

## Boron removal in purifying metallurgical grade silicon by CaO–SiO<sub>2</sub> slag refining

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**Abstract:** Boron removal from metallurgical grade silicon (MG-Si) using a calcium silicate slag was studied. The results show that it is impossible basically to remove boron using a pure SiO<sub>2</sub> refining. The oxidizing ability of CaO–SiO<sub>2</sub> slag for boron removal was characterized by establishing the thermodynamic relationship between the distribution coefficient of boron ( $L_B$ ) and the activities of SiO<sub>2</sub> and CaO. The experimental results show that the distribution coefficient and the removal efficiency of boron are greatly improved with the increase of CaO proportion in the slag. The maximal value of  $L_B$  reaches 1.57 with a slag composition of 60%CaO–40%SiO<sub>2</sub> (mass fraction). The boron content in the refined silicon is reduced from  $18 \times 10^{-6}$  to  $1.8 \times 10^{-6}$  using slag refining at 1600 °C for 3 h with a CaO–SiO<sub>2</sub>/MG-Si ratio of 2.5, and the removal efficiency of boron reaches 90%.

**Key words:** metallurgical grade silicon; boron removal; thermodynamics; distribution coefficient; slag refining

### 1 Introduction

Renewable energy is the only way to substitute fossil fuel [1,2]. In recent years, solar cell has been paid close attention for its potential advantages. Traditionally, the off-spec silicon feeds from semiconductor industry produced by the Siemens process have been supplied to the photovoltaic industry [3,4]. The metallurgical route is an increasingly popular method in the range from purifying metallurgical grade silicon (MG-Si) to solar grade silicon (SoG-Si). However, boron removal from MG-Si is still a puzzle as a result of the low vapour pressure for boron [5] and the large segregation coefficient for boron to silicon [6]. Boron, which will accelerate the auger recombination and reduce the mobility ratio of minority carriers in silicon wafer [7,8] on account of the existence of the recombination centers such as Fe–B pairs, B–O metastable complexes and Cu-related extended defects [9], is a trouble maker to the

photoelectric conversion.

As a rule, the boron content in SoG-Si is required to be lower than  $0.5 \times 10^{-6}$ . To meet this requirement, a purification process for boron removal must be operated. These methods include gas blowing, thermal plasma and slag refining. Through blowing the reactive gases such as O<sub>2</sub>, H<sub>2</sub> and H<sub>2</sub>O into MG-Si melt, the gaseous boric species B<sub>x</sub>O<sub>y</sub> can be generated and volatilized. It is similar to the thermal plasma refining, which usually has preferable boron removal efficiency [10,11]. Slag refining employed in the steelmaking industry is also an efficient way to remove boron from MG-Si and these slags include CaO–SiO<sub>2</sub>, CaO–SiO<sub>2</sub>–CaF<sub>2</sub>, CaO–SiO<sub>2</sub>–Al<sub>2</sub>O<sub>3</sub>, CaO–SiO<sub>2</sub>–MgO and CaO–SiO<sub>2</sub>–Na<sub>2</sub>O [12,13]. KHATTAK and SCHMID [14] studied the boron removal using a CaO–SiO<sub>2</sub> slag refining and the boron content in MG-Si was reduced from  $18 \times 10^{-6}$  to  $1.0 \times 10^{-6}$ . JOHNSTON and BARATI [15] and LUO et al [16] found that the slag basicity interpreted as the concentration of free oxygen ions in slag had an

important effect on boron removal.

In the present work, the boron removal from MG-Si using CaO–SiO<sub>2</sub> slag was studied, and the role of CaO in slag was illustrated. The distribution of boron between slag and silicon ( $L_B$ ) was determined using different slag composition. Simultaneously, the oxidizing ability of CaO–SiO<sub>2</sub> slag to impurity boron was calculated and characterized. The boron removal was finally carried out with different ratios of slag to silicon and refining time.

