

Removal of Residual Element Antimony from Molten Steel by CaC₂-Containing Refining Slag

Zihong Chen ^{1,2,3}, Mengke Liu ^{1,2,*}, Guojun Ma ^{1,2,*}, Xiang Zhang ^{1,2}, Tianyu Du ^{1,2} and Zhengliang Xue ^{1,2}

¹ Key Laboratory for Ferrous Metallurgy and Resources Utilization of Ministry of Education, Wuhan University of Science and Technology, Wuhan 430081, China

² Hubei Provincial Key Laboratory of New Processes of Ironmaking and Steelmaking, Wuhan University of Science and Technology, Wuhan 430081, China

³ Shanghai Xinlanhai Automation Technology Co., Ltd., Shanghai 201203, China

* Correspondence: liumengke@wust.edu.cn (M.L.); gma@wust.edu.cn (G.M.)

Abstract: Increasing the use of steel scrap and enhancing its recycling utilization are important strategies for fostering the low-carbon and environmental-friendly growth of the iron and steel industry in China. However, the current steelmaking processes cannot efficiently remove the residual elements in the scrap, such as Cu, Sn, As, and Sb. As a result, the above elements are recycled and accumulate in the scrap, which will eventually have a negative impact on the properties of steel. Currently, there are few studies on Sb removal from molten steel. To remove the residual element Sb in molten steel, the CaO-SiO₂-Al₂O₃ refining slag system containing CaC₂ was used, and the effect of the CaC₂ content in the molten slag, slag quantity, smelting temperature, and initial Sb and C contents in molten steel on the Sb removal ratio in the steel was investigated, and the mechanism of Sb removal by the aforementioned refining slag system was discussed in order to provide some experimental and theoretical basis for industrialization practice. When the smelting time is 5~10 min, the removal ratio of Sb from molten steel is at its peak and can reach 45.8%. The “Sb-reversion” phenomenon will appear in the molten steel when the smelting period is progressively extended. In molten steel, CaC₂ will preferentially react with O and S, and as the smelting temperature decreases, the distribution ratio of Sb, L_{Sb} , improves. An increased initial Sb content in molten steel and slag quantity are beneficial to improving the removal ratio of Sb, but an increased initial C content in molten steel is detrimental to the progress of the Sb removal reaction. The removal reaction of Sb from molten steel by CaC₂ is a reversible reaction, and the diffusion of the products from the interface is the limiting factor of the overall reaction.

Keywords: steel scrap; calcium carbide; refining slag system; molten steel; removal; residual element; antimony



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1. Introduction

The solemn pledge of “carbon peak and carbon neutrality” has been made by China in an effort to combat climate change. The China State Council noted in the “Carbon Peak Action Plan Before 2030” that the iron and steel industry should promote structural optimization and clean energy substitution, as well as increase the recycling level of scrap steel resources, as it is the iron and steel industry that produces the most carbon emissions out of 31 categories of China’s manufacturing industry [1]. Scrap steel is a kind of green and low-carbon iron resource; its recycling can effectively reduce CO₂ and solid waste emissions, and reduce the consumption amount of iron ore and standard coal [2].

However, since the residual elements in scrap steel, such as Cu, Sn, As, and Sb, have a lower affinity for O than that of Fe, it is difficult to remove them using the traditional steel-smelting process. As a result, the content of residual elements in steel increases, which negatively affects the thermoplasticity, temper brittleness, and hot workability of steel [3–5].

If the content of residual elements in steel cannot be effectively reduced, it may lead to scrap steel becoming a “real steel scrap” [6]. With the increasing recycling capability of steel scrap, one of the major issues in the metallurgical industry is the problem caused by residual elements in steel [7].

