Boron removal through multistage refining treatment using $CaO-SiO_2-Al_2O_3$ slag

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ABSTRACT

Multistage slag treatment was carried out to investigate boron removal from metallurgical grade silicon. Boron is known to be one of the most difficult elements to remove from silicon. Depending on boron initial concentration, boron can be removed from silicon into certain level; most previous studies have attained final 50-100 ppmw boron in the silicon using slag treatment. The term 'multistage' refers to a process where the resulting silicon from the initial slag treatment is further refined multiple times using a new slag. In this study, an initial slag composition of $45CaO-45SiO_2-10Al_2O_3$ (wt. pct.) was utilised. Using this technique, boron level in the silicon was expected to be gradually lowered because the new slag will always have more capacity to reach the equilibrium distribution coefficient ($L_B = (ppmwB)_{slag} / (ppmwB)_{Si}$). The current study was carried out through combined thermodynamic calculations (using FactSage thermochemical package) and high-temperature experimentation. The thermodynamic calculations were carried out for reactions at 1500, 1550, and 1600 ºC for three different scenarios: (1) single stage refining with varied slag to Si ratio; (2) multistage refining with slag to Si ratio of 1:1; and (3) multistage refining with slag to Si ratio of 2:1. The multistage experimental work was carried out at 1550 ºC under inert gas atmosphere each time for 2 hours, with slag to Si ratio of 2:1. The simulation results indicate that the boron distribution coefficient stay essentially constant with varying slag to Si ratio and temperature, around 1.8 to 2, and the experimental results measured L_B in the same range, between 1.5 and 2.5. By harnessing this feature, the simulation showed that boron can be removed by approximately 78% in each stage. The experimental result confirmed that the boron concentration in the silicon was reduced by a total of 97.8% from its initial value using four stages of slag treatment, from initial 270 ppmw down to 6 ppmw.

INTRODUCTION

Boron is one of the most difficult elements to remove from silicon (Chen *et al.*, 2019; Hou *et al.*, 2019; White, 2013). The element has a high segregation coefficient, meaning that it tends to concentrate in the solid phase during solidification. It also has a low vapor pressure, making it difficult to remove by evaporation. In addition, boron is chemically stable and does not form volatile compounds with common refining agents such as chlorine and oxygen (Betharia, 2014).

The difficulty of boron removal is a major challenge for the production of high-purity silicon by the semiconductor industry. Boron is used as a dopant to control the electrical conductivity of silicon (Betekbaev et al., 2016; Hwang et al., 2014), but even small distortion in boron concentration in the silicon wafer have a significant impact on the performance of the semiconductor device. Metallurgical grade silicon (MG-Si) has the boron concentration of approximately 80 ppmw (Yang, 2019). Whereas, solar grade silicon (SOG-Si), the raw materials for silicon PV cell production, should have boron concentration below 0.38 ppmw (Karabanov *et al.*, 2019).

There are a number of methods that have been developed for boron removal from silicon, specifically slag treatment. In the slag treatment processes, the extent of boron removal relies on the thermodynamic equilibrium between slag and silicon phase, which is represented as the boron distribution coefficient (L_B) (White, 2020; 2013). Boron distribution coefficient is affected by the basicity and composition of the slag which is reported to be as high as 2.1 for basic slag (40CaO-40SiO_2-20K_2CO_3, wt.pct.) (Yang, 2019). The L_B can be described as:

$$L_B = \frac{(B)}{[B]} \quad (1)$$

where (B) and [B] are the boron concentrations in the slag and silicon phase in ppmw, respectively.

