

Sb DISTRIBUTION IN THE PHASES OF SiO₂ SATURATED Sb-Fe-O-SiO₂-CaO SYSTEM IN AIR

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Abstract

Sb distribution in the phases of SiO₂ saturated Sb-Fe-O-SiO₂-CaO system was determined for the first time through high-temperature experiment and quenching techniques, followed by Electron probe X-ray microanalysis (EPMA) in air ($P_{\text{tot}} = 1 \text{ atm}$, $P_{\text{O}_2} = 0.21 \text{ atm}$). The phases were quantified in the temperature range of 900 °C-1200 °C and the effects of Fe/SiO₂ (mass fraction) and CaO/SiO₂ (mass fraction) on Sb₂O₃ content in the Sb-Fe-O-SiO₂-CaO system were investigated at 1200 °C. The results indicated that the Sb-containing phase primarily existed in the solid solution phase at 1200 °C. With the increase of temperature from 1100 °C to 1200 °C, the Sb₂O₃ content in the solid solution phase increased drastically from 7.52 wt% to 17.36 wt%. Lowering the values of CaO/SiO₂ and Fe/SiO₂ in the smelting process effectively reduced Sb₂O₃ content in the slag. The verification of the experiment results suggested that the antimony content in slag was 0.57 wt%, the crude antimony yield rate was lower than 4%, and the crude antimony grade was beyond 94 wt%, which meant that the reduction of antimony content in the slag could be achieved.

Keywords: Phase relations; Liquidus; Microstructure; Pyrometallurgy

1. Introduction

Antimony is considered an important strategic resource that is widely used in the production of ceramics, fire retardants, medicaments, and alloys [1]. Currently, antimony sulfide concentrate is the main raw material for the antimony production. The metallic antimony is primarily produced via different techniques, such as the antimony blast furnace and volatilization bath smelting processes [2-4]. Due to the shortage of antimony resources and increase in amount of secondary raw materials in metallurgical industry, the production processes urgently need to be re-designed or optimized [5]. Regarding the raw material composition during smelting, the Sb-Fe-O-SiO₂-CaO system is widely adopted in the pyrometallurgical processing for antimony extraction. Phase relations of the Sb-Fe-O-SiO₂-CaO system are significant in the development of new flow sheets and reduction of operating costs. Also, the data of antimony content in Sb-Fe-O-SiO₂-CaO system is important to limit the antimony content in the slag predictions and process philosophy improvement. However, the experimental phase relations regarding this system are still ambiguous.

To develop accurate descriptions of the high-order

systems, the phase relations of simple binary and ternary systems should be investigated. The phase relations of the CaO-SiO₂, FeO-Fe₂O₃-SiO₂, and FeO_x-CaO-SiO₂ subsystems have been systematically studied by several researchers using high-temperature experiments and rapid quenching technique [6-13]. The phase relations of FeO-Fe₂O₃-SiO₂ system in equilibrium with O₂ pressures ranging from 10-10.9 to 1 atm have been studied and two ternary invariant points were determined in this system. The phases of tridymite (SiO₂), fayalite (2FeO·SiO₂), magnetite (FeO·Fe₂O₃), a liquid with composition 35 wt% SiO₂, 11 wt% Fe₂O₃, and 54 wt% FeO coexisted in equilibrium at 1140 °C, while the phases of fayalite (2FeO·SiO₂), magnetite (FeO·Fe₂O₃), wüstite with a composition corresponding to 35 wt% Fe₂O₃, a liquid with composition 22 wt% SiO₂, 14wt% Fe₂O₃, and 64 wt% FeO coexisted in equilibrium at 1150 °C [7]. The phase relations in the FeO_x-CaO-SiO₂ system at intermediate oxygen partial pressures and the temperatures ranging from 1200 °C to 1350 °C were analyzed via equilibration/rapid-quenching/Electron probe X-ray microanalysis [8]. As per the above-mentioned works, the high-temperature experiment and rapid quenching technique is one of the most precise and flexible technique employed to obtain

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accurate experimental phase relations for systems with “sluggish” phase change [14,15].