Several studies have found effective techniques for removing residual elements from iron-based melts, including melt filtration, the electric field method, lead solvent method, calcium reaction method (including calcium flux or calcium alloy), sulfide flux method, and vacuum evaporation method [8–13]. Current research concentrates on the residual elements Cu and Sn since they are particularly prevalent in scrap steel. Sb can be employed as an alloying element in free-cutting steel, weathering steel, and non-oriented silicon steel [14–16]; however, it is an unfavorable element for most steel grades. Typically, the Sb contents in ordinary steel grades need to be controlled under 0.05 wt%, and for some special steel grades, the Sb content restriction is lower than 0.01 wt%. Nevertheless, the elimination of residual element Sb in molten steel is rarely studied. CaC_2 as a refining agent in the steelmaking process can decompose to Ca and C at steelmaking temperatures, and Ca could react with the residual elements in the molten steel. Moreover, the cost of CaC_2 treatment is cheaper than other calcium treatment methods. However, the reaction mechanism between CaC_2 and residual element Sb needs to be revealed.

This work investigated the removal of residual element Sb in molten steel by the CaO-SiO₂-Al₂O₃ refining slag system incorporating CaC_2 based on the practicality in industrial use and the effect and cost of Sb removal. The effects of the CaC_2 content, slag quantity, smelting temperature, and initial Sb and C content of molten steel on Sb removal were investigated, and the mechanism of Sb removal in the above refining slag system was discussed, aiming to provide the experimental and theoretical basis for industrial application.

2. Experiment

2.1. Experimental Procedure

The raw materials used in the experiment mainly include industrial pure iron (Fe), ferrosilicon (Fe-Si), ferroniobium (Fe-Nb), ferromolybdenum (Fe-Mo), nickel ingot (Ni), aluminum ingot (Al), antimony ingot (Sb), chromium ingot (Cr), electrolytic manganese (Mn), titanium sponge (Ti), and graphite (C). The main element contents in the above raw materials are shown in Table 1, and the chemical reagents, including CaO, SiO₂, Al₂O₃, and CaC_2 , are analytically pure.

Table 1. Content of main elements in various smelting raw materials (wt%).

Smelting Raw Materials	Fe	Fe-Si	Fe-Nb	Fe-Mo	Ni	Al	Sb	Cr	Mn	Ti	C
Contents	99.96	79 (Si)	66 (Nb)	59.5 (Mo)	99.9	99	99	98	99	99	99

The ingredients were first estimated based on the goal composition of low-carbon pipeline steel, which is displayed in Table 2. Then, the mixture of about 40 kg was melted by medium frequency vacuum induction furnace (ZGJW-0.05-100-2.5). The steel sample (sample number: S0) was taken after the raw material had been melted to ascertain the initial composition of the molten steel. After that, the furnace chamber was filled with Ar gas, and the induction furnace’s output power was adjusted. A specific quantity of pre-melted refining slag was added to the molten steel when it reached the desired temperature, and timing was then begun once the slag had melted. At intervals of 5, 10, 15, and 30 min, sampling was carried out, and the corresponding samples (sample number: S1–S4) were obtained. Table 3 displays the experimental parameters for each trial.

Table 2. Initial target compositions of molten low-carbon steel (wt%).

C	Si	Mn	P	S	Als	Ni	Nb	Ti	Cr	Mo	Sb	O
0.05/0.2	0.25	1.80	0.003	0.003	0.03	0.18	0.06	0.015	0.2	0.16	0.06/0.11	3.2×10^{-3}

Table 3. Experimental parameters of each trial.

No.	Temperature (K)	Slag Quantity (wt%)	Slag Composition (wt%)				Initial Sb Content (wt%)	Initial C Content (wt%)
			CaO	SiO ₂	Al ₂ O ₃	CaC ₂		
1	1873	5	30	6	24	40	0.06	0.05
2	1873	5	40	8	32	20	0.06	0.05
3	1873	5	45	9	36	10	0.06	0.05
4	1923	5	40	8	32	20	0.06	0.05
5	1823	5	40	8	32	20	0.06	0.05
6	1873	1	40	8	32	20	0.06	0.05
7	1873	3	40	8	32	20	0.06	0.05
8	1873	5	40	8	32	20	0.11	0.05
9	1873	5	40	8	32	20	0.11	0.2

2.2. Analysis Method

The C content and S content of steel samples were determined by an infrared carbon sulfur analyzer (Leco CS600), and the Sb content was detected by an inductively coupled plasma emission spectrometer (ThermoElemental IRIS Advantage Radial).

