

EFFECT OF ZnO/PbO AND FeO_x/SiO₂ RATIO ON THE VISCOSITY OF LEAD SMELTING SLAGS

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Abstract

The effect of ZnO/PbO and FeO_x/SiO₂ on the viscosity of the PbO-ZnO-Fe₃O₄-SiO₂-CaO slag was measured using the rotating spindle method. The slag viscosity decreased with decreasing ZnO/PbO mass ratio because of the depolymerization of the silicate structures. The viscosity decreased with increasing FeO_x/SiO₂ mass ratio as the experimental temperature was above 1623 K, while the viscosity increased significantly with the increase of FeO_x/SiO₂ as the experimental temperature below 1623 K because of the phase transition and the change of slag melting point. According to XRD analysis of as-quenched slag, the spinel and the zincite phase increased with increasing FeO_x/SiO₂. Increasing ZnO/PbO and FeO_x/SiO₂ could enhance the crystallization capacity of the slag. FTIR analysis revealed that the degree of polymerization of the as-quenched slags decreased with decreasing ZnO/PbO and increasing FeO_x/SiO₂. The temperature dependencies of the viscosity on ZnO/PbO and FeO_x/SiO₂ were investigated, and the apparent activation energies of each system were found to be between 169.5 to 227.4 KJ/mol, and 151.1 to 676.4 KJ/mol, respectively.

Keywords: Viscosity; High lead and zinc slag; Slag structure; Apparent activation energy

1. Introduction

Lead sulfide concentrate is the main raw material for lead smelting. The traditional routes of extracting lead from lead sulfide concentrate include a desulfurization process in a bath smelting process, followed by a reduction process to obtain metal lead. The oxygen-enriched bottom blown smelting in a desulfurization process is widely used in lead and copper smelting processes in China due to low energy consumption and due to it being environmentally friendly [1-2]. The limited resources of high grade lead concentrate have aroused interest in the use of alternative lead bearing material which is seldom used in the bottom blown furnace due to the restrictions on the composition of raw materials in the existing lead smelting processes. The high Zn content raw materials such as lead and zinc mixed concentrate are especially abundant in China. Due to the high zinc content these raw materials will lead to the reduction of PbO content in the oxidation slag, the variation in the mass transfer in desulfurization process, the increase of slag melting point, and the change of slag viscosity. Viscosity is one of the important physiochemical properties of slag, and low viscosity could promote the efficiency of the desulfurization reaction of slag. Considering the effect of slag composition on its

physical properties, the slag composition must be optimized to accommodate changes to its viscosity.

FeO_x/SiO₂ is an effective means to adjust slag physical property in the desulfurization process of lead smelting [3]; it is important to study the effect of FeO_x/SiO₂ on the viscosity of slag with high ZnO content. Therefore, in order to make the oxidation furnace operate effectively, it is necessary to understand the dependence of slag viscosity on temperature and composition. Gupta [4] studied the viscosity of PbO-SiO₂ melts and found that the viscosity of slag decreased with the increasing content of PbO because of the depolymerization of the silicate network structure. Battle and Hager [5] found that the addition of SiO₂ increased the viscosity of lead smelting slag, while the additions of ZnO decreased the viscosity at high temperatures, but raised the slag liquidus temperature. Lv et al. [6] investigated the effect of FeO content on the viscosity and structure in the SiO₂-MgO-FeO-CaO-Al₂O₃ slag system. The results showed that the viscosity decreased and the mass fraction of the suspended particles increased with the increase of FeO content.

The final desulfurization slag can usually be approximately considered a mixture of oxides including PbO, ZnO, FeO_x, SiO₂, and CaO from the oxidation of lead raw material and the flux of oxygen-

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enriched bottom blown furnace. However, there is a lack of the available data with respect to the viscosity of PbO-ZnO-Fe₃O₄-SiO₂-CaO system slag. Therefore, the effect of ZnO/PbO and FeO_x/SiO₂ on the viscosity in PbO-ZnO-Fe₃O₄-SiO₂-CaO system was initially investigated in this article, and the effect of ZnO/PbO and FeO_x/SiO₂ on the activation energy (E_a) for the viscous flow was estimated as well. The phase relations of the slag were studied by XRD. Furthermore, Fourier transform infra-red (FTIR) techniques were used to obtain the structural information of the molten slag.