Inspired by the above approach, herein, Sb distribution in the phases of SiO_2 saturated Sb-Fe-O- SiO_2 -CaO system in air was investigated via the high-temperature experiment and quenching techniques, followed by EPMA and the verification experiments. The work would provide novel experimental data for the fundamental research.

2. Experimentals

2.1. Preparation of initial mixtures

In the high-temperature experiments, chemically pure calcium oxide powder (CaO), silicon dioxide powder (SiO_2), antimony trioxide powder (Sb_2O_3), and ferrous oxalate dehydrate powder ($\text{FeC}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$) were employed as raw materials. The powders were dried at 373 K, weighed in desired proportions, mixed thoroughly in an agate mortar, and then, flaked at 5 MPa with a 9 mm-diameter pressing tool. The total weight of each flake was 0.15 g. The thin flakes have advantage in the molten phase quenching. The flakes reacted at a fixed temperature for 4 h. Thereafter, flakes were quenched in ice water, dried and analyzed.

2.2. Experimental apparatus and technique

The experiments were carried out in a horizontal laboratory tube furnace with silicon carbide heating elements (Figure 1). The experiments were conducted with the left and right gates of the reaction tube open to ambient atmosphere ($P_{\text{tot}} = 1 \text{ atm}$, $P_{\text{O}_2} = 0.21 \text{ atm}$).

Firstly, the specimen was placed into a quartz crucible. Subsequently, the quartz crucible containing the specimen was placed into an alumina ark. Then, the alumina ark was pushed into the uniformly hot temperature zone of the furnace with a steel rod. The temperature of the uniformly hot zone was measured using a calibrated thermocouple. The furnace temperature accuracy was $\pm 2 \text{ K}$.

To avoid the effect of the quartz crucible on the experimental results, a spacer that had the same composition as the specimen was placed at the bottom of the quartz crucible. The specimen was taken out and quenched rapidly into the ice water at the end of the reaction time. It was consequently dried and inlaid into the resin. The specimen was analyzed after it was prepared by pre-grinding and polishing techniques.

2.3. Analysis

The microstructures and phase compositions were examined via Electron probe X-ray microanalysis (EPMA-1720H) using the cross section of the polished samples. Cross sections of polished samples were also characterized using X-ray diffraction analysis ($\text{Cu K}\alpha$, $\lambda = 0.154056 \text{ nm}$; Rigaku-TTR III).

3. Results and discussion

3.1. Chemical compositions and microstructures of samples

The compositions of the initial mixtures are listed in Table 1. All of the well-quenched samples were carefully examined and analyzed and each sample