There are several studies of boron removal from silicon using slag treatment with majority of the results showing that the refined silicon still has considerably high concentration of boron. The summary of the previous relevant studies is given in Table 1. Krystad *et al.* (2012) refined silicon with the initial boron concentrations of 260–280 ppmw at 1600 ºC using SiO_2-CaO and SiO_2-CaO-MgO slags. The results show that the boron concentration in the silicon started to stabilize after 4000 seconds at 50–80 ppmw. Nishimoto et al. (2012) studied boron removal from silicon (318 ppmw) using 55CaO-45SiO_2 (wt.pct.) at 1550 ºC and slag to silicon ratio of 1.25–2.5 and found that the boron concentration stabilized at 85–110 ppmw after approximately 4000 seconds. Islam and Rhamdhani (2018) studied similar process with various slag compositions (CaO-SiO_2 with 0; 9.59; and 15.9 wt.pct Al_2O_3), various slag to silicon ratios (1.5 to 2.5, w/w), and temperatures (1500–1600 ºC) and found similar final boron concentrations in the silicon at 110–120 ppmw from the initially 370 ppmw boron in the silicon.

TABLE 1 - Summary of initial and final boron concentrations in the silicon during slag treatment under various working conditions. *Approximated value by visual inspection.

Author	Slag system, wt.pct.	Temperature, ºC	Slag to Si ratio, w/w	Initial [B], ppmw	Final [B], ppmw
(Krystad *et al.*, 2012)	50CaO-50SiO_2	1600	1	260*	75*
	50CaO-50SiO_2	1600	2	260*	49*
	40CaO-40SiO_2-20MgO	1600	2	260*	50*
	35CaO-65SiO_2	1650	1	280*	80*
(Nishimoto *et al.*, 2012)	55CaO-45SiO_2	1550	1.25	318	85.1
	55CaO-45SiO_2	1550	1.33	318	118
	55CaO-45SiO_2	1550	2.5	318	100
(Islam & Rhamdhani, 2018)	49CaO-41SiO_2-9.6Al_2O_3	1500	2.5	370	130*
	49CaO-41SiO_2-9.6Al_2O_3	1550	2.5	370	120*
	49CaO-41SiO_2-9.6Al_2O_3	1600	2.5	370	100*
	46CaO-38SiO_2-15.9Al_2O_3	1500	2.5	370	110*
	46CaO-38SiO_2-15.9Al_2O_3	1550	2.5	370	120*
	46CaO-38SiO_2-15.9Al_2O_3	1600	2.5	370	125*
(Wu *et al.*, 2016)	37.5CaO-37.5SiO_2-25K_2CO_3	1550	1	22	2.6
	40CaO-40SiO_2-20K_2CO_3	1550	1	22	1.8
	42.5CaO-42.5SiO_2-15K_2CO_3	1550	1	22	2.7
	45CaO-45SiO_2-10K_2CO_3	1550	1	22	3.8
	47.5CaO-47.5SiO_2-5K_2CO_3	1550	1	22	4.1
(Fang *et al.*, 2014)	67Na_2O-33SiO_2	1700	0.5	10.6	1.8*
	67Na_2O-33SiO_2	1700	1	10.6	0.62*
	67Na_2O-33SiO_2	1700	1.5	10.6	0.4*
	67Na_2O-33SiO_2	1700	2	10.6	0.38*
	67Na_2O-33SiO_2	1700	2.5	10.6	0.36*

A work by Wu *et al.* (2019) stated that the efficiency of boron removal using slag treatment can reach 90 %, with a final boron concentration in the silicon of 1.8 ppmw using 40CaO-40SiO_2-20K_2CO_3 (wt.pct.) slag at 1550 °C for 3 hours. However, the initial boron concentration in this study was only 18 ppmw (Wu *et al.*, 2016). Another method to effectively lower boron concentration has been shown to occur by combining the slag treatment method with Ar-H_2-O_2 gas blowing. The result shows that boron removal reached 96.6% at 0.5 ppmw. However, the initial boron concentrations in this study were very low at 2–3 ppmw (Wu *et al.*, 2017).

Multiple slag treatment method was attempted by Fang *et al.* (2014) to remove boron from silicon, using Na_2O-SiO_2 slag at 1550–1700 ºC. The study shows that multiple slag operation consistently reduced boron concentration in the silicon to very low levels, at around 0.2 ppmw. However, this study also used a low initial concentration of boron, at 10.6 ppmw. In this study, an effort to decrease the boron concentration in the silicon from considerably high concentrations (above 100 ppmw) to very low concentrations (around 1-10 ppmw), with the removal rate >90 %, was done using multiple stages of slag treatment. The method was studied through combined thermodynamic calculation using FactSage and experimental work.