2. Experimental

2.1 Sample preparation

The slag used in this experiment was synthesized with analytical pure grade PbO, ZnO, Fe₃O₄, SiO₂, and CaO provided by Sinopharm Chemical Reagent co., Ltd. The slag composition (FeO_x/SiO₂=1.08, CaO/SiO₂=0.5) was based on the chemical

Table 1. Chemical composition of the slag used (wt. %)

No.	PbO	ZnO	SiO ₂	Fe ₃ O ₄	CaO	FeO _x /SiO ₂	CaO/SiO ₂
1	45	15	15.52	16.72	7.76	1.08	0.5
2	42.5	17.5	15.52	16.72	7.76	1.08	0.5
3	40	20	15.52	16.72	7.76	1.08	0.5
4	37.5	22.5	15.52	16.72	7.76	1.08	0.5
5	35	25	15.52	16.72	7.76	1.08	0.5
6	32.5	27.5	15.52	16.72	7.76	1.08	0.5
7	30	30	15.52	16.72	7.76	1.08	0.5
8	27.5	32.5	15.52	16.72	7.76	1.08	0.5
9	27.5	32.5	18.61	12.08	9.3	0.65	0.5
10	27.5	32.5	16.95	14.58	8.475	0.86	0.5
11	27.5	32.5	12.83	20.74	6.42	1.62	0.5

Table 2. The chemical composition of slag after the viscosity testing (wt. %)

Number	PbO	ZnO	SiO ₂	Fe ₃ O ₄	CaO	Al ₂ O ₃	Fe ²⁺ /TFe	FeO _x /SiO ₂	CaO/ SiO ₂
1	45.3	14.42	14.68	15.7	7.45	2.45	32.6	1.07	0.507
2	42.9	17.15	14.53	15.55	7.47	2.4	32.2	1.07	0.514
3	40.42	19.56	14.47	15.72	7.36	2.47	32.1	1.09	0.509
4	37.76	22.15	14.67	15.64	7.47	2.31	32.9	1.07	0.509
5	35.3	24.56	14.62	15.78	7.39	2.35	32.6	1.08	0.505
6	32.75	27.1	14.45	15.75	7.53	2.42	32.4	1.09	0.521
7	30.32	29.85	14.56	15.53	7.35	2.39	32.7	1.07	0.505
8	27.2	32.2	14.92	15.95	7.41	2.32	32.5	1.07	0.497
9	27.4	32.21	17.61	11.56	8.92	2.3	32.8	0.66	0.507
10	27.28	32.4	15.97	13.98	8.02	2.35	32.1	0.88	0.503
11	27.25	32.08	12.12	20.05	6.09	2.41	32.1	1.65	0.502

composition of oxidation slag of typical oxygen-enriched bottom blown furnaces in the lead smelting industry in China.

The reagent was weighted according to the required composition given in Table 1 and mixed to become homogeneous. The loss caused by PbO volatilization [7] at high temperature was compensated by the excess addition of PbO (~1.09 times the theoretical quantity).

The composition of the slag after the experiment was presented in Table 2; it can be seen that there were some changes in the composition of the slag after the viscosity measurement. Besides, the test results showed that Al₂O₃ concentration (less than 2.5 wt. %) was dissolved in the slag from the corrosion of the crucible in viscosity measurement. In addition, the Al₂O₃ content in the slag before and after the viscosity measurement was measured. The results illustrated that the Al₂O₃ content varied in the range from 2.32 wt. % to 2.45 wt. %, and the change of Al₂O₃ content before and after the viscosity measurement was less than 0.15 wt. %, which indicated that the enrichment of Al₂O₃ in the slag from the corrosion of the crucible was mainly in the process of heating and equilibrium (1623 K). Therefore, the influence of the change of Al₂O₃ content in the slag on viscosity measurement could be neglected. The same method was used to measure the viscosity of copper smelting slag [8-9], and the results demonstrated that the solubility of Al₂O₃ was negligible in the copper smelting or "Cu₂O"-SiO₂-Al₂O₃ melt slag under experimental conditions.

