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RELATIONSHIP BETWEEN THE CALCULATED OXYGEN ACTIVITY AND THE SULFUR PARTITION RATIO FOR $CaO-Al_2O_3-SiO_2-MgO$ SLAG DURING LADLE REFINING

RAZMERJE MED IZRAČUNANO AKTIVNOSTJO KISIKA IN DELEŽEM PORAZDELITVE ŽVEPLA V ŽLINDRI $CaO-Al_2O_3-SiO_2-MgO$ MED RAFINACIJO V LIVNEM LONCU

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A slag-metal equilibrium study was carried out to investigate the effect of oxygen activity on the sulfur partition for $CaO-Al_2O_3-SiO_2-MgO$ slag. The sulfide capacity C_s prediction models by Sosinsky-Sommerville based on the optical basicity Λ and the KTH model in terms of the defined interaction coefficient of the component i to j $\xi^{i-j}_{\mathrm{Interaction}}$ is used in this work for both a comparison and an estimation of the sulfur partition ratio between ladle-treated slag and liquid steel. From the obtained results, it was shown that the Sosinsky-Sommerville optical basicity approach gives higher values for the sulfur partition ratio (L_s) compared with the KTH model.

Keywords: sulfide capacity, sulfur partition ratio, oxygen activity, optical basicity, KTH model

Opravljena je bila raziskava učinka aktivnosti kisika na razporeditev žvepla v žlindri $CaO-Al_2O_3-SiO_2-MgO$. V tem delu sta bila uporabljena model Sosinsky-Sommerville za napovedovanje kapacitete sulfida C_s, ki temelji na optični bazičnosti Λ, ter model KTH definiranega interakcijskega koeficienta komponent od i do j $\xi^{i-j}_{\mathrm{Interaction}}$ tako za primerjavo kot tudi za določanje deleža porazdelitve žvepla med žlindro v loncu in jeklom. Iz dobljenih rezultatov izhaja, da Sosinsky-Sommervillov približek optične bazičnosti daje v primerjavi z modelom KTH višje vrednosti za delež razporeditve žvepla (L_s).

Ključne besede: zmogljivost sulfida, delež porazdelitve žvepla, aktivnost kisika, optična bazičnost, model KTH

1 INTRODUCTION

Over the past few decades the sulfur level in steel has been improved enormously. Therefore, the more intensive development industries, such as automotive and pipelines for the transportation of gas and oil, require good control of the sulfur level in steel products.[1] Due to these facts, close control of the sulfur level is essential for the production of good-quality steel. One of the main subjects when investigating the slag/metal interface is the behavior of the oxygen in the liquid steel.[2] The state of oxidation of a bath is of vital importance in controlling the reactions between the slag and the metal in steelmaking. It influences both the metal losses in the slag and the quality of the produced steel.[3] The present paper is focused on predicting the oxygen activities calculated using the sulfur equilibrium between the top slag and the steel.

1.1 Sulfide capacity models

The concept of sulfide capacity was proposed by Finchman and Richardson,[4] and it was defined as:

$$C_s = (\%S)(P_{O2}/P_{S2})^{1/2} \tag{1}$$

The sulfide capacity is a property of the slag that is dependent only on the temperature and the slag's composition. The sulfide capacity can be used to describe the potential ability of an arbitrary homogeneous molten slag to remove sulfur and to compare the desulfurization characteristics of different slags.

The slag desulfurization capacity in the system slag-metal may be expressed as the slag sulfide capacity:[5]

$$[S] + (O^{2-}) = (S^{2-}) + [O] \tag{2}$$

Some authors have preferred to define it with reference to the slag metal reaction, in which case the definition becomes:[6]

$$C'_s = (\%S)[a_o]/[a_s] \tag{3}$$

Using Turkdogan's formulation[7] the relation between C_s and C_s' can be written as $C'_s = (\%S)[a_o]/[a_s]$, where C_s' can be converted to C_s using the relationship:

$$C_s = C'_s/K_{os} \tag{4}$$

The equilibrium constant K_{os} for the above equation is:

$$\lg K_{os} = -935/T + 1.375 \tag{5}$$

1.2 Calculation of the activity of oxygen in the slag and the steel

It is well known that control of the desulfurization process is impossible if the oxygen activity is not known. When the steel is deoxidised with aluminum and silicon, the reactions deciding the oxygen content are the $Al/O/Al_2O_3$ and the $Si/O/SiO_2$ equilibrium. **Table 1** summarizes the chemical reactions used in this work that take part in the deoxidation of the steel melt, in the slag-melt equilibrium during ladle treatment, their mole free-energy changes in the standard state, as well as their reaction constants.