The removal ratio of Sb from molten steel was calculated by the following formula:

$$\eta_{\text{Sb}} = \frac{w_{[\text{Sb}]_{\text{S0}}} - w_{[\text{Sb}]_{\text{Si}}}}{w_{[\text{Sb}]_{\text{S0}}}} \times 100\% \quad (1)$$

where η_{Sb} is the removal ratio of molten steel, %; $w_{[\text{Sb}]_{\text{S0}}}$ is the initial Sb content of liquid steel, wt%; and $w_{[\text{Sb}]_{\text{Si}}}$ is the Sb content of the steel samples S_i ($i = 1, 2, 3,$ and 4), wt%.

3. Experimental Results

3.1. Effect of CaC₂ Content in Slag on Sb Removal

When the smelting temperature is 1873 K, the slag quantity is 5 wt%, the initial Sb content of the liquid steel is 0.06 wt%, the initial C content is 0.05 wt%, and the slag basicity ($w_{\text{CaO}}/w_{\text{SiO}_2}$) is 5; the effect of CaC₂ content on Sb removal from molten steel is shown in Figure 1. The Sb removal ratio of the molten steel reaches its peak at 5 to 10 min, as shown in Figure 1, and the difference between the maximum values are not statistically significant when the CaC₂ content of the slag is 20 wt% or 40 wt%. The overall smelting process exhibits a weak “Sb-reversion” phenomenon as smelting time increases, and at 30 min, 25% and 29.3% of Sb have been removed, respectively. However, the effectiveness of the Sb removal ratio is substantially lower than that with a 20 wt% or 40 wt% CaC₂ content of the slag, when the CaC₂ level of the slag is 10 wt%. It achieves its peak value after 15 min of reaction. After that, the Sb removal ratio does not change significantly with the extension of reaction time, and the Sb removal ratio is 9.7% at 30 min.

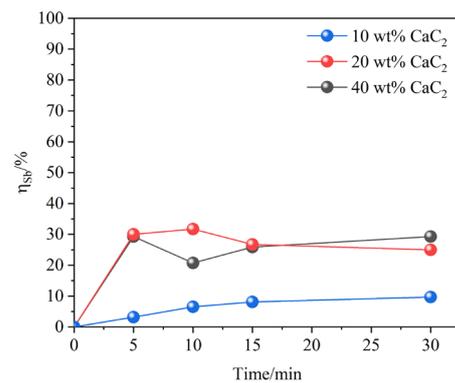


Figure 1. Effect of CaC₂ content in slag on Sb removal ratio at $T = 1873$ K, slag = 5 wt%, initial Sb = 0.06 wt%, initial C content = 0.05 wt%.

3.2. Effect of Slag Quantity on Sb Removal

Figure 2 depicts the fluctuation in Sb removal ratio with smelting time under various slag quantities of 1 wt%, 3 wt%, and 5 wt%. The other experimental conditions are smelting temperature of 1873 K, slag composition of 40 wt% CaO, 8 wt% SiO₂, 32 wt% Al₂O₃, 20 wt% CaC₂, initial Sb content of 0.06 wt%, and initial C content of 0.05 wt%. When the slag content is 1 wt%, as shown in Figure 2, the ratio of Sb removal from molten steel gradually rises as the smelting duration is extended. However, under the circumstances of 3 wt% and 5 wt% slag quantity, the Sb removal ratio achieves its maximum at 5 to 10 min and then quickly drops. Additionally, the Sb removal ratio under the condition of 5 wt% slag quantity is much higher than that of 3 wt% slag quantity at the same smelting time.