2.2 Experimental apparatus and procedures

In this research, the viscosity of molten slag was determined by the rotating cylinder method. Fig. 1 shows the schematic diagram of the viscosity experimental device. An alumina crucible (inner



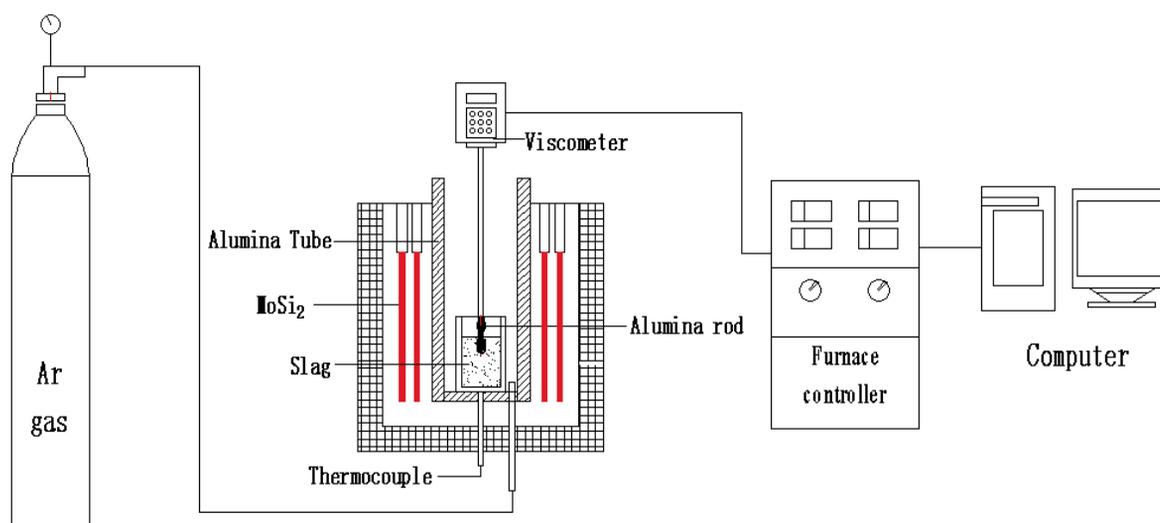


Figure 1. Schematic diagram of viscosity experimental device

diameter: 40 mm, height: 115mm) was put in a vertical tube furnace with MoSi_2 heating elements. The Pt-13 pct Rh/Pt thermocouple at the bottom of the crucible was used to monitor the slag temperature. Meanwhile, the furnace temperature was controlled by the computer program. The rotating bob was made of alumina, and the viscometer was calibrated with castor oil with known viscosity before the experiment. It should be pointed out that the bob which was used to measure the viscosity had almost no change in the diameter before and after the experiment, which indicated that the influence of the diameter variation on the viscosity measurement could be neglected.

About 250 g of mixture was heated to 1623 K and equilibrated for 1h to obtain the homogeneous slag. The bob was inserted into the molten slag and maintained at 10 mm above the bottom of the crucible, and then the viscometer (model: RTW-10) started measuring with the spindle at a fixed rate of 200 rpm. The value of slag viscosity was calculated and recorded by the computer. The Ar was introduced into the furnace at a flow rate of 1.2 L/min to prevent the oxidation of slag. The errors of viscosity measurement were less than 0.002 Pa·S. When the temperature reached at each target temperature, the slag equilibrated at each experimental temperature for 30 minutes.

Kim and Sohn [10] used the breaking temperature to describe the property of slag. The breaking temperature is the critical temperature at which the viscosity varied greatly during the cooling cycle. The molten slag could be assumed to be full liquid above the breaking temperature [11].

