

EFFECT OF TEMPERATURE ON MgO ACTIVITY IN THE CaO-SiO₂-MgO-Al₂O₃ SYSTEM MEASURED BY MOLTEN TIN

VPLIV TEMPERATURE NA AKTIVNOST MgO V SISTEMU CaO-SiO₂-MgO-Al₂O₃, MERJENE S STALJENIM KOSITROM

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Blast-furnace slag is one of the main by-products produced in the process of blast-furnace ironmaking. Its metallurgical properties have an important impact on the smooth operation and long life of blast furnaces. In this paper, the activity of MgO in a CaO-SiO₂-MgO-Al₂O₃ quaternary slag system was measured using the reference-slag method at 1773–1873 K. The effects of temperature on MgO activity and the activity coefficient of Mg in Sn of the CaO-SiO₂-MgO-Al₂O₃ quaternary slag system were discussed. The results showed that when $w(\text{MgO})/w(\text{Al}_2\text{O}_3) = 0.40$, $B(w(\text{CaO})/w(\text{SiO}_2)) = 1.30$ and $w(\text{Al}_2\text{O}_3) = (12\%; 15\%; 18\%; 20\%)$, the activity of the MgO and the activity coefficient of the Mg in Sn increased with increasing temperature. With increasing temperature, the grooves in the SiO₄ tetrahedral structure at wavenumbers of 800–1200 cm⁻¹ gradually became shallow and flat, indicating that the complex silicate structure synthesized in the form of SiO₂ decreased and the activity of the MgO increased. According to the CaF₂-MgO binary phase diagram, the dissolution enthalpy of MgO was calculated to be -16.409 kJ/mol.

Keywords: blast furnace, slag system, activity of MgO, reference-slag method, enthalpy, solution

Plavžna žilindra je ena od glavnih stranskih produktov v procesu izdelave surovega železa (grodlja). Njene metalurške lastnosti imajo pomemben vpliv na nemoteno obratovanje plavža in njegovo dolgo življensko dobo. V tem članku avtorji opisujejo merjenje aktivnosti MgO v kvaternernem žilindernem sistemu CaO-SiO₂-MgO-Al₂O₃ v temperaturnem območju med 1773 K in 1873 K z referenčno žilinderno metodo. Avtorji v članku razpravljajo o vplivu temperature na aktivnost MgO in o koeficientu aktivnosti Mg v Sn v kvaternernem žilindernem sistemu CaO-SiO₂-MgO-Al₂O₃. Rezultati analiz so pokazali, da aktivnost MgO in koeficient aktivnosti Mg v Sn naraščata z naraščajočo temperaturo pri razmerjih $w(\text{MgO})/w(\text{Al}_2\text{O}_3) = 0,40$, $B(w(\text{CaO})/w(\text{SiO}_2)) = 1,30$ in vsebnostih $w(\text{Al}_2\text{O}_3) = (12\%; 15\%; 18\%; 20\%)$. Z naraščanjem temperature brazde v tetraedrični strukturi SiO₄ pri valovnih številih med 800 cm⁻¹ in 1200 cm⁻¹ postopno postajajo plitvejše in bolj ploske, kar kaže na to da se delež sintetizirane kompleksne silikatne strukture v obliki SiO₂ zmanjšuje in zato se povečuje aktivnost MgO. V skladu z binarnim faznim diagramom CaF₂-MgO so avtorji izračunali, da je entalpija topnosti MgO enaka -16,409 kJ/kmol.

Ključne besede: plavž, žilinderni sistem, aktivnost MgO, referenčna žilindrna metoda, entalpija, raztopina

1 INTRODUCTION

The increased use of high Al₂O₃ iron ore has led to blast-furnace slag with a significant increase in Al₂O₃ content, which has a certain impact on the viscosity, fluidity and desulfurization capacity of blast-furnace slag.¹⁻⁶ According to the molecular-ion-coexistence theory, only free (Ca²⁺ + O²⁻) and (Mg²⁺ + O²⁻) ion pairs in the slag can have a desulfurization ability. The activity of each component involved in the desulfurization reaction is an important factor affecting the desulfurization process. Therefore, the activity of MgO in a blast-furnace slag system is measured experimentally in this paper to provide a theoretical basis for further study of the desulfurization ability of blast-furnace slag.

