TiO}_2$ Blast Furnace Slag System at 1823 K

Yongchun Guo ^{1,2,*}, Mengyao Li ¹ and Zhimin Ding ²

¹ School of Aeronautical Manufacture Engineering, Xi'an Aeronautical Polytechnic Institute, Xi'an 710089, China; lms0612@126.com

² School of Metallurgy, Northeastern University, Shenyang 110819, China; naires@126.com

* Correspondence: gycneu@163.com; Tel.: +86-18329702808

Abstract

In this study, $\text{CaO-SiO}_2\text{-Al}_2\text{O}_3\text{-MgO-TiO}_2$ slag was used as the research object to simulate the blast furnace ironmaking process. Based on the experimental data, the influences of basicity ($R(w(\text{CaO})/w(\text{SiO}_2)))$ and the magnesia–alumina ratio ($w(\text{MgO})/w(\text{Al}_2\text{O}_3)$) on desulfurization ability are discussed. Additionally, the influences of dissimilarity, basicity, and the magnesia–alumina ratio on slag structure were analyzed using Fourier transform infrared spectroscopy (FT-IR). The results show that when $w(\text{Al}_2\text{O}_3) = 20\%$ and $w(\text{MgO})/w(\text{Al}_2\text{O}_3) = 0.50$, sulfide capacity ($\lg C_s$) accretion with the increment in R . Moreover, when $w(\text{Al}_2\text{O}_3) = 20\%$ and $R = 1.30$, sulfide capacity accretion with the increment in $w(\text{MgO})/w(\text{Al}_2\text{O}_3)$. Fourier transform infrared spectroscopy was used to confirm that, with increasing basicity and the magnesia–alumina ratio, the concentration of dissociated free oxygen ions (O^{2-}) in slag increases, and these ions interact with the bridging oxygen (O^0) of silicate to depolymerize the complex Si-O structure into simpler units.

Keywords: blast furnace (BF); sulfide capacity; slag structure; R ; $w(\text{MgO})/w(\text{Al}_2\text{O}_3)$

1. Introduction

Steel is the most important engineering material in the global economy. Vanadium titanomagnetite has large global reserves and is a major raw material for ironmaking. The large-scale manufacture of steel is inevitably accompanied by a large amount of slag [1–3]. An increase in the use of high- Al_2O_3 iron ore resulted in a significant increase in Al_2O_3 content in blast furnace slag, which in turn increased its viscosity, poor fluidity, and a reduction in its desulfurization ability [4–9].

Much research, through experiments, has been conducted on the thermodynamic properties of slag systems. Tanaka T [10] attempted to use fusional $\text{CaO-Al}_2\text{O}_3$ and $\text{CaO-SiO}_2\text{-MgO-Al}_2\text{O}_3$ slag systems to carry out desulfurization of molten iron and carbon-saturated molten iron. Liao J L [11] used a $\text{MgO-saturated CaO-SiO}_2\text{-CaF}_2\text{-Na}_2\text{O}$ quaternary slag system in a hot metal desulfurization experiment. Additionally, the sulfur content of $\text{CaO-SiO}_2\text{-MnO}$ slag across the entire composition range was determined by Park G H [12] using the gas–slag equilibrium approach, and the impacts of alkalinity and the sulfide activity coefficient on the sulfur capacity of molten slag were investigated. Taniguchi Y [13] studied the sulfidation capacity of $\text{CaO-Al}_2\text{O}_3\text{-SiO}_2\text{-MgO-MnO}$ slags in the temperature range of 1673–1773 K. Based on modified optical basicity, Hao X [14] established the sulfide capacity model of a $\text{CaO-Al}_2\text{O}_3\text{-SiO}_2\text{-MgO}$ slag system, with the average deviation of this model in an alkaline Al_2O_3 slag system being 3.23%, significantly inferior to that of