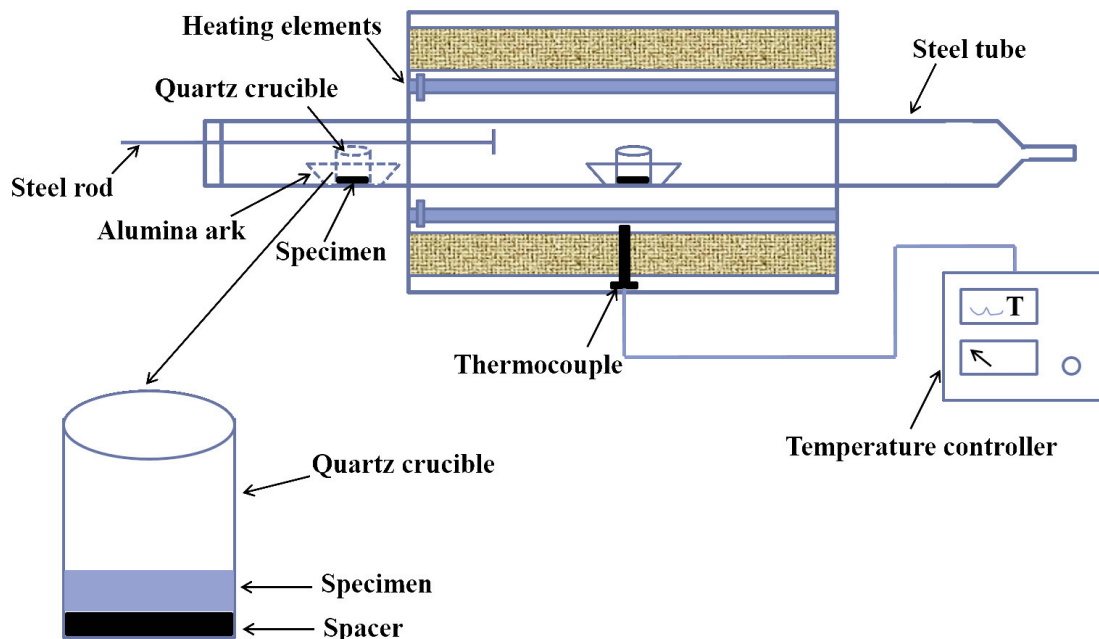


Figure 1. Schematic vertical section of the furnace and its auxiliaries

Table 1. The composition of the initial mixtures (wt%)

FeO	SiO ₂	CaO	Sb ₂ O ₃
28.66	53.07	13.27	5.00

consisted of four homogeneous phases. The compositions of the phases were measured using EPMA. At least five independent measuring points from each phase were analyzed with EPMA for each sample, taken from their different well-quenched parts to quantify the chemical composition. The obtained EPMA results of the high-temperature experiments of SiO₂ saturated Sb-Fe-O-SiO₂-CaO system in the temperature range from 900 °C to 1200 °C in air are listed in Table 2.

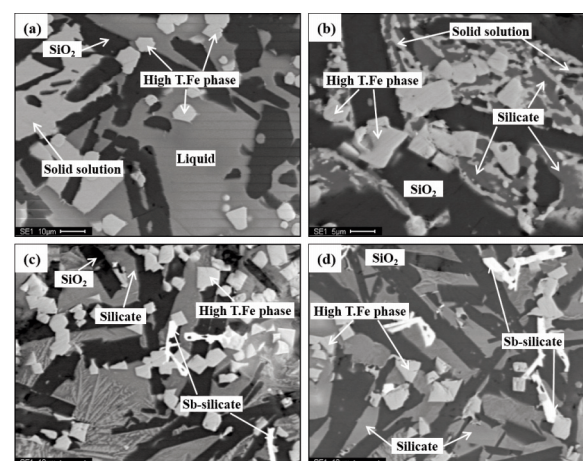
Four phases were found in the samples reacted within the temperature range from 900 °C to 1200 °C. As listed in Table 2, the Sb-containing phase primarily existed in the solid solution phase at 1200 °C. When the temperature dropped to 1100 °C, the silicate phase

Table 2. Phase composition of SiO₂ saturated Sb-Fe-O-SiO₂-CaO system at 900-1200 °C in air

No.	T / °C	Time / h	Phase	EPMA (wt%)			
				FeO	CaO	SiO ₂	Sb ₂ O ₃
1	1200	4	Solid solution	28.60	24.65	29.39	17.36
			Liquid	20.99	22.37	53.47	3.17
			SiO ₂	5.29	2.79	90.67	1.25
			High T.Fe phase	75.49	2.60	18.88	3.03
2	1100	4	Solid solution	23.71	26.82	41.94	7.52
			SiO ₂	7.00	3.03	88.75	1.22
			High T.Fe phase	75.34	2.93	18.94	2.79
			Silicate	9.75	38.91	51.34	0.00
3	1000	4	Silicate	17.14	30.80	52.05	0.00
			SiO ₂	5.99	3.43	89.22	1.36
			High T.Fe phase	78.36	2.36	15.80	3.48
			Sb-silicate	11.97	21.03	18.68	48.32
4	900	4	Silicate	15.90	29.80	50.97	3.33
			SiO ₂	4.76	3.01	90.81	1.42
			High T.Fe phase	79.36	2.23	14.43	3.98
			Sb-silicate	10.80	21.32	13.56	54.32