Also, the Ohta and Suito[8] expressions were used to calculate the Al_2O_3 and the SiO_2 activities in the slag, while Wagner's expressions[9] in equations (6,7) were used to calculate the alumina $[a]_{Al}$ and silicon $[a]_{Si}$ activities in the steel. All the used oxides are in weight percent.

The activity coefficients of the elements in the metal are calculated by using Wagner's equation,[9] as follows:

$$\lg f_i = \Sigma\,(e_i^j\,[\%\,j]) \tag{6}$$

where:

e_i^j – interaction coefficient of *j* on *i*

f_i – Henry's activity coefficient for the species *i* in the metal

From equation 6, the *a* activity of element *i* in the steel can be calculated as:

$$a_i = f_i\,[\%i\,]\ (i = \mathrm{Si, Al, S}) \tag{7}$$

The interaction coefficients used in this work are as follows:

$e_S^S = (-0.153 + 233/T)$, $e_S^C = 0.113$, $e_S^{Si} = 0.063$, $e_S^{Al} = 0.035$, $e_S^{Mn} = -0.026$, $e_{Al}^{Al} = (0.011+63/T)$, $e_{Al}^C = 0.091$, $e_{Al}^{Si} = 0.056$, $e_{Al}^S = 0.030$, $e_{Si}^C = 0.18$, $e_{Si}^{Si} = (0.089+34.5/T)$, $e_{Si}^S = 0.056$, $e_{Si}^{Al} = 0.058$, and $e_{Si}^{Mn} = 0.002$. [10–12]

From the equations of the equilibrium constants given in **Table 1**, it was possible to derive an expression for the oxygen activity:

$$a_O = \sqrt[3]{\frac{a_{Al_2O_3}}{a_{Al}^2 \cdot e^{-\frac{\Delta G^0}{RT}}}} \tag{8}$$

$$a_O = \sqrt{\frac{a_{SiO_2}}{a_{Si} \cdot e^{-\frac{\Delta G^0}{RT}}}} \tag{9}$$

The sulfur partition ratio between the slag and the metal may be expressed by combining equations (4) to (9) as follows:[13]

$$\lg L_s = -\,935/T + 1.375 + \lg C_s + \lg f_S - \lg\,[a_o] \tag{10}$$

where:

C_S – sulphide capacity

L_S – sulfur partition between the slag and the metal, $L_s = (\%S)/[\%S]$

$[a_o][a_s]$ – oxygen and sulfur activity in the molten metal,

[%S] – sulfur weight percent in the steel

(%S) – sulfur weight percent in the slag

1.3 Optical basicity concept

The optical basicity of the molten slag can be calculated using the following relationships: [14]

$$\Lambda = \sum_{i=1}^{n} \Lambda_i N_i \tag{11}$$

$$N_i = \frac{X_i n_{0i}}{\sum_{i=1}^{n} X_i n_{0i}} \tag{12}$$

where:

Λ – optical basicity of the slag

Λ_i – optical basicity value of the component "*i*"

N_i – compositional fraction

X_i – mole fraction of component "*i*" in the slag

n_{Oi} – number of oxygen atoms in the component "*i*"

Sosinsky and Sommerville (S–S)[15] derived the following empirical correlation between the optical basicity, the temperature and the sulfide capacity of the slag at temperatures between 1400 °C and 1700 °C:

$$\lg C_s = \frac{22690 - 54640\Lambda}{T} + 43.6\Lambda - 25.2 \tag{13}$$

The values of the optical basicity for the common steelmaking oxides used in this work have been taken from the literature.[16]

Table 1: Thermodynamic data on chemical reactions taking place in deoxidation, and slag-metal equilibrium
Tabela 1: Termodinamski podatki o kemijskih reakcijah, ki potekajo med dezoksidacijo pri ravnotežju žlindra-kovina