Academic Editor: George D. Verros

Received: 9 January 2026

Revised: 9 February 2026

Accepted: 9 February 2026

Published: 13 February 2026

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other formers. Additionally, the application scope of the model used was given. Shankar A [15] studied the desulfurization ability of CaO-SiO₂-MgO-Al₂O₃ slag and found that the desulfurization ability of slag increased with the increment in R and $w(\text{MgO})$. Similarly, Ma et al. [16,17] measured the desulfurization ability of molten slag at 1773~1823 K. In their study, the desulfurization ability of molten slag accretion with the increment in R or $w(\text{MgO})$. Condo A [18] studied the desulfurization ability of molten slag at 1713 K, 1743 K, and 1773 K, respectively, discovering that when $w(\text{MgO}) > 14\%$, the effect of MgO content on the desulfurization ability of slag is reduced; that is, the $w(\text{MgO})$ of a blast furnace slag system should be less than 14%. Wang's research shows that increasing the contents of both MnO and Mn was found to improve the removal efficiency of slag, primarily due to the reduction in the slag viscosity [19]. Song's research shows that the reason for the decrease in sulfur distribution ratio and sulfur capacity of the slag is that the overall alkalinity of the slag is reduced, and more Ca²⁺ is occupied by the charge compensation of [AlO₄] [20]. Liu's research shows that the sulfur partition ratio was derived from sulfide capacity, and the values of the sulfur partition ratio at basicity of 0.4 and 0.6 were much smaller than those at basicity of 0.8, 1.0, and 1.2, indicating a weak desulfurization ability of the slag with a low basicity [21]. Xin H, Wang Y, Kang Y B [22–24], and other scholars have additionally studied the desulfurization performance of slag. However, in their research, the problem of saturated vapor pressure was not considered in the experimental process. At present, we have conducted extensive research on the high-aluminum-oxide slag system and put forward a reasonable smelting system. However, there are relatively few studies on the titanium-bearing blast furnace slag system, particularly regarding the role of TiO₂ in protecting the furnace during the production process. Research on high-Al₂O₃-type slag systems is relatively scarce, and research explaining the desulfurization reaction mechanism of high-Al₂O₃-type blast furnace slag at the microstructure level of blast furnace slag is rare. Therefore, it is imperative to study the desulfurization theory at the microstructural level of blast furnace slag so as to provide scientific underpinnings and theoretical insights for the utilization of raw materials such as high-alumina iron ore in blast furnaces.

At a temperature condition of $T = 1823\text{ K}$, the desulfurization experiment was carried out. Based on experimental data, the effects of basicity and the magnesia–alumina ratio on the desulfurization ability are discussed in this thesis. Moreover, an investigation into the microstructure of molten slag was conducted, and the influence of different basicity and the magnesia–alumina ratio on the molten slag's structure was examined via FT-IR. This tool can better reveal the implications of chemical content and the metallurgical properties of blast furnace slag, providing a theoretical foundation for determining an appropriate slagging system.

2. Experimentation

2.1. Principle of Desulfurization Experiment

The desulfurization capability of molten slag primarily proceeds via the metastasis of sulfur from molten iron into the slag phase in the form of sulfide, through reactions between sulfur and alkaline oxides present in the slag. Figure 1 contains a schematic diagram of the experimental principle behind blast furnace slag desulfurization. According to the ion theory, the slag desulfurization reaction formula is as follows [25]:



$$\Delta G^{\theta} = 124,455 - 50.26T / (\text{J} \cdot \text{mol}^{-1}) \quad (2)$$

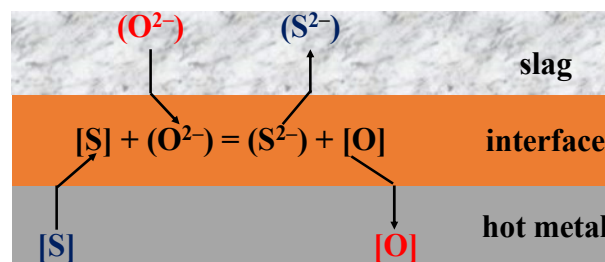


Figure 1. Desulfurization experimental theory diagram.

2.2. Experimental Scheme

The definition of sulfide capacity is defined as follows:

$$C_S = w(S)_{\%} \cdot \left(\frac{p_{O_2}}{p_{S_2}} \right)^{\frac{1}{2}} \quad (3)$$

where p_{O_2} and p_{S_2} are the partial pressures of oxygen and sulfur, respectively (atm).

The sulfur distribution ratio, L_S , is defined as follows:

$$L_S = \frac{w(S)_{\%}}{w[S]_{\%}} \quad (4)$$

In the formula, $w(S)_{\%}$ represents the sulfur content in slag, and $w[S]_{\%}$ represents the sulfur content in molten iron.

$$\frac{1}{2}S_2 = [S] \quad (5)$$

$$\lg K_5^{\theta} = \frac{7054}{T} - 1.224 \quad (6)$$

The equilibrium constant K_5 of Formula (5) can also be represented as follows:

$$K_5 = \frac{f_S \cdot w[S]_{\%}}{p_{S_2}^{\frac{1}{2}}} \quad (7)$$

where f_S is the Henrian activity coefficient of sulfur dissolved in the metal.