began to appear. The solid solution phase vanished and Sb-silicate phase appeared at the temperature of 900 °C and 1000 °C. Moreover, as the temperature increased, the Sb₂O₃ content in solid solution phase increased dramatically, from 7.52 wt% at 1100 °C to 17.36 wt% at 1200 °C. The possible reason for the phenomenon is that high temperature is beneficial for the increase of Sb₂O₃ solubility in the solid solution phase. The Sb₂O₃ content in SiO₂ and High T.Fe phase displayed a stable tendency within the temperature range of 900 °C to 1200 °C, wherein, the average contents were approximately 1.3 wt% and 3.3 wt%, respectively. Also, the Sb-containing phase primarily changed from the solid solution phase to the Sb-silicate phase at the temperatures below 1100 °C. The Sb₂O₃ contents in the Sb-silicate phases were 48.32 wt% and 54.32 wt% at 1000 °C and 900 °C, respectively, indicating that as the temperature decreased during the reaction, the Sb-containing phases primarily existed in the Sb-silicate phases.

Selected microstructures of the quenched samples from the polished sections are presented in Fig. 2. The

**Figure 2.** BSE micrographs of selected quenched and polished Sb-Fe-O-SiO₂-CaO samples of the present study at: (a) 1200 °C; (b) 1100 °C; (c) 1000 °C, and (d) 900 °C. (Fe/SiO₂ 0.42, CaO/SiO₂ 0.25)

Sb-containing phase mainly existed in the off-white block area (solid solution phase) at 1200 °C. Subsequently, when the reaction temperature was decreased from 1200 °C to 1100 °C, the Sb-containing phase (solid solution phase) changed from the off-white block to strip structure. Finally, at 1000 °C and 900 °C, the Sb-containing phase (the Sb-silicate phase) gathered into bright white block structure from strip structure.

Figure 3 illustrates the XRD patterns of the selected samples reacting at 900 °C-1200 °C. As presented in Fig. 3, the phases of Fe₂O₃, SiO₂ and Fe₂SiO₄ were determined at 1200 °C. The phases of



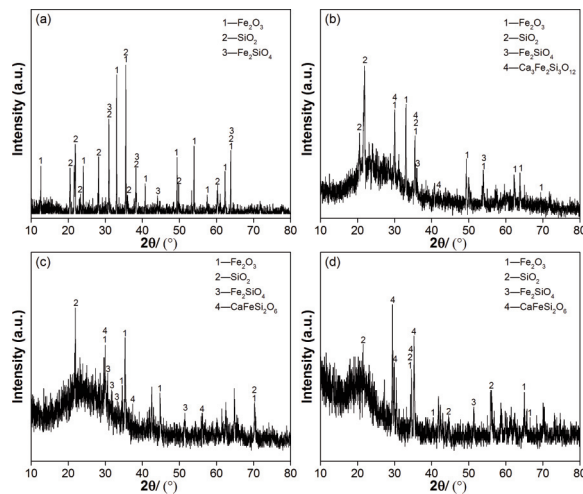


Figure 3. The XRD patterns of selected samples reacted at: (a) 1200 °C; (b) 1100 °C; (c) 1000 °C, and (d) 900 °C

Fe_2O_3 , SiO_2 , Fe_2SiO_4 , and $\text{Ca}_3\text{Fe}_2\text{Si}_3\text{O}_{12}$ were detected at 1100 °C. The phases of Fe_2O_3 , SiO_2 , Fe_2SiO_4 , and $\text{CaFeSi}_2\text{O}_6$ were detected at 1000 °C and 900 °C. Therefore, it can be inferred from the aforementioned analysis that the phases of Fe_2O_3 and Fe_2SiO_4 corresponded to the High T.Fe phase at 900 °C-1200 °C, and the phase of $\text{Ca}_3\text{Fe}_2\text{Si}_3\text{O}_{12}$ corresponded to the silicate phase at 1100 °C (Fig. 2(b)). Notably, the Sb-containing phase mainly existed in the solid solution phase and Sb-silicate at the temperature of 1100 °C-1200 °C and 900 °C-1000 °C, respectively. However, the Sb-containing phases were not detected in the XRD patterns, may be due to low Sb content in the slag system.