METHODOLOGY

Thermodynamics assessment

Thermodynamic assessment was carried out using FactSage® 8.2 with the Equilib module. The module calculates the equilibrium state of a system in a specific working condition, such as temperature and pressure based on chosen database(s) by utilizing Gibbs energy minimisation. The built-in databases used in this study are: (1) FactPS, for pure substances, (2) FTOxid, for oxides, and (3) FSupsi, for silicon alloy.

The boron concentration in the Si alloy was at 300 ppm and the slag component compositions were 0.45 wt. pct. CaO, 0.45 wt. pct. SiO_2, and 0.1 wt. pct. Al_2O_3. The resulting melts were saved as new mixtures using ‘Save Stream’ feature in the FactSage. This feature was also used to save the silicon melt in each stage of the multi-stage slag treatment. In this current study, only solid and liquid states are included in the modelling. Therefore, no vaporisation (either from silicon or boron) occurred in the simulation results.

Raw materials preparation

The following raw materials for slag making were supplied by Sigma Aldrich (Merck KGaA, Darmstadt, Germany): calcium oxide (CaO) 99 wt.pct., silicon dioxide (SiO_2) 99.5 wt.pct., aluminium oxide (Al_2O_3) 99.5 wt.pct. Master slags were prepared by melting mixed oxides with appropriate composition (45CaO-45SiO_2-10Al_2O_3, wt.pct.) in an SX2-2-17TP muffle furnace. The oxides were previously mixed in a ball mill using cylinder container and ceramic balls; rotated at 60 rpm for 60 minutes. After mixing, the mixture was placed in a Pt crucible (of approximately 30 g each run) and melted in a muffle furnace at 1550 °C for 30 minutes before casting on a steel mould. The casting process is shown in Figure 1.

The master silicon alloy materials were made from 99.99 wt.pct. silicon from Sigma Aldrich and crystalline boron powder 99 wt.pct. from Alfa Aesar (Thermo Fisher Scientific Inc. Australia Pty Ltd). The method to make the master silicon alloy (containing 300 ppmw boron) in presented in the previous work (Islam & Rhamdhani, 2018). Both master slag and master silicon were analysed using ICP-AES to confirm the compositions, as shown in Table 3.

FIG 1 - Slag casting process.

Slag treatment experiment

The furnace configuration is shown in Figure 2. The sample included initially 7.2 g of Si-B master alloy and 14.3 g of master slag in an alumina crucible. The crucible was placed inside a sacrificial crucible that was attached to alumina pedestal. Initially, the sample was placed in the cold zone inside the vertical tube furnace and then, the furnace was sealed. Argon gas with a flow rate of 200 ml/min was flushed into the furnace for 15 minutes to ensure an inert atmosphere. Gas bubbling was regularly checked to ensure the furnace was completely sealed. After 15 minutes of stabilising the gas atmosphere, the pedestal-crucible setup was inserted into the hot zone. It took approximately 1 minute to insert the pedestal into the hot zone and re-seal the system gas tight. The samples were reacted for 120 minutes under Argon gas after which they were lowered to the cold zone, for sample quenching, to stop the reactions. In this process, it took approximately 20 to 30 seconds to move the pedestal to the cold zone.

After approximately 60 minutes, the bottom flange and the sample were removed from the furnace. The sample was crushed, and a small amount (approximately 1 g) of the silicon phase was taken to analyse by inductively coupled plasma atomic emission spectroscopy (ICP-AES). It is important to note that ICP-AES is a destructive analysis, which means that the analysed sample cannot be used again in the next batch. In addition, a small portion of silicon was expected to be vaporized during the slag treatment (Fang *et al.*, 2014). Therefore, in the second batch, approximately 5 g of silicon was reacted with 10 g of new slag with the same composition (the slag to Si ratio was kept at 2:1, w/w). The approach is depicted in Figure 3 and the multi-stage slag treatment was carried in 4 stages in this study. A complete record of measured weights after each stage is provided in Table 2.