Chemical reactions	Mole free energy changes, ΔG° (J/mol)	Constants, $K = \exp(-\Delta G°/RT)$
$2[Al]+3[O]= (Al_2O_3)$	$\Delta G^0_{Al} = -1205115 + 386.7T$ [13]	$\frac{a_{Al_2O_3}}{[a_{Al}]^2[a_O]^3}$ (Eq.8)
$[Si]+2[O] = (SiO_2)$	$\Delta G^0_{Si} = -581900 + 221.8T$ [10]	$\frac{a_{SiO_2}}{[a_{Si}]\,[a_O]^2}$ (Eq.9)
$\lg a_{Al2O3} = \{-0.275(\%CaO) + 0.167\,(\%MgO)\}/(\%SiO_2)+0.033(Al_2O_3) - 1.560$ [8]		
$\lg a_{SiO2} = 0.036(\%MgO)+0.061(Al_2O_3)+0.123(\%SiO_2) - (\%SiO_2)/(\%CaO) - 6.456$ [8]		

1.4 The KTH model

The KTH model was developed by the Department of Metallurgy in the Royal Institute of Technology (Sweden).[17] According to the definition of the sulfide capacity, C_s can be expressed as:

$$C_{s} = \exp\left(\frac{-\Delta G^{0}}{RT}\right)\left(\frac{a_{O^{2-}}}{f_{S^{2-}}}\right) = \exp\left(\frac{-\Delta G^{0}}{RT}\right)\exp\left(\frac{\xi}{RT}\right) \qquad (14)$$

where $a_o{}^{2-}$ is the activity of the oxygen ions in the slag, $f_s{}^{2-}$ is the activity coefficient of the sulfide ions in the slag, R is the gas constant, T is the temperature in K, and ΔG is the standard Gibbs energy change. In the model, the ratio of the activity of O^{2-} to the activity coefficient of S^{2-}, $a_O{}^{2-}/f_S{}^{2-}$ is expressed as

$$\frac{a_{O^{2-}}}{f_{S^{2-}}} = \exp\left(-\frac{\xi}{RT}\right) \qquad (15)$$

In the case of unary systems, ξ is a function of the temperature only. However, in a multicomponent system, ξ is described as a function of both the temperature and the composition:

$$\xi = \Sigma(X_i\,\xi_i + \xi_{mix}) \qquad (16)$$

where the subscript i denotes the component i, X_i is the mole fraction of this component, ξ_i is expressed as a linear function of the temperature for each component in the slag in the absence of an interaction between the different species, ξ_{mix} is the mutual interaction between the different species.

According to the model,[17] the sulfide capacities of the six-component slags can be expressed as follows:

$$\begin{aligned}&RT(\ln C_s) = 58.8157T - 118535 - \{X_{Al2O3}\cdot 157705.28 - X_{CaO}\cdot 33099.43 + X_{MgO}\cdot 9573.07 - X_{MnO}\cdot 36626.46 + X_{SiO2}\cdot 168872.59\} - \{\xi^{Al2O3-CaO}_{Interaction} + \xi^{Al2O3-SiO2}_{Interaction} + \xi^{Al2O3-MnO}_{Interaction}\\ &\xi^{CaO-SiO2}_{Interaction} + \xi^{MgO-SiO2}_{Interaction} + \xi^{MnO-SiO2}_{Interaction} + \xi^{CaO-FeO}_{Interaction} + \xi^{MnO-FeO}_{Interaction} +\\ &\xi^{FeO-SiO2}_{Interaction} + \xi^{Al2O3-CaO-MgO}_{Interaction} + \xi^{Al2O3-CaO-SiO2}_{Interaction} + \xi^{Al2O3-MgO-SiO2}_{Interaction} +\\ &\xi^{Al2O3-MgO-MnO}_{Interaction} + \xi^{Al2O3-MnO-SiO2}_{Interaction} + \xi^{CaO-MgO-SiO2}_{Interaction} +\\ &\xi^{CaO-MnO-SiO2}_{Interaction} + \xi^{CaO-MnO-SiO2}_{Interaction} + \xi^{MgO-MnO-SiO2}_{Interaction} + \xi^{Al2O3-FeO-SiO2}_{Interaction}\\ &+ \xi^{MgO-FeO-SiO2}_{Interaction} + \xi^{MnO-FeO-SiO2}_{Interaction}\}\end{aligned} \qquad (17)$$

Although the KTH model is valid for the atmosphere-slag interaction, in this paper it will be used to compare the values of C_s.