After the viscosity measurement, the slag was reheated to the target temperature. Subsequently, parts of the slag were removed from the slag by the iron rod and quenched in water for the structure analysis. The slag phase and the structure were analyzed by X-ray

diffractometer (Bruker, D8 ADVANCE, Germany) and Fourier Transform Infrared Spectrometer (Nicolet, iS50, USA), respectively.

3. Results and Discussion

3.1 Effect of ZnO/PbO on slag viscosity

Fig. 2 shows the viscosity changes of the $\text{PbO-ZnO-Fe}_3\text{O}_4\text{-SiO}_2\text{-CaO}$ system as a function of temperature with varying ZnO/PbO and a constant $\text{FeO}_x/\text{SiO}_2$ of 1.08 and CaO/SiO_2 of 0.5. The variation of ZnO/PbO in $\text{PbO-ZnO-Fe}_3\text{O}_4\text{-SiO}_2\text{-CaO}$ slag system has a significant influence on the slag viscosity. The viscosity increased as the ZnO/PbO increased. A further increase of ZnO/PbO above 1 had a remarkable effect in enhancing the viscosity. According to work done by Gupta [4] and Jin [12] et al., both PbO and ZnO suggested to be a weak basic oxide in a silicate melts, which could have decreased the slag viscosity by depolymerizing the slag network structure. It seems that PbO is better than ZnO in

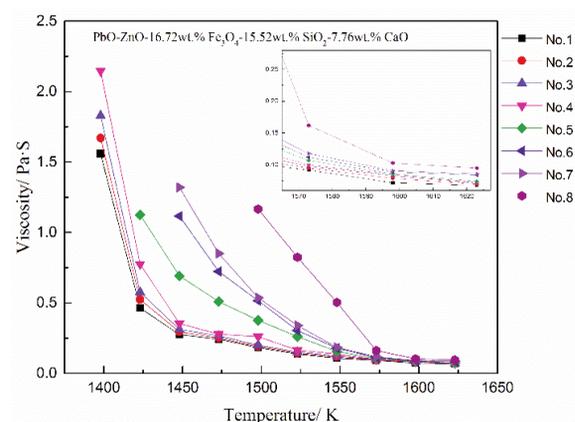


Figure 2. Viscosity of $\text{PbO-ZnO-Fe}_3\text{O}_4\text{-SiO}_2\text{-CaO}$ system as a function of temperature with different ZnO/PbO



depolymerizing the network structure; thus, increasing PbO/ZnO decreases the slag viscosity. According to the study of Jin et al. [12], the increasing ZnO content in slag would lead to the increase of crystallization capacity of the slag. In this study, the breaking temperature increased rapidly with the increase of PbO/ZnO, which further confirmed this point.

Fig. 3 shows the XRD analysis of the as-quenched slags with various ZnO/PbO in the PbO-ZnO-Fe₃O₄-SiO₂-CaO slag system. It can be seen that the main crystalline phase for the ZnO/PbO = 0.33 (ZnO=15 wt. %, PbO=45 wt. %) was identified as the spinel phase (Zn_xFe_{3-x}O_{4+y}). The peaks intensity of the spinel phase was gradually increased as the increase of ZnO/PbO. When the ZnO/PbO increased to 0.714 (ZnO=25 wt. %, PbO=35 wt. %), the diffraction peaks for the zincite phase (ZnO) emerged. As the ZnO/PbO continued to increase, the diffraction peaks for ZnO and Fe₃O₄ became more pronounced. It is reported that the increase of the spinel and the zincite phase in the slag is the main reason for the increase of viscosity in the lead bath smelting process [13]. Thus, a subsequent decrease of ZnO/PbO ratio showed an increase effect in lowering the slag viscosity.