3.2. Effect of Fe/SiO_2 and CaO/SiO_2 on Sb_2O_3 content in $\text{Sb-Fe-O-SiO}_2\text{-CaO}$ system

The selection of slag type during smelting has an important impact on Sb_2O_3 content in the slag. To explore the change of Sb_2O_3 content during the

Table 3. Selected slag phase compositions in the present study (wt%)

Slag No.	CaO/SiO_2 mass ratio	Fe/SiO_2 mass ratio	Composition/ wt%			
			FeO	CaO	SiO_2	Sb_2O_3
1	0.25	0.40	28.66	13.27	53.07	5.00
2	0.25	0.80	42.22	okt-56	42.22	5.00
3	0.40	0.40	26.44	19.59	48.97	5.00
4	0.40	0.80	39.58	15.83	39.58	5.00
5	0.55	0.40	24.55	25.00	45.45	5.00
6	0.55	0.80	37.25	20.49	37.25	5.00
7	0.70	0.40	22.90	29.69	42.41	5.00
8	0.70	0.80	35.19	24.63	35.19	5.00

reaction, the effect of Fe/SiO_2 and CaO/SiO_2 on the Sb_2O_3 content were investigated for the current system. Selected slag phase compositions are listed in Table 3.

The selected samples were carefully analyzed and the phase compositions were measured using EPMA. The obtained EPMA results of SiO_2 saturated $\text{Sb-Fe-O-SiO}_2\text{-CaO}$ system at 1200 °C in air with various values of CaO/SiO_2 and Fe/SiO_2 are listed in Table 4. The Sb-containing phase mainly existed in the solid solution phase at 1200 °C. The Sb_2O_3 content in the solid solution phase demonstrated a stable tendency within the content range from 14.45 wt% to 17.36 wt% with CaO/SiO_2 0.84-1.03 and Fe/SiO_2 0.76-0.97. Moreover, the Sb_2O_3 content in SiO_2 and High T.Fe phase also demonstrated a stable tendency at 1200 °C. The average contents were approximately 1.45 wt% and 3.1 wt%, respectively, which is consistent with

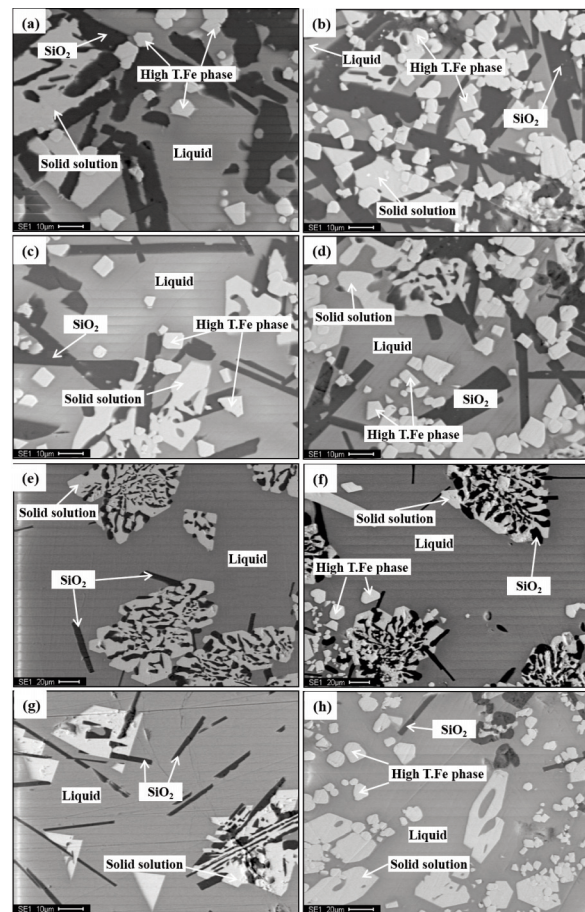


Figure 4. BSE micrographs of selected quenched and polished $\text{Sb-Fe-O-SiO}_2\text{-CaO}$ samples of the present study at 1200 °C; (a) CaO/SiO_2 0.25, Fe/SiO_2 0.40; (b) CaO/SiO_2 0.25, Fe/SiO_2 0.80; (c) CaO/SiO_2 0.40, Fe/SiO_2 0.40; (d) CaO/SiO_2 0.40, Fe/SiO_2 0.80; (e) CaO/SiO_2 0.55, Fe/SiO_2 0.40; (f) CaO/SiO_2 0.55, Fe/SiO_2 0.80; (g) CaO/SiO_2 0.70, Fe/SiO_2 0.40; (h) CaO/SiO_2 0.70, Fe/SiO_2 0.80