Many scholars have performed considerable research on the activity of various components in blast-furnace slag systems. The activity of MgO in a MgO-B₂O₃ binary slag system was determined by the slag-metal-equilib-

rium method at 1723 K, 1773 K and 1823 K.^{7,8} Chipman selected an Fe-Si-C alloy as the solvent at 1550 °C, 1600 °C and $p_{\text{CO}} = 101325$ Pa. The activity of SiO₂ in the CaO-SiO₂, CaO-Al₂O₃, SiO₂-MgO binary systems, CaO-SiO₂-Al₂O₃, CaO-SiO₂-MgO, SiO₂-Al₂O₃-MgO ternary systems and CaO-SiO₂-Al₂O₃-(10 %; 20 %; 30 %) MgO quaternary systems was measured.^{9,10} Morita K. et al. selected a silicon-based alloy as the solvent at 1823 K and 1873 K. According to the distribution of Ca, Al and Mg in the slag silicon at equilibrium, the activity of SiO₂ in the CaO-SiO₂, CaO-SiO₂-Al₂O₃ and CaO-SiO₂-MgO slag systems was calculated using the Gibbs-Duhem equation.¹¹ Ohta studied the activity of Al₂O₃ in CaO-SiO₂-Al₂O₃ and CaO-MgO-Al₂O₃ slag systems at 1823 K and 1873 K using the slag-metal-balance method and the Gibbs-Duhem equation.^{12,13} To sum up, there is a lot of research on measuring the activity of silicate melt components, but there are few reports on the experimental measurement of MgO activity in a CaO-SiO₂-Al₂O₃-MgO quaternary slag system for a blast-furnace slag system with a high Al₂O₃ content; additionally, there

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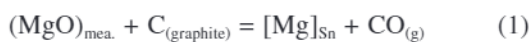
are few studies that explain the activity mechanism from the perspective of the blast-furnace slag structure. Therefore, the experimental determination of the activity of components of blast-furnace slag systems provides a theoretical basis for the further study of the desulfurization ability of blast-furnace slag.

In this paper, using Sn as the metal solvent and CaF₂-MgO as the reference slag, the activity of MgO in a CaO-SiO₂-MgO-Al₂O₃ quaternary slag system is measured using the slag-metal-equilibrium method at 1773–1873 K. The effects of temperature on the activity of MgO in the CaO-SiO₂-MgO-Al₂O₃ quaternary slag system and the number of activity systems of Mg in Sn are discussed. The microstructure of the blast-furnace slag can be studied to better understand the relationship between its chemical composition and metallurgical properties, and provide a theoretical basis for determining a reasonable slag-making system. Finally, this study is of great significance to further understand the desulfurization ability of blast furnace slag and enrich the activity database.

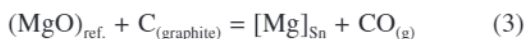
2 EXPERIMENTAL

2.1 Principle of activity determination

The activity of MgO in the CaO-SiO₂-MgO-Al₂O₃ quaternary slag system was determined using the reference-slag method with Sn metal as the solvent and C as the reducing agent. The reaction of reducing MgO in slag with C is as follows:



$$K_1 = \frac{\alpha_{[Mg]} \cdot \alpha_{CO}}{\alpha_{(MgO)} \cdot \alpha_C} \quad (2)$$



$$K_3 = \frac{\alpha_{ref[Mg]} \cdot \alpha_{CO}}{\alpha_{ref(MgO)} \cdot \alpha_C} \quad (4)$$