## 2 Experimental

The MG-Si with a boron content of  $18 \times 10^{-6}$  was used in this work. It was smashed and ground to powders with sizes of 50–200  $\mu\text{m}$ . The MG-Si powder and the reagent grade chemicals of CaO and SiO<sub>2</sub> were mixed and loaded in a graphite crucible. The crucible was put into a quartz tube of intermediate frequency inductive furnace. Argon with 99.9% purity was blown into quartz tube for protection, and the temperature was determined by an infrared thermometer. The schematic diagram of experimental apparatus is shown in Fig. 1.

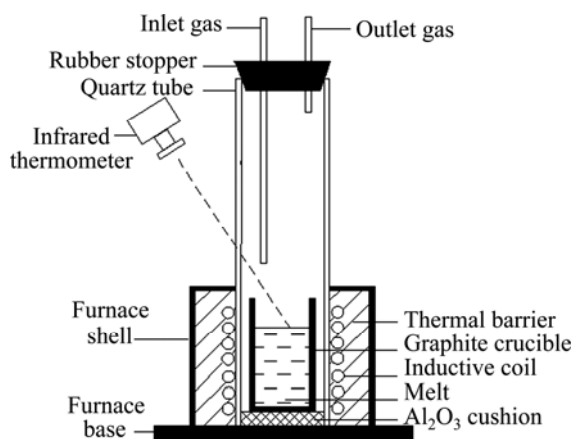


Fig. 1 Schematic diagram of apparatus for molten slag refining

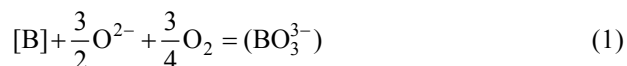
Boron removal using the pure SiO<sub>2</sub> and CaO–SiO<sub>2</sub> binary slag refining was carried out under different conditions of slag composition, mass ratios of slag to silicon and refining time, respectively. The boron contents in the refined slag and silicon samples were determined by inductively coupled plasma mass spectrometry (ICP-MS, Elan-5000A, USA).

## 3 Results and discussion

### 3.1 Boron removal using pure oxide SiO<sub>2</sub>

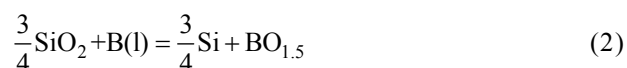
Boron removal from MG-Si using pure SiO<sub>2</sub> refining was carried out with different refining temperatures and mass ratios of SiO<sub>2</sub> versus MG-Si. TEIXEIRA and MORITA [17], JOHNSTON and

BARATI [18] described that boron in silicon (expressed as [B]) could be oxidized into boric oxide by free oxygen ion ( $\text{O}^{2-}$ ) and oxygen ( $\text{O}_2$ ). Then, the generated boric oxide would enter a basic slag phase based on calcium oxide in the form of negative ion  $\text{BO}_3^{3-}$ , and a calcium borate phase was finally generated via the ionic equation as follows:



The studies focused on the free oxygen ion released by the basic oxides CaO and oxygen decomposed by SiO<sub>2</sub>. In thermodynamics, the activity of free oxygen ion and the partial pressure of oxygen were crucial for boron removal. However, the oxygen partial pressure would be restricted by the decomposition of SiO<sub>2</sub>.

In addition, TEIXEIRA et al [19] detailed that boron in silicon was directly oxidized into  $\text{BO}_{1.5}$  by SiO<sub>2</sub> and it was expressed as:



It is found that this reaction would only take place at a higher temperature than 1961.8 °C according to the standard Gibbs free energy of formation, and it could be thought that the oxidation of boron by the pure SiO<sub>2</sub> was especially difficult. Experimentally, boron removal was done in the intermediate frequency furnace at the refining temperatures of 1500 and 1750 °C, respectively. The mass ratios of SiO<sub>2</sub> to MG-Si were set as 1:10, 2:10, 3:10 and 4:10. Figure 2 shows the refined sample using pure SiO<sub>2</sub> at 1500 °C.