By using the equilibrium constant of Equation (7), Equation (8) can be deduced as follows:

$$p_{S_2}^{\frac{1}{2}} = \frac{w[S]_{\%} \cdot f_S}{K_5} \quad (8)$$

According to Equations (3), (4) and (8), the expression for sulfide capacity can be derived, as shown in Equation (9) below:

$$C_S = w(S)_{\%} \cdot \left(\frac{p_{O_2}}{p_{S_2}} \right)^{\frac{1}{2}} = w(S)_{\%} \cdot p_{O_2}^{\frac{1}{2}} \cdot \frac{K_5}{f_S \cdot w[S]_{\%}} = \frac{w(S)_{\%}}{w[S]_{\%}} \cdot p_{O_2}^{\frac{1}{2}} \cdot \frac{K_5}{f_S} = L_S \cdot p_{O_2}^{\frac{1}{2}} \cdot \frac{K_5}{f_S} \quad (9)$$

Sulfide capacity can also be written as Equation (10) below:

$$\lg C_S = \lg L_S + \frac{1}{2} \lg p_{O_2} + \lg K_5 - \lg f_S \quad (10)$$

The activity coefficient of sulfur in the metal phase may be determined using the following expression:

$$\lg f_s = \sum e_s^i \cdot [\%i] \quad (11)$$

where e_s^i is the interaction parameter of species i on S in the molten iron, and $[\%i]$ is the concentration of elements in hot metal. The interaction coefficient of e_s^i is shown in Table 1.

Table 1. Values of interaction parameters.

e_s^s	e_s^c	e_s^p	e_s^{Si}	e_s^{Mn}
0.028	0.113	0.029	0.063	0.026

The oxygen partial pressure in the furnace can be managed by the C/CO equilibrium, as expressed below:



$$\Delta G^\theta = -114,400 - 85.77T = -8.314T \ln K_{12} / (J \cdot mol^{-1}) \quad (13)$$

$$K_{12} = \frac{p_{CO}}{a_C \cdot p_{O_2}^{\frac{1}{2}}} \quad (14)$$

where a_C , the activity of carbon, is 1 when a pure substance standard is taken as the standard state.

$$K_{12} = \frac{p_{CO}}{p_{O_2}^{\frac{1}{2}}} \quad (15)$$

p_{CO} is the partial pressure of CO and is 1.0 atm in this examination ($p_{CO} = 1$), and $p_{O_2}^{\frac{1}{2}}$ can be accounted for using Formulation (15).

The equilibrium constant K_{12} can also be expressed as follows:

$$K_{12} = \frac{1}{p_{O_2}^{\frac{1}{2}}} \quad (16)$$

According to Equations (13) and (16), we can calculate the oxygen partial pressure.

2.3. Experimental Procedure

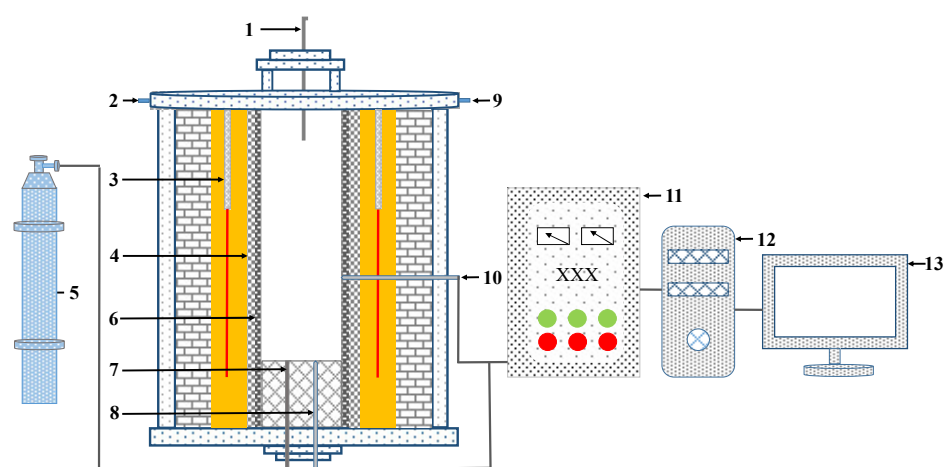
The entire process of desulfurization proof was conducted in a high-temperature tube furnace. The equipment is designed by Northeast University of China (Shenyang, China), and the equipment model is RTW-10. The high-temperature tube furnace used a proportional-integral-derivative controller to accurately control the temperature of the desulfurization experiment. The furnace is equipped with a resistance heating system, including an alumina reaction tube and a thermocouple for temperature measurement. The PID controller is used to control the temperature within ± 2 K. The slag composition set up in the desulfurization proof is shown in Table 2, and the pig iron composition used in this experimental study is shown in Table 3. Prior to initiation of the experiment, iron and pre-melted slag were collected as samples with a quality rate of 3:1. The pre-melted slag is shown in Figure 2. The desulfurization process is displayed in Figure 3. After the slag and iron samples were separated, the graphite plug switch is opened so that the molten iron flowing in the gap between the graphite plug and the crucible can flow through the slag blanket to the bottom of the diminished graphite crucible and deposit, as shown in Figure 4. Timing was initiated once the sample temperature in the tubular furnace reached 1823 K. The time of the equilibrium experiment is shown in Figure 5. As shown in Figure 5, the desulfurization capacity of slag has basically reached equilibrium after 70 min. After the sample was held at 1823 K for 1.5 h, preliminary experiments confirmed that it was sufficient to reach equilibrium, and the sample was quenched. All experiments were performed at 1823 K. Table 4 summarizes the chemical compositions of the experimental samples, the analyzed sulfur content of the resulting slag, and the calculated parameters for sulfide capacity.