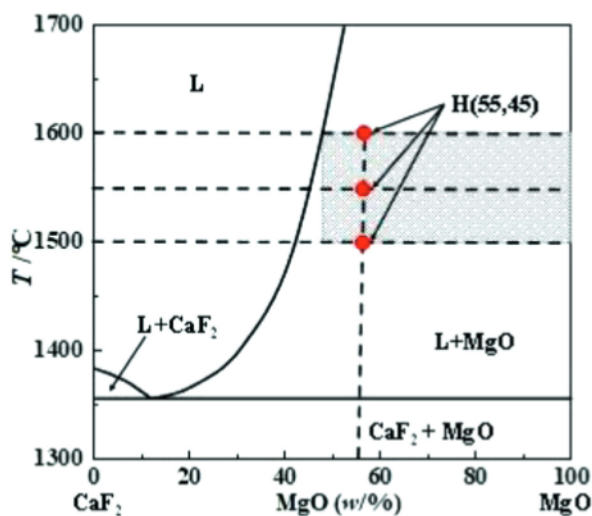


Figure 1: CaF₂-MgO binary phase diagram¹⁵

where $\alpha_{(MgO)}$ and $\alpha_{ref(MgO)}$ are the MgO activities of the measured slag and reference slag relative to the pure solid, respectively. $\alpha_{[Mg]}$ and $\alpha_{ref[Mg]}$ are the activities of Mg in molten Sn at equilibrium with the measured slag and the reference slag relative to an infinitely dilute solution, respectively. Reactions (1) and (3) are carried out under the same conditions, and the standard state of the corresponding component activity is the same ($K_1 = K_3$), leading to:

$$\alpha_{(MgO)} = \alpha_{ref(MgO)} \cdot \frac{\alpha_{[Mg]}}{\alpha_{ref[Mg]}} = \alpha_{ref(MgO)} \cdot \frac{f_{[Mg]} \cdot x_{[Mg]}}{f_{ref[Mg]} \cdot x_{ref[Mg]}} \quad (5)$$

When the reaction is in equilibrium, because the concentration of Mg in Sn is very low, it can be considered to obey Henry's law, i.e., $f_{[Mg]} = f_{ref[Mg]} = 1$.¹⁴

Figure 1 shows the CaF₂-MgO binary phase diagram. In the CaF₂-MgO binary phase diagram, H($w(MgO) = 55\%$ and $w(CaF_2) = 45\%$) is selected as the reference slag system, i.e., $\alpha_{ref(MgO)} = 1$. Then, Equation (5) can be further simplified to Equation (6).

$$\alpha_{(MgO)} = \frac{x_{[Mg]}}{x_{ref[Mg]}} \quad (6)$$

where $x_{[Mg]}$ and $x_{ref[Mg]}$ denote the concentration of Mg in Sn at equilibrium with the measured slag and reference slags, respectively. $f_{[Mg]}$ and $f_{ref[Mg]}$ are the activity coefficient of Mg in molten Sn at equilibrium with the measured slag and the reference slag, respectively.

2.2 Experimental procedure

The experiment was carried out in a high-temperature tubular furnace, as shown in Figure 2. The temperature of the tubular furnace is controlled by a PID controller, and a Pt-6Rh/Pt-30Rh double platinum-rhodium thermocouple is selected. To avoid damage to the furnace tube caused by hot charging and the hot taking of the samples, a special graphite sleeve is used for protection. The raw materials used for the experiment were high-purity CaO, SiO₂, MgO and Al₂O₃, Sn metal particles, CO gas and oil. The composition of the experimental slag sample is shown in Table 1. A six-hole graphite crucible was used for the experiment, and 5 g of Sn metal particles and 10 g of slag samples were put into each hole. Early in the experiment, to ensure balance of the reaction in the experimental process and the accuracy of the experimental results, a balance was reached and held for 24 h when the temperature of the tubular furnace was increased to 700–800 °C and CO was injected at a flow rate of 0.98–1.0 L/min. The graphite crucible loaded with the sample was slowly put into the constant-temperature zone in the furnace. When the temperature of the tubular furnace sample was increased to the test temperature, after the balance time of 24 h, the crucible was removed with a graphite rod and cooled with oil. After the cooled sample was removed, the slag and Sn metal were carefully separated, and the Sn metal phase was sampled and