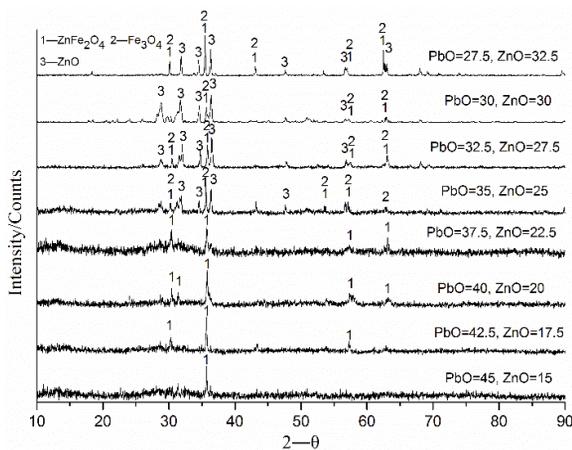


Figure 3. The XRD analysis of as-quenched slag with different ZnO/PbO

The temperature dependence of viscosity can be represented by the Arrhenius equation [8].

$$\ln \eta = A + \frac{E_{\eta}}{R \cdot T} \quad (1)$$

where η is the viscosity, Pa·S; A is a constant, Pa·S; R is the gas constant, 8.314 J/mol⁻¹·K⁻¹; T is the absolute temperature, K; E_{η} is the apparent activation energy stand for the sensibility of viscosity to temperature, and the change of E_{η} can illustrate the variation in the frictional resistance of viscous flow and the change of the slag structure [14], J/mol;

Fig. 4 shows a linear fit between the natural

logarithm of the viscosity ($\ln \eta$) and the reciprocal temperature ($1/T$). Clearly, the viscosity decreased with increasing temperature. The value of E_{η} could be obtained from the linear fitting results of $\ln \eta$ with $1/T$. The corresponding E_{η} with variation slag composition was calculated to be between 169.5 and 227.4 KJ/mol, and the results were listed in Table 3. It can be seen that the increase of ZnO/PbO led to the increase of apparent activation energy of the viscosity. Moreover, the decrease of E_{η} illustrated the formation of small structural units in the slag and the decrease of resistance for viscous flow [12], which was similar to the change of measured viscosity.

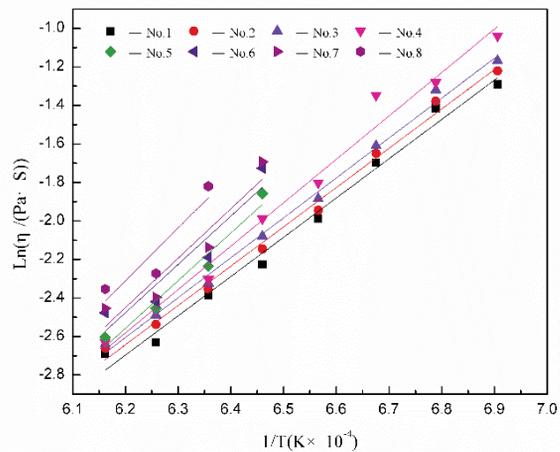


Figure 4. The relationship between natural logarithm of the viscosity ($\ln \eta$) and the reciprocal temperature ($1/T$) at different ZnO/PbO

Table 3. The apparent activation energy of viscosity with different ZnO/PbO in PbO-ZnO-Fe₃O₄-SiO₂-CaO slag system

Slag Number of Table 1	Range of Temperature (K)	E_{η} (KJ/mol)
1	1448 to 1623	169.5
2	1448 to 1623	169.8
3	1448 to 1623	172.2
4	1448 to 1623	187.3
5	1548 to 1623	205.8
6	1548 to 1623	208.5
7	1548 to 1623	213.3
8	1573 to 1623	227.4

3.2 Effect of FeO_x/SiO₂ on slag viscosity

Fig. 5 shows the viscosity variation of the PbO-ZnO-Fe₃O₄-SiO₂-CaO system as a function of temperature with varying FeO_x/SiO₂ and a constant ZnO/PbO of 1.18 and CaO/SiO₂ of 0.5, it can be seen that the increase of FeO_x/SiO₂ could lower the viscosity of the slag as experimental temperature was

at 1623 K. The basic oxide behavior of Fe_xO , which, like CaO, is a modifier of the network structure of silicate [15]; it was confirmed by the FTIR results as well. The same result was reported by other investigators [6]. However, when experimental temperature was below 1598 K, the viscosity increased significantly with the increase of FeO_x/SiO_2 . Note that the increase of Fe_3O_4 in lead smelting slag can increase the melting point of the slag, thus causing various viscosity [13]. With the increase of FeO_x/SiO_2 , the gradient of the viscosity curve got sharper below the breaking temperature, which further confirmed that the breaking temperature of the slag increased with the increase of Fe/SiO_2 .