Table 4. Phase composition of SiO_2 saturated $\text{Sb-Fe-O-SiO}_2\text{-CaO}$ system at 1200 °C in air with various values of CaO/SiO_2 and Fe/SiO_2

No.	Time / h	Phase	EPMA (wt%)				Fe/SiO_2	CaO/SiO_2
			FeO	CaO	SiO_2	Sb_2O_3	mass ratio	mass ratio
1	4	Solid solution	28.60	24.65	29.39	17.36	0.76	0.84
		Liquid	20.99	22.37	53.47	3.17	0.31	0.42
		SiO_2	5.29	2.79	90.67	1.25	/	/
		High T.Fe phase	75.49	2.60	18.88	3.03	/	/
2	4	Solid solution	31.71	23.60	29.27	15.42	0.84	0.81
		Liquid	24.51	22.44	51.77	1.28	0.37	0.43
		SiO_2	9.21	3.29	86.33	1.17	/	/
		High T.Fe phase	79.53	2.27	14.60	3.60	/	/
3	4	Solid solution	28.91	25.51	29.46	16.12	0.76	0.87
		Liquid	22.05	23.97	53.01	0.96	0.32	0.45
		SiO_2	6.25	4.27	88.15	12055	/	/
		High T.Fe phase	76.34	3.65	17.01	3.00	/	/
4	4	Solid solution	30.56	24.70	30.29	14.45	0.78	0.82
		Liquid	24.36	23.67	51.26	0.71	0.37	0.46
		SiO_2	9.71	4.49	84.30	1.50	/	/
		High T.Fe phase	79.44	3.46	14.14	2.96	/	/
5	4	Solid solution	29.04	26.77	28.12	16.07	0.80	0.95
		Liquid	23.71	25.59	50.33	0.37	0.37	0.51
		SiO_2	6.83	7.34	84.23	1.60	/	/
6	4	Solid solution	30.77	26.41	26.77	16.05	0.89	0.99
		Liquid	24.30	25.06	49.97	0.67	0.38	0.50
		SiO_2	6.96	5.47	85.69	1.88	/	/
		High T.Fe phase	78.67	4.39	13.77	3.17	/	/
7	4	Solid solution	29.06	27.33	28.71	14.90	0.79	0.95
		Liquid	23.51	26.25	50.11	0.13	0.36	0.52
		SiO_2	7.67	8.51	82.68	1.14	/	/
8	4	Solid solution	31.68	26.25	25.48	16.59	0.97	1.03
		Liquid	25.04	24.80	49.45	0.71	0.39	0.50
		SiO_2	8.31	5.61	84.44	1.64	/	/
		High T.Fe phase	78.99	4.70	13.46	2.85	/	/



the above results. Also, as presented in Table 3, Sb_2O_3 content in the liquid phase was lower than that in the solid solution phase within the content range from 0.13 wt% to 3.17 wt% with CaO/SiO_2 0.42-0.50 and Fe/SiO_2 0.31-0.39.

Figure 4 presents the BSE micrographs of the selected quenched and polished SiO_2 saturated Sb-Fe-O- SiO_2 -CaO samples at 1200 °C in air with various values of CaO/SiO_2 and Fe/SiO_2 . As shown in Fig. 4, the High T.Fe phase disappeared when the CaO/SiO_2 increased to 0.55 with Fe/SiO_2 fixed at 0.40. The possible reason for the phenomenon may be that as the CaO content increased, CaO combined with other phases to form a low melting phase in the liquid phase. In contrast, when the CaO/SiO_2 increased from 0.25 to 0.70 with Fe/SiO_2 fixed at 0.80, the phase composition only changed slightly. Therefore, the above analysis illustrates that lowering the values of CaO/SiO_2 and Fe/SiO_2 in the smelting process can effectively reduce Sb_2O_3 content in the slag.