It is seen from the longitudinal section of refined sample in Fig. 2(a) that SiO<sub>2</sub> distributes on the top of refined silicon and takes on the state of granule, which implies that SiO<sub>2</sub> is unmelted during the refining process. The cross section image of sample in Fig. 2(b) shows a same distribution for SiO<sub>2</sub> as the longitudinal section. The boron removal using SiO<sub>2</sub> with a refining time of 2 h and different mass ratios of SiO<sub>2</sub> to MG-Si at 1500 and 1750 °C, respectively, are shown in Fig. 3.

It is found that the boron content is reduced from  $18 \times 10^{-6}$  to  $16 \times 10^{-6}$  at 1500 °C and the removal efficiency of boron is only 11%. However, it is reduced to  $11.3 \times 10^{-6}$  at a refining temperature of 1750 °C and the removal efficiency reaches 37%. Though it is hardly effective for boron removal using pure SiO<sub>2</sub> refining, it is more helpful for a higher refining temperature. The result is also in accordance with the thermodynamic calculation. It is thought that the liquid SiO<sub>2</sub> may make the oxidation of boron in molt silicon more easily compared with the solid state one. As shown in the SiO<sub>2</sub>–B<sub>2</sub>O<sub>3</sub> binary phase diagram [20], the acidic B<sub>2</sub>O<sub>3</sub> generated in the interface

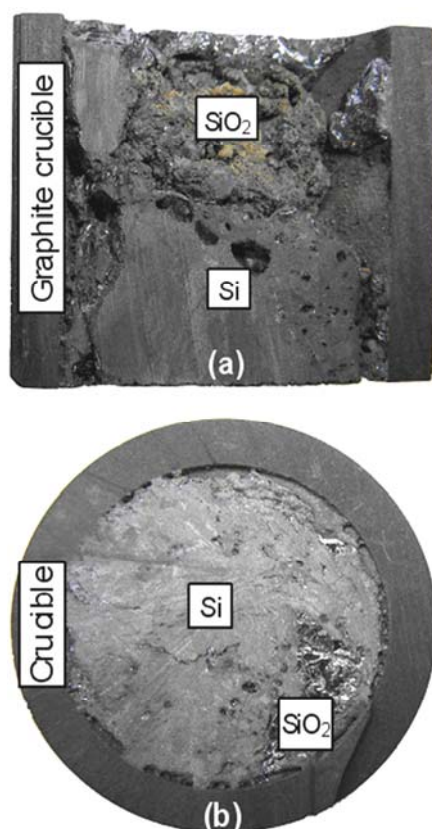


Fig. 2 Silicon sample refined by pure SiO<sub>2</sub>: (a) Longitudinal section; (b) Cross section

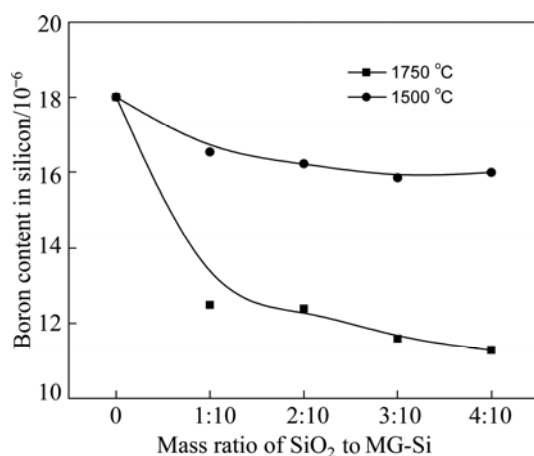
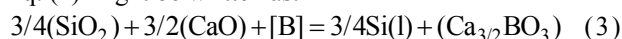


Fig. 3 Effects of mass ratio of SiO<sub>2</sub> to MG-Si and refining temperature on boron removal

of SiO<sub>2</sub> and MG-Si can not be easily dissolved into a same acidic slag. Therefore, the diffusion of B<sub>2</sub>O<sub>3</sub> towards slag phase is obstructed on account of the accumulation of B<sub>2</sub>O<sub>3</sub> in the interface of slag and silicon.