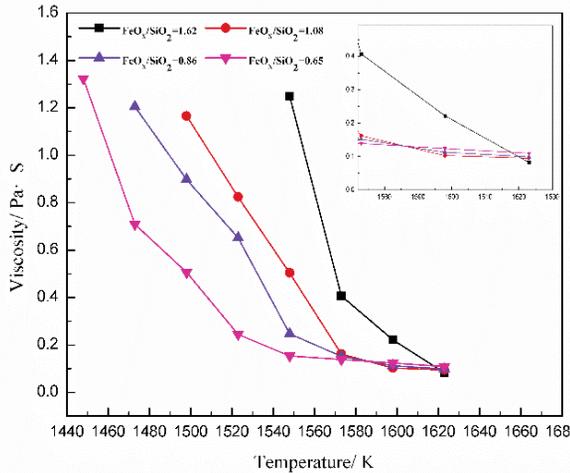


Figure 5. Viscosities in the PbO-ZnO- Fe_3O_4 - SiO_2 -CaO slag system as a function of temperature with different FeO_x/SiO_2

In order to know the relationship between the viscosity and the slag composition with the increase of FeO_x/SiO_2 , the XRD analysis of the as-quenched slags with various FeO_x/SiO_2 in the PbO-ZnO- Fe_3O_4 - SiO_2 -CaO slag system was carried out. As shown in Fig. 6, the main crystalline phase of the slag was identified as zincite when FeO_x/SiO_2 was at 0.65. With FeO_x/SiO_2 increasing to 0.86, the diffraction peaks for the spinel phase became observable. Moreover, the spinel phase increased as FeO_x/SiO_2 continued to increase. This illustrated that the addition of silica could inhibit the formation of the spinel phase.

Based on the above study results, the increase of FeO_x/SiO_2 would not only increase the slag melting point, but also led to the phase transformation of the slag. This could be the reason that led to the change of the slag with various FeO_x/SiO_2 ratio as experimental temperature was below 1598 K.

The viscosities of various FeO_x/SiO_2 slag as a function of reciprocal of the temperature were shown in Fig. 7. E_η in this system was estimated to be

between 151.1 and 676.4 KJ/mol, and the corresponding E_η with various FeO_x/SiO_2 was listed in Table 4.