Table 2. Chemical constitution table of molten slag (mass%).

No.	Chemical Constitution					R , -	$w(\text{MgO})/w(\text{Al}_2\text{O}_3)$, -
	CaO	SiO ₂	MgO	Al ₂ O ₃	TiO ₂		
1#	34.73	33.07	10.0	20	2.20	1.05	0.50
2#	36.27	31.53	10.0	20	2.20	1.15	0.50
3#	37.67	30.13	10.0	20	2.20	1.25	0.50
4#	38.95	28.85	10.0	20	2.20	1.35	0.50
5#	41.15	31.65	5.0	20	2.20	1.30	0.25
6#	40.02	30.78	7.0	20	2.20	1.30	0.35
7#	38.89	29.91	9.0	20	2.20	1.30	0.45
8#	37.76	29.04	11.0	20	2.20	1.30	0.55

Table 3. The content of corresponding chemical elements in pig iron.

$w(\text{C})$	$w(\text{Si})$	$w(\text{P})$	$w(\text{S})$	$w(\text{Mn})$	$w(\text{Fe})$
4.63	0.260	0.104	0.105	0.229	94.672

**Figure 2.** The pre-melted slag sample.**Figure 3.** Laboratory equipment: 1—gas export (carbon monoxide); 2—water import; 3—heating unit of MoSi₂; 4—Al₂O₃ tube; 5—carbonic oxide (CO); 6—graphite crucible; 7—gas inlet (CO); 8—30%Rh/Pt-60%Rh; 9—water outlet; 10—30%Rh/Pt-60%Rh; 11—temperature control system; 12—computer; 13—computer.

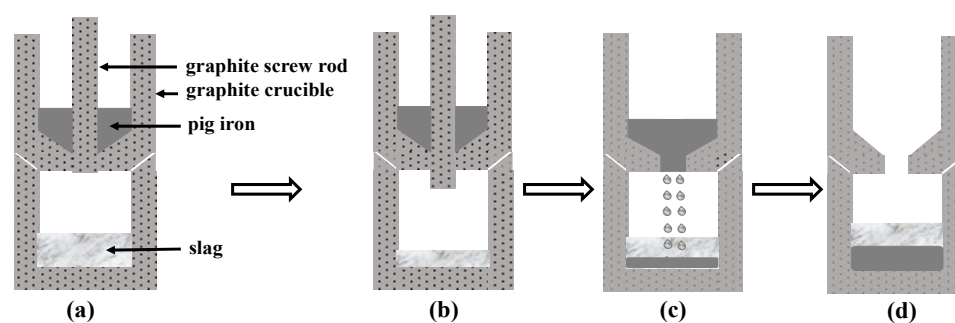


Figure 4. Experimental procedure: (a) assembly drawing; (b) slag; (c) pig iron melts and drips; (d) slag gold reaction.

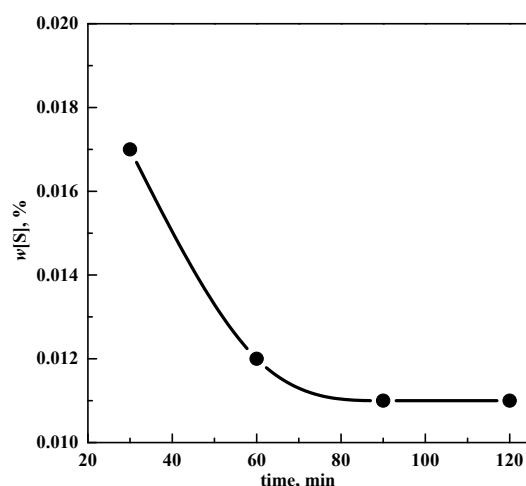


Figure 5. Discussion on desulfurization equilibrium time.