### 3.2 Boron removal using CaO–SiO<sub>2</sub> binary slag

The basic oxide CaO was added to SiO<sub>2</sub>, however, the oxidation of boron became possible. At the moment, Eq. (2) might be written as:



It is found that the theoretical initial temperature of Eq. (3) is greatly reduced compared with that of Eq. (2) according to the standard Gibbs free energy of formation. The molten silicon and slag are considered as a dilute solution. On condition that the standard state for the Henry's law is chosen, the equilibrium constant ( $K_p$ ) of Eq. (3) might be written as:

$$K_p = \frac{w(\text{Ca}_{3/2}\text{BO}_3) \cdot f_{\text{Ca}_{3/2}\text{BO}_3} \cdot a_{\text{Si(l)}}^{3/4}}{a_{\text{SiO}_2}^{3/4} \cdot a_{\text{CaO}}^{3/2} \cdot w[\text{B}] \cdot f_{[\text{B}]}} \quad (4)$$

where  $w(\text{Ca}_{3/2}\text{BO}_3)$  and  $w[\text{B}]$  are the mass fraction of calcium borate in slag and the mass fraction of boron in silicon, respectively;  $a$  and  $f$  are the activity and the activity coefficient, respectively. For the Henry's law, it is considered that  $f_{\text{Ca}_{3/2}\text{BO}_3} = f_{[\text{B}]} = 1$  and  $a_{\text{Si(l)}} = 1$ .  $w(\text{Ca}_{3/2}\text{BO}_3)$  can be substituted with the concentration of boron in slag ( $w(\text{B})$ ), and it can be expressed as:

$$w(\text{Ca}_{3/2}\text{BO}_3) = 11w(\text{B}) \quad (5)$$

The distribution coefficient of boron between slag and silicon ( $L_B$ ) is expressed as:

$$L_B = w(\text{B})/w[\text{B}] \quad (6)$$

Lastly, the distribution coefficient of boron might be written as:

$$\lg L_B = 3/4 \lg a_{\text{SiO}_2} + 3/2 \lg a_{\text{CaO}} + \lg K_p - 1.04 \quad (7)$$

The activities of SiO<sub>2</sub> and CaO in CaO–SiO<sub>2</sub> binary system ( $0.4 < x_{\text{SiO}_2} < 0.7$ ) were determined by ZOU et al [21], as shown in Fig. 4.

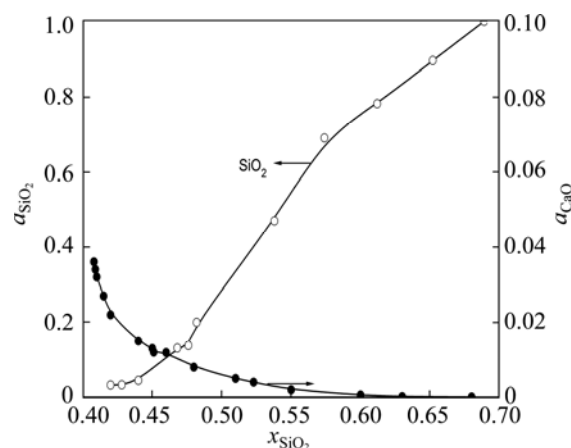
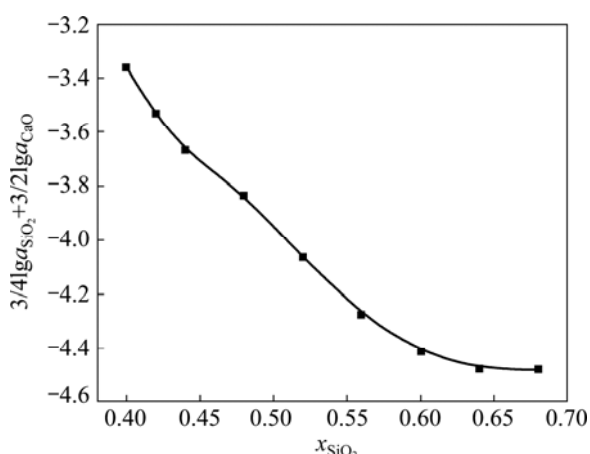