2 EXPERIMENTAL

The plant data of 12 heats of low-carbon, Al-Si killed steel from the Dillinger Hütte Steel-plant in Dillingen, Germany, taken after the vacuum-degassing operation in the ladle-refining process, were used in this study. The liquid steel samples were taken using an automatic sampling system, while the slag samples were manually collected with a spoon and subjected to a chemical analysis. Because the oxygen analyses of the steel samples were not available, a logical way to estimate the oxygen levels in the steel bath was to estimate the activities of the Al_2O_3 and SiO_2 in the slag by thermodynamic calculations from the contents obtained by sampling and chemical analysis, and use them to estimate the oxygen potential in the steel bath, assuming a slag/metal equilibrium.[18]

3 RESULTS AND DISCUSSION

The average chemical compositions of the analyzed metal and slag phases are summarized in **Table 2**.

The optical basicity for all the analyzed slags was in the narrow range Λ = 0.77–0.79.

3.1 Comparison of the sulfide capacity results

In this work, we applied equations (3) to (7) in order to calculate the measured values of the sulfide capacity with the calculated oxygen activity $[a_o]$ in the steel according to Eq. (8) (subsequently called Case A) and Eq. (9) (subsequently called Case B). Then the results were compared with the calculated values of the sulfide capacity by Sosinsky-Sommerville (S-S) and the KTH model. **Table 3** shows the calculated values of optical basicity, the measured and calculated values of the sulfide capacity, the oxygen activities $[a_o]$ in the steel and alumina $a_{(Al2O3)}$ and the silica $a_{(SiO2)}$ activities in the slag for the analysed heats. As can be seen in **Table 3**, the calculated values for the alumina activities $a_{(Al2O3)}$ are generally very low, i.e., below mass fractions $w = 10^{-3}$ % and 10^{-4} % and sometimes even lower than $w = 10^{-5}$ %. Comparatively, in the case of the calculated values of the silica activities $a_{(SiO2)}$ after the VD treatment, the calculated values of the silicon activity in the slag are stable at the level of $w = 10^{-4}$ % which corresponds to a SiO_2 content of a few per cent. This is in accordance with the published results. [19]

Table 2: Chemical composition of analysed metal and slag
Tabela 2: Kemijska sestava analizirane kovine in žlindre

w/%	C	Si	Mn	S	Al	*T* [K]
Average	0.10	0.35	1.51	0.0004	0.027	1855
Range	0.03–0.18	0.22–0.46	1.37–1.61	0.0002–0.0005	0.011–0.037	1838–1869
w/%	CaO	SiO_2	MgO	S	Al_2O_3	
Average	54.64	4.38	7.46	0.70	30.64	
Range	52.49-57.00	2.38-8.20	4.10-12.16	0.46-1.03	27.51-34.38	

Table 3: The calculated optical basicity, oxygen activities $[a_o]$ in steel and alumina $a_{(Al2O3)}$ and silica $a_{(SiO2)}$ activities in slag for analysed heats

Tabela 3: Izračunana optična bazičnost, aktivnosti kisika $[a_o]$ v jeklu ter aktivnosti aluminijevega oksida $a_{(Al2O3)}$ in silike $a_{(SiO2)}$ v žlindri analiziranih talin

Heats	Λ	$a(Al_2O_3)$	$a(SiO_2)$	$[a_o]_{Al}$,[%]	$[a_o]_{Si}$,[%]
1	0.78	0.000442	0.000198	2.16E-05	7.74E-05
2	0.78	0.003583	0.000247	7.34E-05	9.84E-05
3	0.77	0.000044	0.000157	9.27E-06	9.08E-05
4	0.78	0.000007	0.000175	5.68E-06	1.04E-04
5	0.78	0.000012	0.000194	5.01E-06	7.94E-05
6	0.77	0.000150	0.000220	2.03E-05	1.22E-04
7	0.78	0.000222	0.000174	1.24E-05	6.37E-05
8	0.79	0.000616	0.000151	1.92E-05	7.58E-05
9	0.78	0.000446	0.000180	3.29E-05	1.05E-04
10	0.79	0.000060	0.000148	8.77E-06	5.86E-05
11	0.78	0.000249	0.000206	1.54E-05	7.72E-05
12	0.79	0.000024	0.000127	1.25E-05	9.27E-05