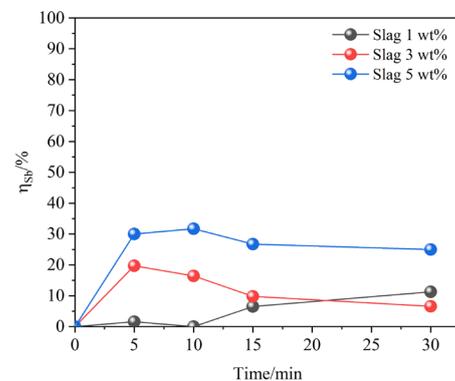


Figure 2. Effect of slag quantity on Sb removal ratio at $T = 1873$ K, initial Sb = 0.06 wt%, initial C content = 0.05 wt%, and initial CaC₂ = 20 wt%.

3.3. Influence of Smelting Temperature on Sb Removal

Figure 3 depicts the effect of the smelting temperature on the Sb removal ratio with the slag composition of 40% CaO, 8 wt% SiO₂, 32 wt% Al₂O₃, 20 wt% CaC₂; the slag quantity of 5 wt%; the initial Sb content of 0.06 wt%; and the initial C content of 0.05 wt%. As shown in Figure 3, for different smelting temperatures of 1823 K, 1873 K, and 1923 K, the Sb removal ratio reached its maximum within 5–10 min, which are 45.8%, 30%, and 40.3%, respectively. The Sb removal ratios at different smelting temperatures all gradually decreased after 10 min of smelting.

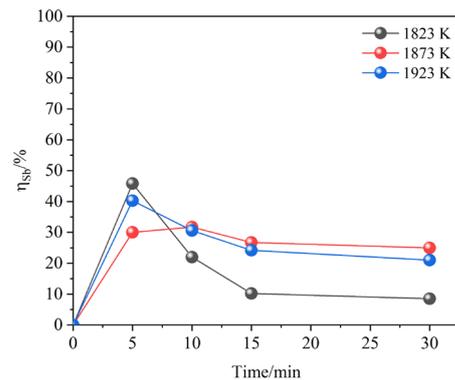


Figure 3. Effect of smelting temperature on Sb removal ratio at slag = 5 wt%, initial Sb = 0.06 wt%, initial C content = 0.05 wt%, and initial CaC₂ = 20 wt%.

3.4. Effect of Initial Sb Content in Molten Steel on Sb Removal

The variation of the Sb removal ratio with smelting time for different initial Sb contents is shown in Figure 4, where the smelting temperature is 1873 K, the slag quantity is 5 wt%, the slag fraction is 40 wt% CaO, 8 wt% SiO₂, 32 wt% Al₂O₃, 20 wt% CaC₂, and the initial C content of the molten steel is 0.05 wt%. As shown in Figure 4, at an initial Sb content of 0.11 wt% and a smelting time of 5 min, the Sb removal ratio of the molten steel reached 44.5%, which is significantly higher than the Sb removal rate at this time with the initial Sb content of 0.06 wt%. Thereafter, the Sb removal ratio decreases significantly, especially when the smelting time reaches 15 min, and this indicates that most of the Sb removed in the early stage has returned to the molten steel.

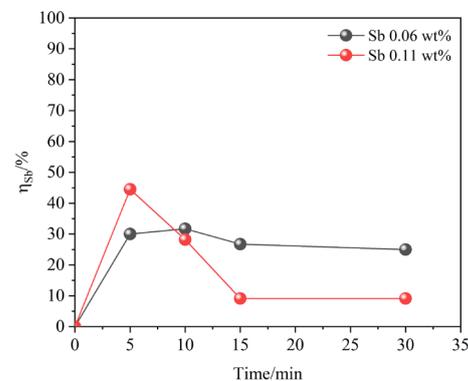


Figure 4. Effect of initial Sb content in molten steel on Sb removal ratio at $T = 1873$ K, slag = 5 wt%, initial C content = 0.05 wt%, and initial CaC₂ = 20 wt%.