Table 4. Composition and experimental results of slag samples (mass %).

No.	Slag Composition					R , -	$w(\text{MgO})/w(\text{Al}_2\text{O}_3)$, -	$w(\text{S})$, -	$w[\text{S}]$, -	L_s , -	$\lg C_s$, -
	CaO	SiO ₂	MgO	Al ₂ O ₃	TiO ₂						
1#	34.73	33.07	10.0	20	2.20	1.05	0.50	0.316	0.016	19.152	−4.286
2#	36.27	31.53	10.0	20	2.20	1.15	0.50	0.319	0.014	23.474	−4.198
3#	37.67	30.13	10.0	20	2.20	1.25	0.50	0.332	0.012	28.492	−4.116
4#	38.95	28.85	10.0	20	2.20	1.35	0.50	0.348	0.011	32.660	−4.058
5#	41.15	31.65	5.0	20	2.20	1.30	0.25	0.313	0.016	18.971	−4.289
6#	40.02	30.78	7.0	20	2.20	1.30	0.35	0.326	0.015	22.386	−4.219
7#	38.89	29.91	9.0	20	2.20	1.30	0.45	0.344	0.014	25.297	−4.167
8#	37.76	29.04	11.0	20	2.20	1.30	0.55	0.343	0.012	29.428	−4.102

Fourier-transform infrared spectroscopy was performed via a TENSOR II infrared spectrometer obtained from Bruker Technology Co., Ltd., ZEISS, Oberkochen, Germany. The infrared spectrum is a result of both the transition of a molecule's vibration energy level after the sample absorbs a certain frequency of infrared radiation and the weakened light of the corresponding frequency in the transmitted beam, which results in a corresponding radiation intensity difference, thereby resulting in the corresponding infrared spectrum. Among the observed wavenumbers, one at 400~600 cm^{-1} corresponds to the Si-O-Al structure, another at 600~800 cm^{-1} corresponds to the $[\text{AlO}_4]$ structure, and another at 800~1200 cm^{-1} corresponds to the $[\text{SiO}_4]$ structure [26–28]. The samples were placed in a 50 mL beaker and dried in a dryer at 80 °C for 1 h. A weighed amount of the sample (50 mg) and 2 g of KBr powder were used, employing a mixer for even mixing. After mixing, 205 mg of the mixture was accurately weighed and placed in a tableting mold. After the cooled sample is removed, the slag and ferroalloy samples are carefully separated

and further sampled and analyzed. In addition, the S quantity in these samples was analyzed by ICP-AES (Shenyang, China).

3. Results and Discussion

In this thesis, the iron-making process of iron droplets passing through the slag layer at 1823 K was simulated by a double-layer graphite crucible. Table 4 shows the results of the desulfurization experiment.

3.1. Impact of Basicity on Desulphurization Ability of Molten Slag

Figure 6 shows the impact of basicity on the sulfur capacity and sulfur distribution ratio in slag when the ratio of magnesium to aluminum is 0.50, and the content of Al_2O_3 is 20%. As illustrated in Figure 6, with increasing basicity, the sulfur capacity in the slag increases from -4.286 to -4.058 , and the distribution ratio of sulfur increases from 19.152 to 32.66—an increase of 70%. Additionally, with the increase in basicity, the amount of MgO and Al_2O_3 in the slag remained basically unchanged, while the relative amount of SiO_2 decreased and the relative amount of CaO increased. With the growth in CaO content, the free oxygen ion in the slag growth, which promotes the forward reaction (1); the sulfur capacity and sulfur distribution ratio in the molten slag gradually increase; and the desulfurization ability of the slag is enhanced.

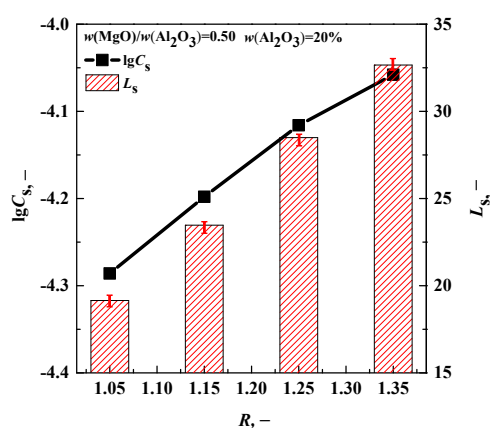


Figure 6. Effect of basicity on sulfur capacity.