Also, it can be seen from the results in **Table 3** that the changes in the obtained values of the sulfide capacities at the end of ladle treatment were relatively small for the S-S model and the measured values derived by a calculation of the sulfide capacities from a prediction of the oxygen activity $[a_o]_{Si}$ (Case B) and the KTH model results compared with those derived by the calculation of the sulfide capacities by a prediction of oxygen activity $[a_o]_{Al}$ (Case A). Moreover, the obtained values of the sulfide capacities of the analyzed slags are in good agreement with earlier published results.[20]

3.2 Comparison of the sulfur distribution ratio

The slag-metal sulfur distribution ratio L_s after desulphurization is another important parameter in the modeling of sulfur removal in steel making. In this work the predicted values of lg L_s were calculated using equation (8), suggested by M. T. Andersson et al. [13] For the purpose of a comparison, the sulfide capacity was also calculated using the Sosinsky-Sommerville (S-S) approach based on the optical basicity concept according to he original parameters, as well as the KTH model. Then, the results were compared with the calculated values of the sulfur distribution ratio with the calculated oxygen activity $[a_o]_{Al}$ (Case A) and the oxygen activity $[a_o]_{Si}$ (Case B).

The comparison of the calculation determined sulfur distribution ratio L_S^{calc} using both cases with the measured L_S^{msr} is shown in **Table 4** and **Figures 1** and **2**.

As can be seen from **Table 4** and **Figures 1** and **2**, both models generally exhibit higher discrepancy for the values of L_s in Case B compared with the results obtained in Case A. Also, it is evident that the correlation between the measured L_S^{msr} and the calculated values of the sulfur distribution ratios L_S^{calc} are more or less scattered. In contrast, it is clear that the predictions with the KTH model agree well with the measured results in Case A. The optical basicity concept, which is represented here by the Sosinsky-Sommerville (S–S) model, gives much higher predicted values. This is in good agreement with earlier published results.[21]

The exceptions are the results obtained when the oxygen activity data is used in the metal phase $[a_o]_{Si}$ (Case B). It is obvious that the data points show larger scattering, but both models give better L_s prediction compared with Case A.

Based on thermodynamic calculations, it can be summarised that the equilibrium state was possibly not

Table 4: The values of sulfur partition ratio for different calculated oxygen activities $[a_o]$ and lg C_s calculated by Sosinsky-Sommerville (S-S) and KTH models

Tabela 4: Vrednosti deleža porazdelitve žvepla za različne izračunane aktivnosti kisika $[a_o]$ in lg C_s, izračunanim s Sosinsky-Sommerville (S-S) modelom in modeli KTH

Heats	L_S^{msr}	L_S^{calc}			
		Case A (S-S)	Case B (S-S)	Case A (KTH)	Case B (KTH)
1	3290	3940	1100	1945	543
2	2967	964	719	801	597
3	2233	8373	854	7454	760
4	1867	19479	1067	7491	410
5	1723	16515	1043	7049	445
6	1527	3463	579	1555	260
7	2060	7104	1381	3723	724
8	1984	5343	1353	3766	954
9	1520	2865	897	1813	567
10	1430	9991	1493	5356	801
11	1228	5263	1048	2066	411
12	1094	11944	1614	5167	698

Table 5: The differences between measured L_S^{msr} and calculated values of sulfur partition ratio L_S^{calc} for different calculated oxygen activities $[a_o]$ and lg C_s calculated by Sosinsky-Sommerville (S-S) and KTH models

Tabela 5: Razlike med izmerjenimi L_S^{msr} in izračunanim deležem porazdelitve žvepla L_S^{calc} za različne izračunane aktivnosti kisika $[a_o]$ in lg C_s, izračunanim s Sosinsky-Sommerville (S-S) in modeli KTH

Heats	ΔL_s Case A (S–S)	ΔL_s Case B (S–S)	ΔL_s Case A (KTH)	ΔL_s Case B (KTH)
1	–650	2190	1345	2747
2	2002	2247	2166	2370
3	–6140	1379	–5220	1473
4	–17613	800	–5624	1456
5	–14792	680	–5326	1278
6	–1936	948	–29	1267
7	–5044	679	–1663	1336
8	–3359	631	–1782	1030
9	–1345	623	–293	953
10	–8561	–63	–3926	629
11	–4035	180	–838	817
12	–10850	–520	–4073	396