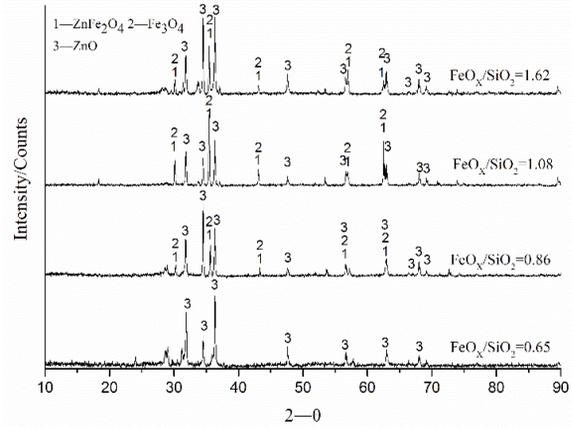


Figure 6. The XRD analysis of as-quenched slag with different FeO_x/SiO_2

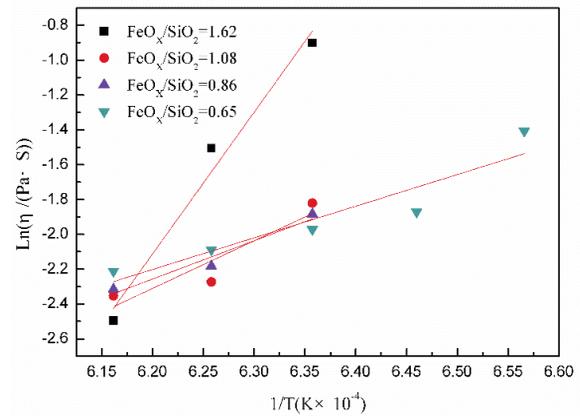


Figure 7. The relationship between natural logarithm of the viscosity ($Ln\eta$) and the reciprocal temperature ($1/T$) at different FeO_x/SiO_2

Table 4. The apparent activation energy of viscosity with different FeO_x/SiO_2 in PbO-ZnO- SiO_2 - Fe_3O_4 -CaO slag system

Slag Number of Table 1	Range of Temperature (K)	E_η (KJ/mol)
9	1523 to 1623	151.1
10	1573 to 1623	182.8
11	1573 to 1623	676.4

3.3 FTIR Analysis

It is generally considered that the viscosity is related to the degree of polymerization of silicate slag [16-17]. The FTIR transmittance of the as-quenched



slag as a function of wavenumbers at different ZnO/PbO and FeO_x/SiO₂ are exhibited in Figure 10 and 11. The infrared spectra of the silicate slag is mostly concentrated in the wavenumber range of 1200 to 400 cm⁻¹ [18-20]. This wavenumber region includes the stretching vibration bands of the [SiO₄]-tetrahedra (wavenumber: 1100-850) and [AlO₄]-tetrahedra (wavenumber: 750-590), and the Si-O bending vibration bands (wavenumber: ~500), respectively [6].

In the study of silicate melt structure, the average number of non-bridge oxygen (NBO/Si) is often used to illustrate the polymerization of silicate slag structures [21], where the lower NBO/Si indicates the higher degree of polymerization of the molten slag [11]. The wavenumber region between 1200 and 800 cm⁻¹ is widely known to be the [SiO₄]-tetrahedra peaks [11] with various NBO/Si (NBO/Si=1, 2, 3, 4). According to the study of B. O. Mysen et al [22], the wavenumber region of 1100-1050 cm⁻¹, 980-950 cm⁻¹, 920-900 cm⁻¹, 880-850 cm⁻¹ represent the band groups of [SiO₄]- tetrahedral with NBO/Si = 1 (sheets), 2 (chains), 3 (dimers) and 4 (monomers), respectively. Clearly, as shown in Fig. 8, the [SiO₄]-tetrahedra

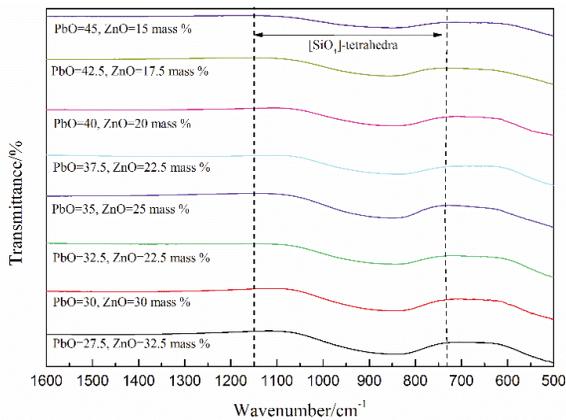


Figure 8. FTIR results of as-quenched slag from 1623 K with different ZnO/PbO ratio