3.5. Effect of Initial C Content in Molten Steel on Sb Removal

Under the conditions of an initial Sb content of 0.11 wt%, smelting temperature of 1873 K, slag quantity of 5 wt%, and slag composition of 40 wt% CaO, 8 wt% SiO₂, 32 wt% Al₂O₃, 20 wt% CaC₂, the influence law of different initial C contents of the steel on the desorption Sb ratio is shown in Figure 5. As can be observed from Figure 5, “Sb-reversion” occurs after 15 min of smelting time, and the Sb removal ratio is less than 10% at an initial C content of 0.2 wt% in the molten steel. Additionally, at the same smelting time in the range of 5 to 15 min, the first C content of 0.05 wt% is substantially larger than the initial C content of 0.2 wt%. According to the aforementioned findings, an increased initial C content in the molten steel is not favorable for Sb removal from molten steel, which is in line with those of Kazuo Kitamura et al. [17].

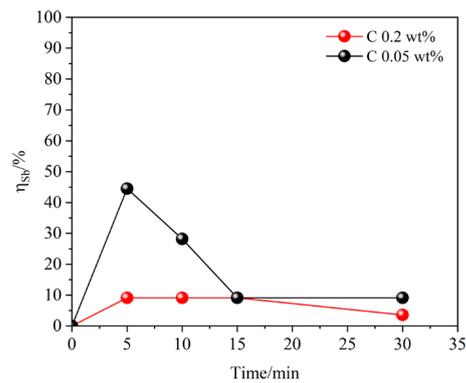
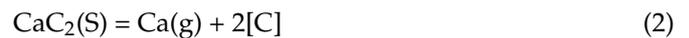


Figure 5. Effect of initial C content in molten steel on Sb removal ratio at $T = 1873$ K, slag = 5 wt%, initial Sb = 0.06 wt%, and initial $\text{CaC}_2 = 20$ wt%.

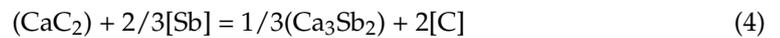
4. Analysis and Discussion

For the $\text{CaO-SiO}_2\text{-Al}_2\text{O}_3$ slag system containing CaC_2 , there is almost no reaction between CaC_2 and other components in the slag system under the present experimental conditions, and the use of this slag system for Sb removal relies mainly on the reaction of the component CaC_2 with Sb in the steel. At the same time, CaC_2 decomposes at the smelting temperature [17], and the reaction is shown in Equation (2).

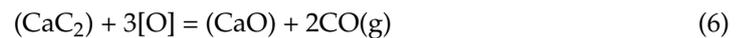


$$\Delta G^\theta (\text{J/mol}) = 61,410 - 32.3 \cdot T(\text{K}) \quad (3)$$

The solute components O, S, and Sb in molten steel will further react with the Ca bubbles created by the decomposition of CaC_2 . As a result, Equations (4) to (9) [18–21] show the primary reaction equations in the molten steel.



$$\Delta G^\theta (\text{J/mol}) = -152,000 + 64.1 \cdot T(\text{K}) \quad (5)$$



$$\Delta G^\theta (\text{J/mol}) = -646,478 - 173.88 \cdot T(\text{K}) \quad (7)$$



$$\Delta G^\theta (\text{J/mol}) = -314,929 + 26.60 \cdot T(\text{K}) \quad (9)$$

The predominant reaction order of CaC_2 with the components in the steel melt under the experimental conditions is [O], [S], and [Sb], as is evident from the aforementioned reaction equation. The removal effectiveness of O and S in the steel melt is much higher than that of Sb, according to the experiment results, which is in line with the thermodynamic analysis. Therefore, it is important to make sure that the amount of O and S in the steel melt is at a low level in order to improve the efficiency of CaC_2 on the steel melt for the Sb removal. Equation (4) shows that raising the CaC_2 content in the slag and the original Sb content in the molten steel can promote the de-Sb reaction, which is in line with the experimental findings displayed in Sections 3.1 and 3.4.