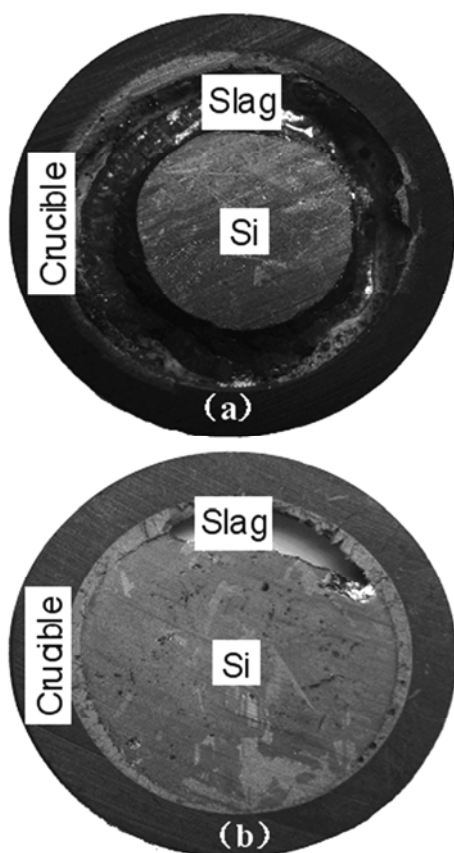
Fig. 4 Activities of CaO and SiO<sub>2</sub> in CaO–SiO<sub>2</sub> binary system at 1600 °C [21]

It is found that the activity of SiO<sub>2</sub> increases with the rise of its proportion in CaO–SiO<sub>2</sub> slag. It is completely opposite to CaO for this case. As shown in Fig. 5, the thermodynamic relationship displays that the value of item  $(3/4 \lg a_{\text{SiO}_2} + 3/2 \lg a_{\text{CaO}})$  in Eq. (7) decreases gradually when the mole fraction of SiO<sub>2</sub> in slag is larger than 0.4.



**Fig. 5** Relationship between item  $(3/4 \lg a_{\text{SiO}_2} + 3/2 \lg a_{\text{CaO}})$  and mole fraction of  $\text{SiO}_2$  in slag

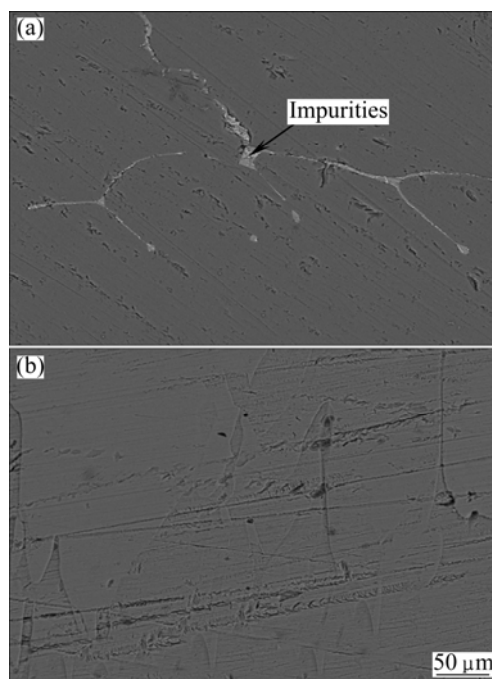
Figure 6 shows the refined silicon sample using  $\text{CaO-SiO}_2$  binary slag at 1600 °C. It is seen that the  $\text{CaO-SiO}_2$  slag is melted completely and separated out from silicon melt. After refining, the refining slags separated are distributed on the top of silicon (Fig. 6(a)) and between silicon and graphite crucible (Fig. 6(b)).



**Fig. 6** Refined silicon sample in crucible using  $\text{CaO-SiO}_2$  slag: (a) Top; (b) Middle

The SEM images of silicon sample before and after refining are shown in Fig. 7. It is found that the amounts

of impurities in MG-Si are greatly reduced after refining using  $\text{CaO-SiO}_2$  slag. Unexpectedly, the content of calcium in silicon increased from 0.015% to 0.042% in the study by DIETL [22]. Obviously, the calcium derived from refining slag should not be ignored using  $\text{CaO-SiO}_2$  system. However, it can be removed using hydrometallurgical treatment or vacuum volatilization [23,24].