When the ratio of magnesium to aluminum is 0.50, and the content of Al_2O_3 is 20%, the oil-cooled Fourier transform infrared spectroscopy of slag samples with different basicity is shown in Figure 7. From these results, it is possible to demonstrate that when the ratio of magnesium to aluminum is 0.50 and the content of Al_2O_3 is 20% in the slag, the relative strength of the Si–O–Al structure and $[\text{AlO}_4]$ did not change significantly with the growth in basicity, but the relative strength of $[\text{SiO}_4]$ decreased gradually at the wavenumbers between 800 and 1200 cm^{-1} , indicating that the polymerization of SiO_2 into complex silicate structures decreased. When the ratio of magnesium to aluminum is 0.50, and the content of Al_2O_3 is 20%, the content of CaO increases with the increase in basicity, and the number of oxygen ions in the slag gradually increases as well. The increase in oxygen ions interacted with the bridging oxygen in the silicate, causing the complex Si–O–Si bond to break. The complex silicon–oxygen ions in the slag disintegrated into simple silicon–oxygen ions ($\text{Si}_{n+1}\text{O}_{3n+4}^{2(n+2)-} + \text{O}^{2-} = \text{SiO}_4^{4-} + \text{Si}_n\text{O}_{3n+1}^{2n+2-}$). As a result, the complex $[\text{SiO}_4]$ became depolymerized, resulting in a growth in the fluidity of the molten slag and promoting the forward reaction $(\text{Ca}^{2+} + \text{O}^{2-}) + [\text{S}] = (\text{Ca}^{2+} + \text{S}^{2-}) + [\text{O}]$, thereby increasing the desulfurization capacity of the molten slag. Therefore, the desulfurization capacity of the molten slag increased with the growth in R from 1.05 to 1.35.

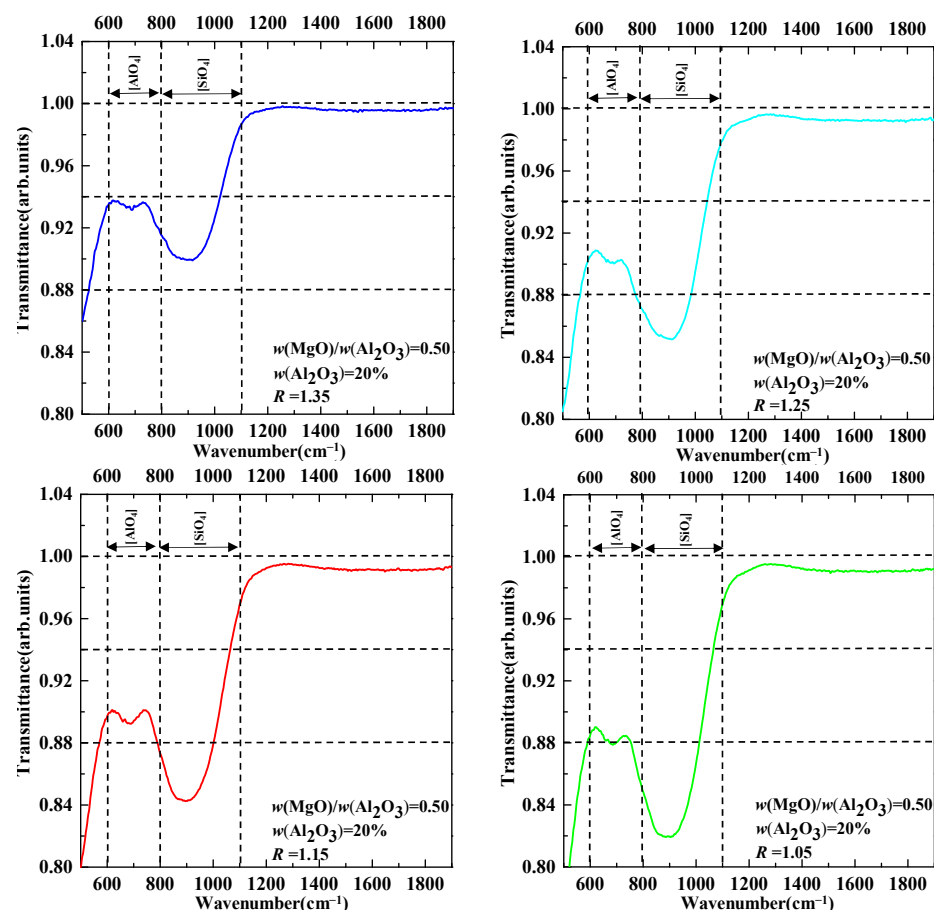


Figure 7. The effect of basicity on the slag sample on FT-IR results.