Furthermore, [C] and [Sb] take the 1 mass% dilute solution as the standard state. (Ca_3Sb_2) and (CaC_2) take the pure liquid as the standard state. Therefore, the equilibrium constant of Equation (4) is

$$K^\theta = \frac{f_{[\text{C}]}^2 \cdot w_{[\text{C}]}^2 \cdot \gamma_{(\text{Ca}_3\text{Sb}_2)}^{1/3} \cdot x_{(\text{Ca}_3\text{Sb}_2)}^{1/3}}{f_{[\text{Sb}]}^{2/3} \cdot w_{[\text{Sb}]}^{2/3} \cdot \alpha_{(\text{CaC}_2)}} \quad (10)$$

where: $f_{[C]}$ and $f_{[Sb]}$ are the activity coefficients of C and Sb in the molten steel, respectively; $w_{[C]}$ and $w_{[Sb]}$ are the mass fractions of [C] and [Sb] in the molten steel, individually, wt%; $\gamma_{(Ca_3Sb_2)}$ and $x_{(Ca_3Sb_2)}$ are the activity coefficients and molar fractions of Ca_3Sb_2 in the molten slag, separately; and $\alpha(CaC_2)$ is the activity of CaC_2 in the molten slag.

The following equation provides the Sb distribution factor as a consequence.

$$L_{Sb} = \frac{x_{(Ca_3Sb_2)}^{1/3}}{w_{[Sb]}^{2/3}} = K^\theta \cdot \frac{f_{[Sb]}^{2/3} \cdot \alpha(CaC_2)}{f_{[C]}^2 \cdot w_{[C]}^2 \cdot \gamma_{(Ca_3Sb_2)}^{1/3}} \quad (11)$$

According to Equations (5) and (11), the equilibrium constant K^θ is larger at lower temperatures, and thus L_{Sb} increases, favoring the Sb removal reaction. Furthermore, while increasing the C content in the molten steel increases the Sb activity coefficient to some extent [22], it is clear from Equation (11) that the influence of the above factors on the partition coefficient L_{Sb} of Sb does not dominate, and L_{Sb} decreases significantly with increasing the C content in the molten steel. When the initial C content of the molten steel is too high, it interferes with the decomposition of CaC_2 and thus limits the reaction, which is consistent with the experimental results shown in Section 3.5. Simultaneously, a large slag quantity reduces the activity coefficient $\gamma_{(Ca_3Sb_2)}$ of Ca_3Sb_2 in the slag, resulting in an increase in L_{Sb} and a more significant Sb removal effect.

According to the previous analysis, the Sb removal by CaC_2 is primarily a gas–liquid phase reaction between the decomposition of CaC_2 and the decomposition product, Ca bubbles, and the solute element Sb in molten steel. The kinetics of the reaction, according to the double film theory, consist primarily of the following processes, and the schematic diagram is shown in Figure 6:

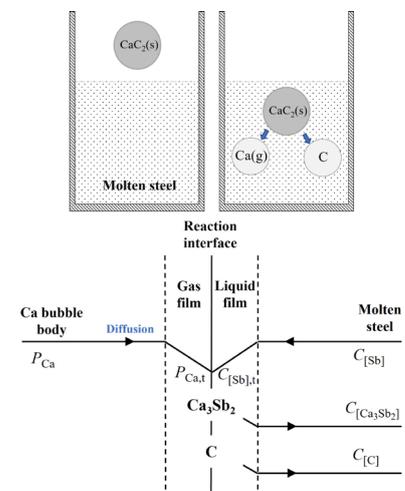


Figure 6. The schematic diagram of the kinetic steps for Sb removal reaction.