Case A = $L_S^{msr} - L_S^{calc\,[ao]Al}$

Case B = $L_S^{msr} - L_S^{calc\,[ao]Si}$

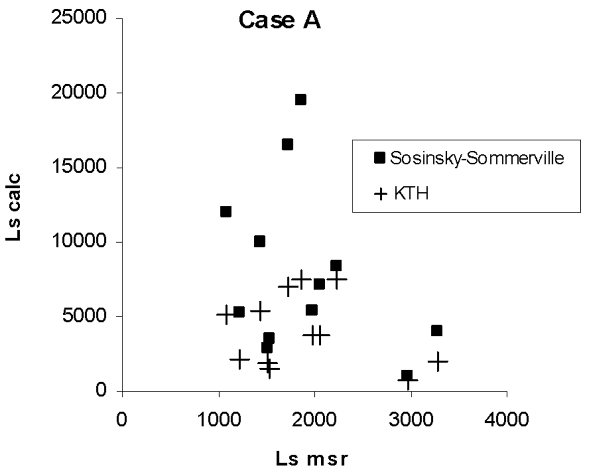

Figure 1: Comparison of the relationship between measured and calculated sulfur partition ratio L_s for the different calculated values of lg C_s (S-S and KTH) and $[a_O]_{Al}$ oxygen activities

Slika 1: Primerjava odvisnosti med izmerjenimi in izračunanimi vrednostmi deleža porazdelitve žvepla L_s za različne izračunane vrednosti lg C_s (S-S in KTH) in aktivnostjo kisika $[a_O]_{Al}$

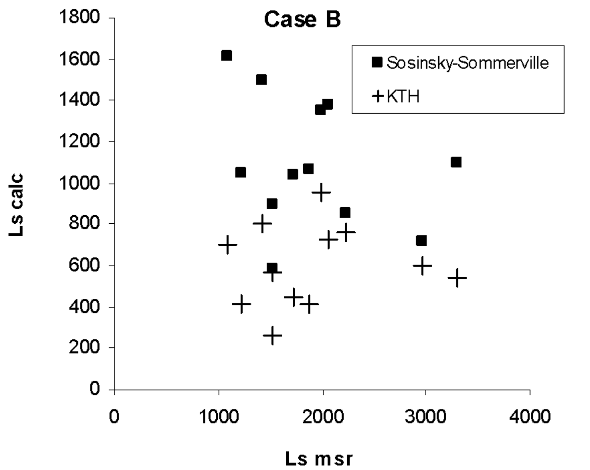

Figure 2: Comparison of the relationship between measured and calculated sulfur partition ratio L_s for the different calculated values of lg C_s (S-S and KTH) and $[a_O]_{Si}$ oxygen activities

Slika 2: Primerjava odvisnosti med izmerjenimi in izračunanimi vrednostmi deleža porazdelitve žvepla L_s za različne izračunane vrednosti lg C_s (S-S in KTH) in aktivnostjo kisika $[a_O]_{Si}$

obtained, since the vacuum degassing time for all the heats was almost the same.

Table 5 shows the differences between the measured and calculated values of the sulfur partition ratio ΔL_s for the analysed cases. The "–" means that the calculated values of L_s are higher than the measured values.

It is clear that in Case A both models give calculated values that are higher than the measured values for almost all the analyzed heats. One possible reason for the increased deviation between the measured and calculated values of the sulfur distribution ratio L_s could be the fact that using the well-known Ohta and Suito equations[8] to calculate the alumina activity might not be appropriate for the slags whose silica content was too far away from the specified lower limit of w = 10 %. The second reason could be the relatively small number of analysed samples.

4 CONCLUSIONS

1. The equilibrium sulfur partition ratio, calculated by considering the reaction 2[Al] + 3[O] = (Al_2O_3) in equilibrium for the calculation of the oxygen activity in the steel during ladle treatment, was much higher compared with the reaction [Si] + 2[O] = (SiO_2) with the measured sulfur partition ratio.
2. The Sosinsky-Sommerville optical basicity model gives higher values of the sulfur partition ratio L_s compared with the KTH model.
3. The possible reason for the increased deviation between the measured and the calculated values of sulfur partition ratio L_s was that the use of the well-known Ohta-Suito equations to calculate the alumina activity might not be appropriate for the slags whose silica content was too far away from the specified lower limit of w = 10 %.
4. The KTH model is an applicable tool to predict the sulfur partition ratio L_s.

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