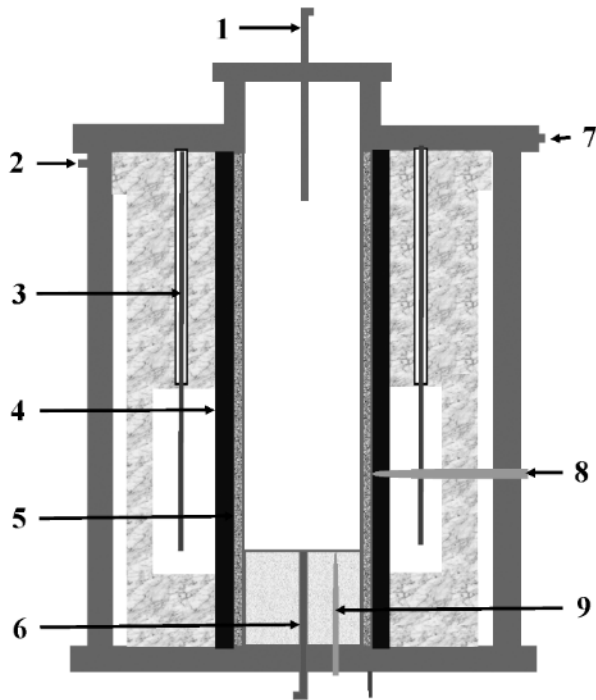


Figure 2: Schematic of experimental apparatus: 1 – gas outlet(CO), 2 – water intake, 3 – heating unit of MoSi₂, 4 – graphite crucible, 5 – Al₂O₃ tube, 6 – gas inlet(CO), 7 – water outlet, 8 – Pt-6Rh/Pt-30Rh, 9 – Pt-6Rh/Pt-30Rh

analyzed. The content of Mg in the Sn metal phase was analyzed with inductively coupled plasma-atomic emission spectrometry (ICP-AES).

Table 1: Chemical compositions of measured slag

No.	slag compositions (w/%)			
	CaO	SiO ₂	MgO	Al ₂ O ₃
1#	47.02	36.17	4.8	12
2#	44.65	34.34	6.0	15
3#	42.27	32.52	7.2	18
4#	40.69	31.30	8.0	20

3 RESULTS AND DISCUSSION

3.1 Activity of MgO in the CaO-SiO₂-MgO-Al₂O₃ slag system

Table 2 shows the mole fraction of Mg in the Sn metal solution that reacted with the reference slag. **Table 3** shows the mole fraction of Mg in the Sn metal solvent and the activity of the MgO in the slag to be measured.

Table 2: Mole fraction of Mg in the Sn metal solvent (reference slag)

reference slag 45 % CaF ₂ - 55 % MgO	x _{ref[Mg]}			average value
	1	2	3	
1773 K	0.001252	0.001194	0.001369	0.001272
1823 K	0.003251	0.002119	0.002050	0.002473
1873 K	0.003018	0.003027	0.003032	0.003026

3.2 Effect of temperature on the activity of MgO

Figure 3 shows the effect of temperature on MgO activity in the CaO-SiO₂-MgO-Al₂O₃ quaternary slag system when w(MgO)/w(Al₂O₃) = 0.40, B(w(CaO)/w(SiO₂)) = 1.30 and w(Al₂O₃) = (12 %; 15 %; 18 %; 20 %). **Figure 3** shows that when w(MgO)/w(Al₂O₃) = 0.40, B(w(CaO)/w(SiO₂)) = 1.30 and w(Al₂O₃) = (12 %; 15 %; 18 %; 20 %) in the slag, the MgO activity increases with increasing temperature. Regarding the MgO components with the pure substance as the standard state, the chemical potential expression is presented in Equation (7):

$$\mu_{(MgO)} = \mu^*_{(MgO)} + RT \ln \alpha_{(MgO)} \quad (7)$$

where $\mu_{(MgO)}$ is the chemical potential of MgO and $\mu^*_{(MgO)}$ is the standard chemical potential of MgO. R is the molar gas constant, and its value is 8.314 J/(mol·K).

For Equation (7), the partial derivative of the temperature under the conditions of constant pressure and constant composition are calculated to obtain the influence of temperature on the obtained results (Equation (8)):

$$\begin{aligned} \frac{\partial \ln \alpha_{(MgO)}}{\partial T} &= \frac{\partial \frac{\mu_{(MgO)} - \mu^*_{(MgO)}}{RT}}{\partial T} = \frac{1}{R} \frac{\partial \frac{G_{(MgO)} - G^*_{(MgO)}}{T}}{\partial T} = \\ &= \frac{1}{R} \frac{\partial \left[\frac{H_{(MgO)} - H^*_{(MgO)}}{T} - (S_{(MgO)} - S^*_{(MgO)}) \right]}{\partial T} = \\ &= -\frac{H_{(MgO)} - H^*_{(MgO)}}{RT^2} = -\frac{\Delta H_{(MgO)}}{RT^2} \end{aligned} \quad (8)$$

where $G_{(MgO)}$ is the standard free energy of MgO, $H_{(MgO)}$ is the partial molar enthalpy of MgO, $H^*_{(MgO)}$ is the standard molar enthalpy of MgO, $S_{(MgO)}$ is the partial molar entropy of MgO, $S^*_{(MgO)}$ is the standard molar entropy of MgO, $\Delta H_{(MgO)}$ is the partial molar enthalpy of MgO ($H_{(MgO)}$) and $H^*_{(MgO)}$ is the standard molar enthalpy.