3.2. Impact of Magnesia–Alumina Ratio on Desulphurization Ability of Molten Slag

Figure 8 indicates the impact of the magnesia–alumina ratio on sulfur capacity and the sulfur distribution ratio in slag when the basicity is 1.30, and the content of Al_2O_3 is 20%. As illustrated in Figure 8, with the increase in magnesia–alumina ratio, the sulfur capacity in the slag increases from -4.286 to -4.102 , and the distribution ratio of sulfur increases from 19.152 to 32.66—an increase of 55%. With the increment in magnesia–alumina ratio, the relative content of MgO in the molten slag increment, and the number of free oxygen ions dissociated from MgO in the molten slag increment, which elevated the progress of the onward reaction (1). Moreover, the sulfur capacity and sulfur distribution increase, and the desulfurization ability of the molten slag is promoted.

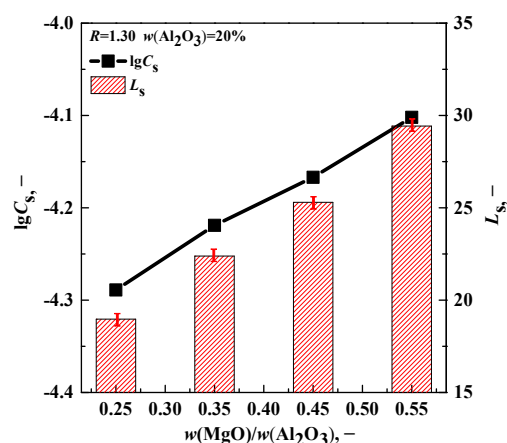


Figure 8. Effect of the magnesia–alumina ratio on sulfur capacity.

When the basicity is 1.30, and the content of Al_2O_3 is 20%, the oil-cooled Fourier transform infrared spectroscopy of slag samples with different magnesia–alumina ratios is displayed in Figure 9. As seen from the results of Fourier-transform infrared spectroscopy in Figure 9, with the elevate in magnesia–alumina ratio, the relative strength of the Si–O–Al structure and $[\text{AlO}_4]$ did not change significantly with the elevate in basicity, but the relative strength of $[\text{SiO}_4]$ decreased gradually at the wave number of 800–1200 cm^{-1} , which indicated that the polymerization of SiO_2 into complex silicate structures decreased. When the basicity is 1.30, and the content of Al_2O_3 is 20%, the content of MgO in the molten slag increased with an increase in the magnesia–alumina ratio. MgO is an alkaline oxide that can dissociate free oxygen ions, and the dissociated free oxygen ions interact with the bridging oxygen in the silicate, breaking the complex Si–O–Si bond in the molten slag and depolymerizing the complex $[\text{SiO}_4]$. This simplifies the network structure of the molten slag, resulting in an increase in the fluidity of the molten slag and promoting the forward reaction $(\text{Mg}^{2+} + \text{O}^{2-}) + [\text{S}] = (\text{Mg}^{2+} + \text{S}^{2-}) + [\text{O}]$, thus increasing the desulfurization capacity of the molten slag. Therefore, the desulfurization capacity of the slag increased, with an increase in the magnesia–alumina ratio from 0.25 to 0.55.