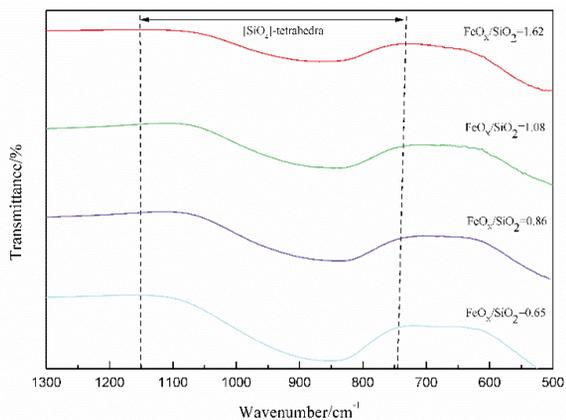


Figure 9. FTIR results of as-quenched slag from 1623 K with different FeO_x/SiO₂ ratio

peaks at about 750 to 1150 cm⁻¹ got weaker when the ZnO/PbO was decreased, which demonstrated that the structure of the slag could be depolymerized by the decrease of ZnO/PbO. The results observed in Fig. 9 show that the characteristic stretching vibration band for the large silicate network structures became less pronounced and the depth of the convoluted band became shallower, which illustrated that the increase of FeO_x/SiO₂ was likely to depolymerize the silicate network structure into simpler polymer type units. Therefore, the depolymerization of the silicate network structure could reduce the polymerization degree of complex viscous units, thus decreasing the slag viscosity.

4. Conclusion

The viscosities of PbO-ZnO-Fe₃O₄-SiO₂-CaO lead smelting slags were measured using the rotational spindle method, and the phase structure was studied by XRD and FTIR analysis. The following conclusions could be obtained:

1. The viscosity of the molten slag decreased as the ZnO/PbO mass ratio decreased from 1.18 to 0.33, and the decrease trend was more obvious when ZnO/PbO was less than 0.714 and the temperature was below 1523 K.

2. The effect of FeO_x/SiO₂ in PbO-ZnO-Fe₃O₄-SiO₂-CaO system on the viscosity was complicated due to the phase transition of the basic slag and the change of the slag melting point ranging from 1523 to 1573 K. XRD analysis of as-quenched molten slag samples revealed that the phase could be changed in the slags with the lower FeO_x/SiO₂ mass ratio.

3. The apparent activation energy of viscosity at different ZnO/PbO and FeO_x/SiO₂ was estimated to 169.5 to 227.4 KJ/mol, and 151.1 to 676.4 KJ/mol, respectively. The variation of the apparent activation energy was similar to the change in the viscosity.

4. The PbO or FeO_x is likely to be a network modifier and provided free O²⁻ to depolymerize [Si_xO_y]ⁿ-tetrahedra into simpler polymertype units, which resulted in the decrease of the viscosity.

Acknowledgements

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UTICAJ ODNOSA ZnO/PbO I FeO_x/SiO₂ NA VISKOZNOST OLOVNE ŠLJAKE

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Apstrakt

Uticao ZnO/PbO i FeO_x/SiO₂ na viskoznost PbO-ZnO-Fe₃O₄-SiO₂-CaO šljake izmeren je korišćenjem metoda rotacione osovine. Viskoznost šljake opadala je sa opadanjem ZnO/PbO masenog odnosa zbog depolimerizacije silikatnih struktura. Viskoznost je opadala sa povećanjem masenog odnosa FeO_x/SiO₂ pri temperaturi iznad 1623 K, dok je viskoznost značajno porasla povećanjem FeO_x/SiO₂ pri temperaturi ispod 1623 K zbog faznog prelaza i promene tačke topljenja šljake. Prema XRD analizi kaljene šljake, faza spinela i cink oksida se povećala povećanjem FeO_x/SiO₂. Povećanje ZnO/PbO i FeO_x/SiO₂ je poboljšalo kapacitet kristalizacije šljake. FTIR analiza je otkrila da se stepen polimerizacije kaljene šljake smanjivao sa smanjenjem ZnO/PbO i povećanjem FeO_x/SiO₂. Ispitivane su temperaturne zavisnosti viskoznosti na ZnO/PbO i FeO_x/SiO₂, i nađeno je da su energije aktivacije u ovim sistemima bile od 169.5 do 227.4 KJ/mol i od 151.1 do 676.4 KJ/mol, respektivno.

Ključne reči: Viskozitet; Šljaka sa visokim udelom cinka i olova; Struktura šljake; Energija aktivacije.