When the MgO composition is certain, $\Delta H_{(MgO)}$ can be approximately taken as a constant (independent of

Table 3: Mole fraction of Mg in the Sn metal solvent (measured slag) and activity of MgO

No.	slag compositions (w/%)				x _[Mg]			α _[Mg]		
	CaO	SiO ₂	MgO	Al ₂ O ₃	1773 K	1823 K	1873 K	1773 K	1823 K	1873 K
1#	47.02	36.17	4.8	12	0.000219	0.000595	0.000882	0.1726	0.2405	0.2917
2#	44.65	34.34	6.0	15	0.000278	0.000785	0.001023	0.2186	0.3174	0.3383
3#	42.27	32.52	7.2	18	0.000390	0.000921	0.001301	0.3067	0.3725	0.4301
4#	40.69	31.30	8.0	20	0.000502	0.001067	0.001491	0.3949	0.4316	0.4928

Table 4: MgO parameters at different temperatures

T/°C	1400	1500	1600	1700	1800	1900	2000
T/K	1673	1773	1873	1973	2073	2173	2273
T/K	1450	1350	1250	1150	1050	950	850
1/T (K ⁻¹)	0.00059	0.00056	0.00053	0.00050	0.00048	0.00046	0.00044
x _(MgO)	0.50526	0.55737	0.58725	0.61615	0.64411	0.67120	0.69744
ln x _(MgO)	-0.68266	-0.58451	-0.53230	-0.48426	-0.43987	-0.39868	-0.36034

temperature). By integrating Equation (8) the relationship between activity and temperature is obtained (Equation (9)):

$$\ln \alpha_{(MgO)} = \frac{\Delta H_{(MgO)}}{RT} + C \quad (9)$$

Where *C* is the integral constant.

The slope (*a*) of the straight line can be obtained by plotting ((lnx_(MgO)) - (1/T)) on the liquidus:¹⁶

$$\Delta H_{(MgO)} = aR - \Delta_{fus}H_{(MgO)} \quad (10)$$

where Δ_{fus}H_(MgO) is the enthalpy of fusion of MgO (Δ_{fus}H_(MgO) = 77 kJ/mol).

Regarding the simple CaF₂-MgO phase diagram, according to the data in **Table 4**, the following Equation (11) is obtained by the binary-regression method with an R² fitting degree of a 0.99.¹⁶

$$\ln x_{(MgO)} = -\frac{1964}{T} + 0.5085 \quad (11)$$

In Equation (11), *a* = -1964 can be obtained. A further calculation shows that the enthalpy of the MgO solution is -16.409 kJ/mol. Therefore, according to Equation (9), ln α_(MgO) is a positive proportional function of temperature.

Figure 4 shows the Fourier-transform infrared spectroscopy (FTIR) of the slag oil cooling to different temperatures when w(MgO)/w(Al₂O₃) = 0.40, B(w(CaO)/w(SiO₂)) = 1.30 and w(Al₂O₃) = 20 %. From the FTIR results in **Figure 4**, it can be found that with an increase

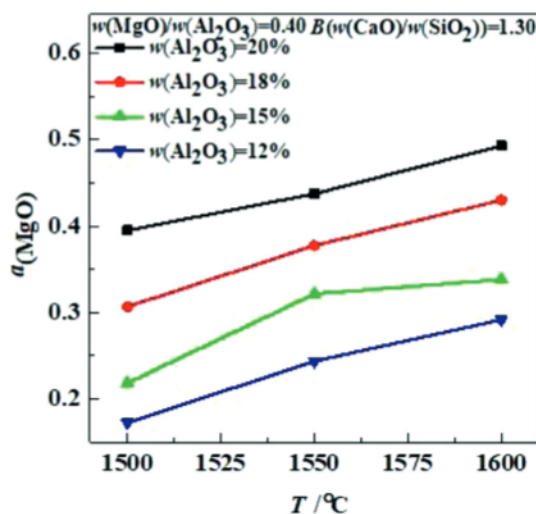
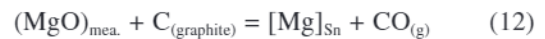