3.3. Verification experiment

As per the foregoing theoretical analysis, the verification experiment on the bath smelting of antimony sulfide concentrate was performed at the smelting temperature of 1200 °C, oxygen concentration of 80%, gas pressure of 0.18 MPa, oxygen flow rate of 0.16 m³/h, slag composition of Fe/SiO_2 0.4, CaO/SiO_2 0.5, and reaction time of 40 min. The experimental results are listed in Table 5. The antimony content in slag was 0.57 wt%, which is consistent with the above analysis. Also, the crude antimony yield rate was lower than 4% and crude antimony grade was beyond 94 wt%, demonstrating that the antimony-containing phases were mainly volatilized into dust during smelting, which further meant that the reduction of antimony content in slag could be achieved.

Table 5. Composition analysis of experimental product

Project	Test results
Antimony content in slag	0.57 wt%
Crude antimony rate	3.6%
Crude antimony grade	94.83 wt%

4. Conclusion

(1) The effect of temperature on the phase compositions within the temperature range of 900 °C to 1200 °C was systematically investigated. The results suggest that the Sb-containing phase mainly existed in the solid solution phase at 1200 °C. At the temperature below 1100 °C, the Sb-containing phase primarily changed from the solid solution phase to the Sb-silicate phase.

(2) As the temperature increased, the Sb_2O_3 content in the solid solution phase increased drastically from 7.52 wt% at 1100 °C to 17.36 wt% at 1200 °C. The Sb_2O_3 content in SiO_2 and High T.Fe phase demonstrated a stable tendency within the temperature range of 900 °C to 1200 °C, while the average contents were approximately 1.3 wt% and 3.3 wt%.

(3) The effects of Fe/SiO_2 and CaO/SiO_2 on the Sb_2O_3 content in the Sb-Fe-O- SiO_2 -CaO system were explored. The results demonstrated that lowering the values of CaO/SiO_2 and Fe/SiO_2 in the smelting process could effectively reduce Sb_2O_3 content in the slag. The verification experiment results implied that the antimony content in slag was 0.57 wt%, which was consistent with the above theoretical analysis.

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RASPODELA Sb U FAZAMA SiO₂ ZASISEĆENOG SISTEMA Sb-Fe-O-SiO₂-CaO U ATMOSFERI VAZDUHA

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Apstrakt

Raspedela Sb u fazama SiO₂ zasisećenog sistema Sb-Fe-O-SiO₂-CaO je prvi put utvrđena eksperimentom koji je izveden na visokim temperaturama i tehnikom brzog hlađenja, nakon čega je izvršena mikroanaliza elektronskom sondom (EPMA) u atmosferi vazduha ($P_{\text{tot}} = 1 \text{ atm}$, $P_{\text{O}_2} = 0.21 \text{ atm}$). Faze su kvantifikovane u temperaturnom opsegu od 900 °C do 1200 °C, a uticaj Fe/SiO₂ (maseni udeo) i CaO/SiO₂ (maseni udeo) na sadržaj Sb₂O₃ u Sb-Fe-O-SiO₂-CaO sistemu je ispitan na 1200 °C. Rezultati su pokazali da je faza koja je sadržala Sb primarno postojala u fazi čvrstog rastvora na 1200 °C. Sa porastom temperature sa 1100 °C na 1200 °C sadržaj Sb₂O₃ u fazi čvrstog rastvora se drastično povećao sa 7,52 wt% na 17,36 wt%. Smanjenje vrednosti CaO/SiO₂ i Fe/SiO₂ tokom procesa topljenja je efikasno smanjilo sadržaj Sb₂O₃ u šljaci. Provera rezultata eksperimenta je pokazala da je sadržaj antimona u šljaci iznosio 0,57 wt%, prinos sirovog antimona je bio niži od 4%, a stepen sirovog antimona je bio veći od 94 wt%, što ukazuje da smanjenje sadržaja antimona u šljaci može biti postignuto.

Ključne reči: Odnosi faza; Likvidus; Mikrostruktura; Pirometalurgija