- (1) CaC_2 decomposition reaction: Ca bubbles and C are the products.
- (2) Reactant diffusion to the phase interface: Ca diffusion from the Ca bubble body across the gas film to the reaction interface, and Sb diffusion from the steel liquid across the liquid film to the reaction interface.
- (3) Interfacial chemical reaction: Ca and Sb react chemically at the reaction interface, producing Ca_3Sb_2 .
- (4) Product diffusion away from the phase interface: decomposition products C and Ca_3Sb_2 diffuse to the molten steel.

As previously stated, although lower temperatures are preferable for the Sb removal by CaC_2 in terms of thermodynamics, higher temperatures favor CaC_2 decomposition and accelerate the diffusion process, so the effect of temperature on Sb removal must be considered thoroughly. Figure 7 depicts the variation in the C content of the steel during the

reaction at different smelting temperatures. Combining Figures 3 and 7, it can be seen that within the smelting time range of 0–10 min, the C content in the melt increases significantly, and the Sb removal ratio corresponding to this stage also increases continuously. As smelting time increases, the trend of increasing C content in the melt slows down and is accompanied by the phenomenon of “Sb-reversion” which suggests to some extent that reaction Equation (4) is reversible and the reaction product Ca_3Sb_2 will be reduced by C, resulting in the generation of “Sb-retraction”. This is because the product diffused slowly from the phase interface, thus becoming the limiting factor of the overall reaction.

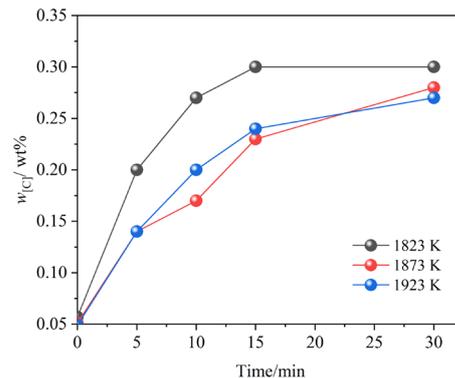


Figure 7. Variation of the C content in molten steel at different smelting temperatures at slag = 5 wt%, initial Sb = 0.06 wt%, initial C content = 0.05 wt%, and initial CaC_2 = 20 wt%.

Further analysis of the experimental results reveals that, under the same experimental conditions, the “Sb-reversion” phenomenon in the late stage is more significant when 5–10 min are presented, owing to the more products of Sb removal reaction generated at this time, and due to the limited diffusion, resulting in the Sb removal reaction in the reverse direction. According to the above analysis, in actual industrial production, a number of slagging, blowing argon stirring, and other operations are required to ensure that the final Sb removal ratio from molten steel is achieved.

5. Conclusions

Based on the experimental results in this work, we have drawn the following conclusions:

(1) When the $\text{CaO-SiO}_2\text{-Al}_2\text{O}_3$ slag system containing CaC_2 is used to treat the molten steel, the Sb removal ratio reaches its maximum value at 5–10 min of smelting time, and with the further extension of smelting time, the steel will show the phenomenon of “Sb-retraction”.

(2) The Sb removal ratio from molten steel can be up to 45.8% at a smelting temperature of 1823 K, a slag composition of 40 wt% CaO, 8 wt% SiO_2 , 32 wt% Al_2O_3 , 20 wt% CaC_2 , a slag quantity of 5 wt%, an initial Sb content of 0.06 wt%, and an initial C content of 0.05 wt%.

(3) CaC_2 reacts preferentially with O and S in the molten steel, and the Sb partition ratio L_{Sb} increases as the smelting temperature decreases from 1923 K to 1823 K. The increase in the initial Sb content (0.06–0.11 wt%) and slag quantity (1–5 wt%) of the molten steel is favorable to the increase in the maximum Sb removal ratio, but the increase in the initial C content (0.05–0.2 wt%) of the molten steel is not favorable to the Sb removal reaction.

(4) The reaction of CaC_2 to remove Sb from molten steel is reversible, and the limiting factor of the overall reaction is the diffusion of the products leaving the phase interface.

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