Figure 3: Effect of temperature on MgO activity in CaO-SiO₂-MgO-Al₂O₃ system

in the temperature from 1773 K to 1873 K, the Si-O-Al structure at wavenumbers 400–600 cm⁻¹ and the AlO₄ structure at wavenumbers 600–800 cm⁻¹ do not change significantly. The groove of the SiO₄ tetrahedral structure at wavenumbers of 800–1200 cm⁻¹ gradually becomes shallow and flat, which indicates that the complex silicate structure polymerized in the form of SiO₂ is reduced. With increasing temperature, the dissociated free-oxygen ions (O²⁻) of MgO increase. The dissociated free-oxygen ions (O²⁻) interact with the bridging oxygen (O⁰) of silicate, which gradually depolymerizes the complex Si-O structure in slag, thus simplifying the slag network structure and increasing the activity of the MgO.

3.3 Activity coefficient of Mg in Sn

According to the present results, the activity coefficient of Mg in Sn can be obtained.



$$K^\theta = \frac{\alpha_{[Mg]} \cdot P_{CO}}{\alpha_{(MgO)} \cdot \alpha_C \cdot P^\theta} \quad (13)$$

$$\Delta G^\theta = 615200 - 289.82T \text{ (J/mol)} \quad (14)$$

$$\lg K^\theta = -\frac{615200}{19147 \cdot T} + \frac{289.82}{19147} \quad (15)$$

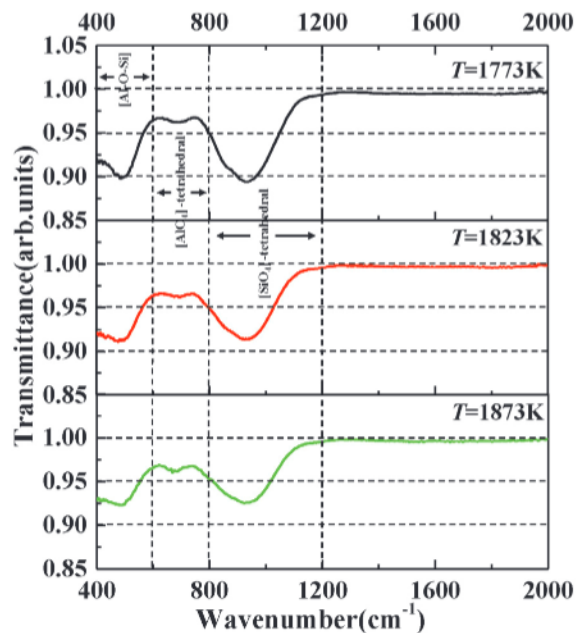


Figure 4: Fourier-transform infrared spectroscopy of oil cooling at different temperatures

Table 5: Activity of Mg in the Mg-Sn system $\alpha_{[Mg](R)}$

No.	1773 K			1823 K			1873 K		
	$\alpha_{[MgO]}$	$x_{[Mg]}$	$\alpha_{[Mg](R)}$	$\alpha_{[MgO]}$	$x_{[Mg]}$	$\alpha_{[Mg](R)}$	$\alpha_{[MgO]}$	$x_{[Mg]}$	$\alpha_{[Mg](R)}$
1#	0.1726	0.000219	0.000178	0.2405	0.000595	0.000781	0.2917	0.000882	0.002799
2#	0.2186	0.000278	0.000226	0.3174	0.000785	0.001031	0.3383	0.001023	0.003247
3#	0.3067	0.000390	0.000317	0.3725	0.000921	0.00121	0.4301	0.001301	0.004127
4#	0.3949	0.000502	0.000408	0.4316	0.001067	0.001401	0.4928	0.001491	0.004729

where $\alpha_{(MgO)}$ is the MgO activity of the measured slag relative to the pure solid. p_{CO} and p_{CO}^θ represent the CO pressures and standard atmospheric pressure ($p_{CO}^\theta = p_{CO} = 101325$ Pa), respectively. $\alpha_{[Mg]}$ is the Mg activity of the measured slag relative to the pure solid, and α_C , which is the activity of carbon, is 1 when the pure substance standard is used.

$$K^\theta = \frac{\alpha_{[Mg]}}{\alpha_{(MgO)}} \tag{16}$$

$$\alpha_{[Mg]} = K^\theta \cdot \alpha_{(MgO)} \tag{17}$$

According to Equation (17), the activity of the Mg-Sn system with pure material as the standard state is calculated, and the results are shown in **Table 5**.