**Fig. 7** SEM images of refined silicon using  $\text{CaO-SiO}_2$  slag before (a) and after (b) refining

### 3.3 Effect of technological conditions on boron removal

In order to display the oxidizing ability of  $\text{CaO-SiO}_2$  slag for boron removal, the distribution of boron between  $\text{CaO-SiO}_2$  slag and silicon was studied with different slag compositions. The mass ratios of slag to silicon and the refining time are set as 1:1 and 3 h, respectively, at 1600 °C. The distribution coefficient of boron was calculated according to Eq. (7), as shown in Fig. 8.

It is found that the distribution coefficient of boron increases with the increase of  $\text{CaO}$  proportion in slag. It reaches 1.57 when the mass ratio of  $\text{CaO/SiO}_2$  is 1.5, the slag composition is 60% $\text{CaO}$ –40% $\text{SiO}_2$ . Unexpectedly, the distribution coefficient of boron reduces when  $\text{CaO/SiO}_2$  composition is exceeded to 1.5, which is in accordance with the studies by SUZUKI et al [25]. It is presumed that the increase of  $\text{CaO/SiO}_2$  composition results in the decrease of  $\text{SiO}_2$  activity, which is crucial to the oxidizing ability of  $\text{CaO-SiO}_2$  slag. Therefore, the distribution coefficient of boron declines as well with the continuous increase of  $\text{CaO}$  in slag.

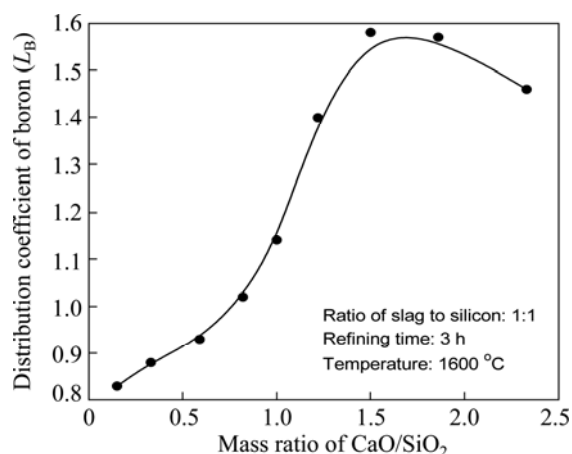


Fig. 8 Distribution coefficient of boron ( $L_B$ ) with different CaO/SiO<sub>2</sub> compositions

Figure 9 shows the effects of slag/silicon mass ratio on boron removal. The slag composed of 60%CaO–40%SiO<sub>2</sub> is used to refine MG-Si. The refining time and temperature are set as 3 h and 1600 °C, respectively. It is found that the removal efficiency of boron increases with the increase of ratios for slag to silicon. The boron content in silicon is reduced from  $18 \times 10^{-6}$  to  $1.8 \times 10^{-6}$  and the removal efficiency reaches 90% when the mass ratio is 2.5. However, the trend is unobvious when the ratio is larger than 2.5. It is thought that the superfluous CaO–SiO<sub>2</sub> slag is non-effective for boron removal in refining process. The optimal ratio of slag to silicon for boron removal is about 2.5 using a 60%CaO–40%SiO<sub>2</sub> slag refining at 1600 °C for 3 h.

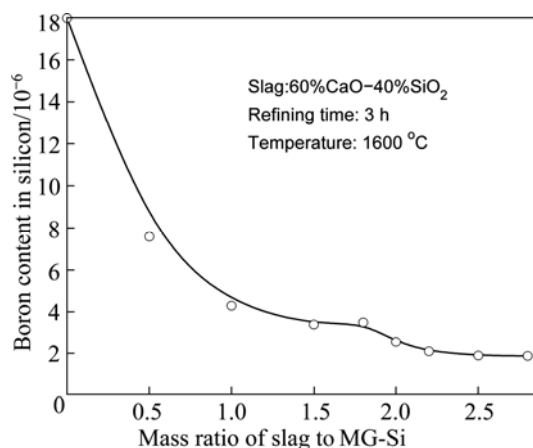


Fig. 9 Effects of mass ratio of CaO–SiO<sub>2</sub> slag to MG-Si on boron removal