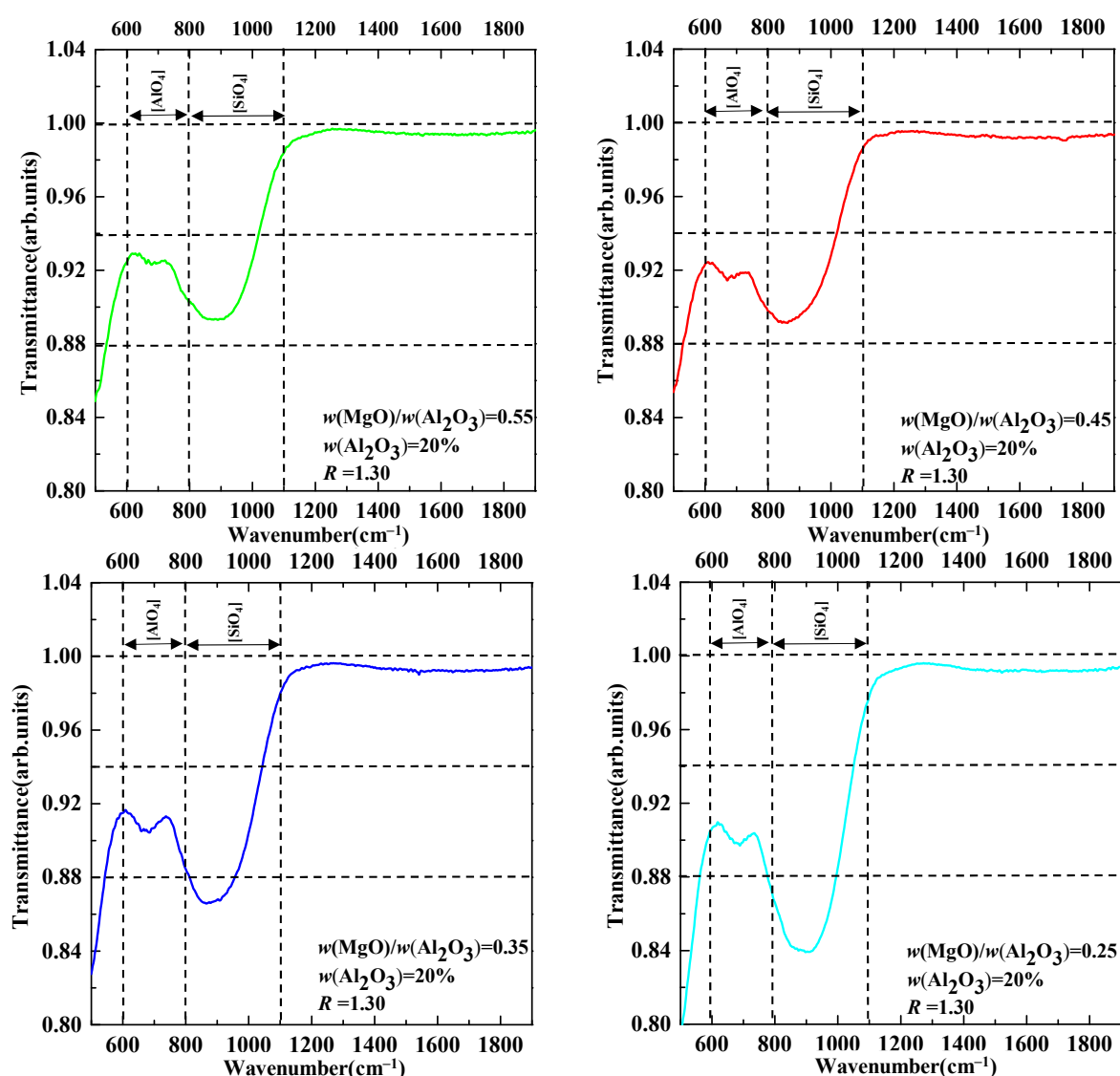


Figure 9. The effect of the magnesia–alumina ratio on the FT-IR results.

4. Conclusions

At a temperature condition of $T = 1823$ K, the desulfurization experiment was carried out. The conclusions are as follows: (1) When the ratio of magnesium to aluminum is 0.50, and the content of Al_2O_3 is 20%, the sulfur capacity in molten slag increases from -4.286 to -4.058 with the increase in basicity, and the distribution ratio of sulfur increases from 19.152 to 32.66—an increase of 70%. (2) When the basicity is 1.30, and the content of Al_2O_3 is 20%, the sulfur capacity in slag increases from -4.286 to -4.102 with the increase in the magnesia–alumina ratio, and the distribution ratio of sulfur increases from 19.152 to 32.66—an increase of 55%. (3) Fourier transform infrared spectroscopy analysis confirms that the proportion of dissociated free oxygen ions within the molten slag phase is augmented by an elevation in either basicity or the magnesia–alumina ratio. These dissociated ions subsequently interact with the bridging oxygen of the silicate structure, facilitating the depolymerization of the complex Si–O network into simpler structural units.

Author Contributions: Conceptualization, Y.G.; methodology, Y.G.; writing—original draft preparation, Y.G.; writing—review and editing, Y.G.; visualization, Z.D.; supervision, M.L. All authors replied to reviewers’ comments and revised the final version. All authors have read and agreed to the published version of the manuscript.

Funding: Natural Science Basic Research Program of Shaanxi Provincial Science and Technology Department (Program No. 2025JC-YBQN-774); Shaanxi Provincial Department of Education Research Project (Program No. 24JK0506); Xi’an Natural Science Foundation Project (Program No. 2025JH-ZRKX-0497). This research was financially supported by the Xi’an Aeronautical Polytechnic Institute (22XHZK-05 and 115020121/76/2023XHQDJ02).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The original contributions presented in this study are included in the article. Further inquiries can be directed to the corresponding author.

Acknowledgments: The authors wish to acknowledge the contributions of associates and colleagues at the Xi’an Aeronautical Polytechnic Institute and Northeastern University of China.

Conflicts of Interest: Authors Yongchun Guo Mengyao Li and Zhimin Ding were employed by the Xi’an Aeronautical Polytechnic Institute and Northeastern University of China. The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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