Notably, $\gamma_{[Mg]}^\theta$ is the activity coefficient of Mg in a dilute solution with a pure solid as the standard state $\alpha_{[Mg](R)} = \gamma_{[Mg]}^\theta \cdot x_{[Mg]}$ and its value is a constant. Regarding the $(\alpha_{[Mg](R)} - x_{[Mg]})$ fitting curve of the solution, the tangent of the curve at $x_{[Mg]} = 0$ was plotted and extended to the intersection on the $x_{[Mg]} = 1$ axis, i.e., $\gamma_{[Mg]}^\theta$. The experimental results are shown in **Table 6**. **Figure 5** shows the effect of temperature on the activity coefficient of Mg in Sn, in which the activity coefficient of Mg in Sn gradually increases with increasing temperature.

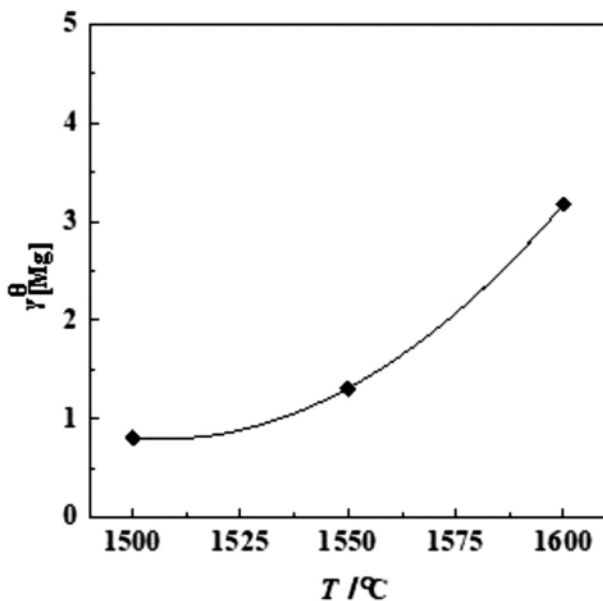


Figure 5: Effect of temperature on activity coefficient of Mg in Sn

Table 6: Activity coefficient of Mg in Sn

T/K	$(\alpha_{[Mg](R)} - x_{[Mg]})$ fit curve	$\gamma_{[Mg]}^\theta$
1773	$\alpha_{[Mg](R)} = 13.115 \cdot x_{[Mg]}^2 + 0.803 \cdot x_{[Mg]} + 2 \cdot 10^{-6}$	0.803
1823	$\alpha_{[Mg](R)} = 1.973 \cdot x_{[Mg]}^2 + 1.311 \cdot x_{[Mg]}$	1.311
1873	$\alpha_{[Mg](R)} = -2.923 \cdot x_{[Mg]}^2 + 0.803 \cdot x_{[Mg]}$	3.176

4 CONCLUSIONS

In this paper, the activity of MgO in a CaO-SiO₂-MgO-Al₂O₃ quaternary slag system was determined using the reference-slag method with Sn as the metal solvent and CaF₂-MgO as the reference slag at 1773–1873 K. Based on the experimental data, the effects of temperature on the MgO activity and activity coefficient of Mg in Sn of the CaO-SiO₂-MgO-Al₂O₃ quaternary slag system were discussed. The results are:

1. When $w(\text{MgO})/w(\text{Al}_2\text{O}_3) = 0.40$, $B(w(\text{CaO})/w(\text{SiO}_2)) = 1.30$ and $w(\text{Al}_2\text{O}_3) = (12\%; 15\%; 18\%; 20\%)$, the activity of MgO increases with increasing temperature.
2. With increasing temperature, the grooves in the [SiO₄] tetrahedral structure at wavenumbers of 800–1200 cm⁻¹ gradually become shallow and flat, indicating that the complex silicate structure synthesized in the form of SiO₂ decreases and the activity of MgO increases.
3. The activity coefficient of Mg in Sn increases with increasing temperature.
4. According to the CaF₂-MgO binary phase diagram, the solution enthalpy of MgO is calculated to be -16.409 kJ/mol.

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