Figure 10 shows the effects of refining time on boron removal from MG-Si. The 60%CaO–40%SiO<sub>2</sub> slag was used and the mass ratio of slag to silicon and the refining temperature were set as 2.5 and 1600 °C, respectively. It is found that the boron content in refined silicon decreases rapidly with the increase of refining

time. It is reduced to  $2.2 \times 10^{-6}$  for 2.5 h and  $1.8 \times 10^{-6}$  for 3 h, respectively. However, the decreasing trend for boron removal becomes unobvious after longer refining time. It is thought that the driving force of boron diffusion towards slag phase decreases and the equilibrium for boron in slag and silicon is reached. Therefore, it is optimal for boron removal at 1600 °C for 3 h in the intermediate frequency inductive furnace using 60%CaO–40%SiO<sub>2</sub> slag refining.

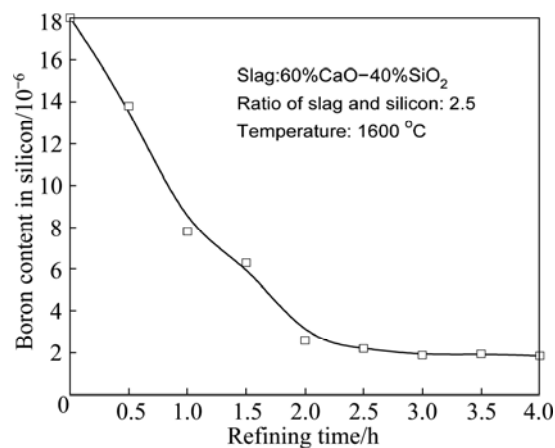


Fig. 10 Effects of refining time on boron removal

## 4 Conclusions

1) Boron removal from metallurgical grade silicon using a CaO–SiO<sub>2</sub> slag refining was studied. It is non-effective basically to remove boron using a pure SiO<sub>2</sub> refining. However, the removal efficiency of boron is greatly improved with the addition of basic CaO in slag, which is consistent with the thermodynamic calculation.

2) The oxidizing ability of CaO–SiO<sub>2</sub> slag for boron removal was calculated and characterized. The distribution coefficient of boron between slag and silicon increases when the mole fraction of CaO in slag rises.

3) The mass fraction of boron in MG-Si can be reduced from  $18 \times 10^{-6}$  to  $1.8 \times 10^{-6}$  using 60%CaO–40%SiO<sub>2</sub> slag refining for 3 h when the mass ratio of slag to MG-Si is set as 2.5.

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## CaO–SiO<sub>2</sub> 熔渣精炼去除冶金级硅中杂质硼

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**摘 要:** 利用 CaO–SiO<sub>2</sub> 熔渣去除冶金级硅(MG-Si)中的杂质硼。热力学分析和实验结果表明: 纯 SiO<sub>2</sub> 基本上不能去除冶金级硅中的杂质硼。通过建立硼的分配系数与熔渣中 SiO<sub>2</sub> 和 CaO 活度之间的关系, 从热力学上对 CaO–SiO<sub>2</sub> 熔渣的除硼能力进行表征。结果表明: 随着渣中 CaO 配比的升高, 硼的分配系数和去除效率大大提高。当熔渣组成为 60%CaO–40%SiO<sub>2</sub>(质量分数)时, 硼的分配系数达到最大值 1.57。当渣硅比为 2.5, 精炼温度为 1600 °C 以及精炼时间为 3 h 时, 利用 60%CaO–40%SiO<sub>2</sub> 熔渣可以将冶金级硅中的硼含量从  $18 \times 10^{-6}$  降低至  $1.8 \times 10^{-6}$ , 去除效率达到 90%。

**关键词:** 冶金级硅; 除硼; 热力学; 分配系数; 熔渣精炼

(Edited by Chao WANG)