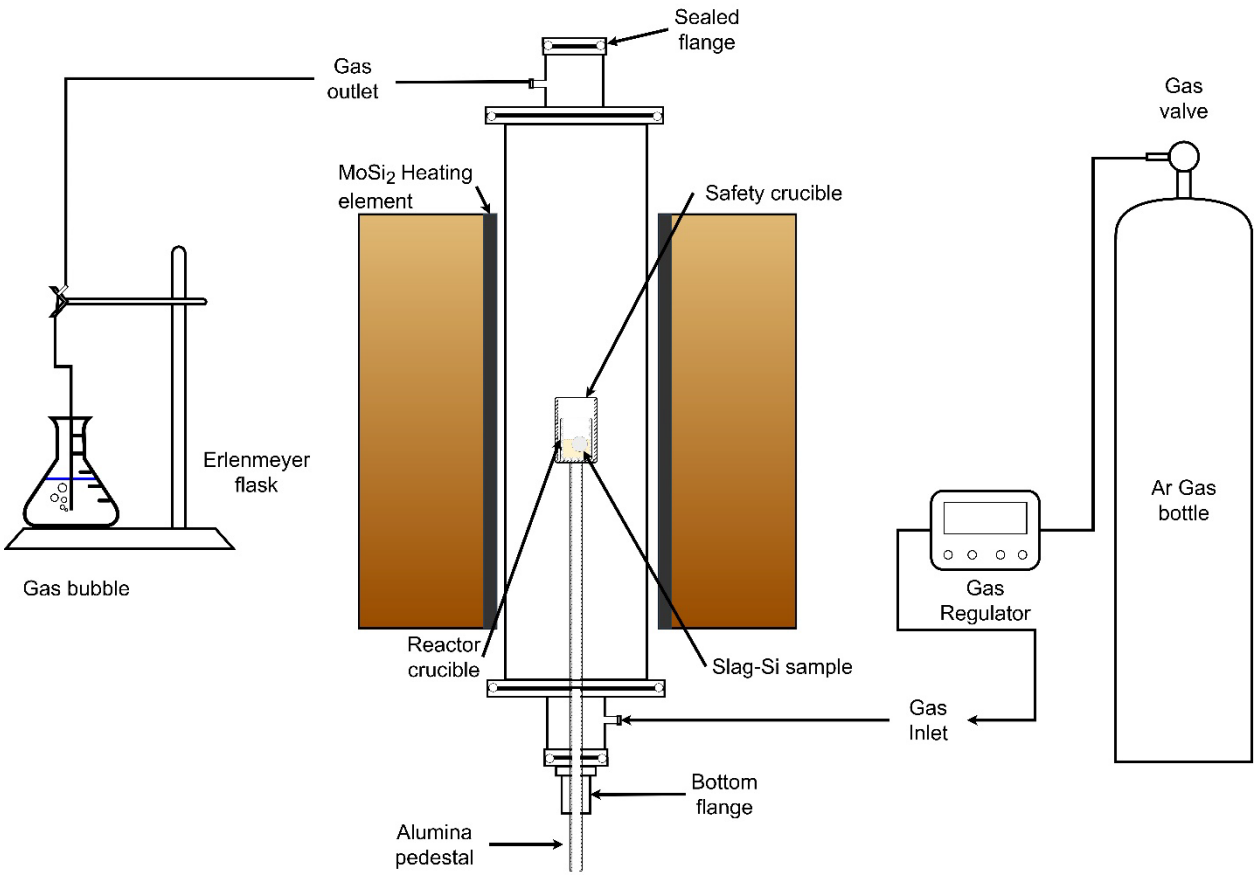

FIG 2 - Schematic diagram of the vertical tube furnace setup for slag treatment experiment.

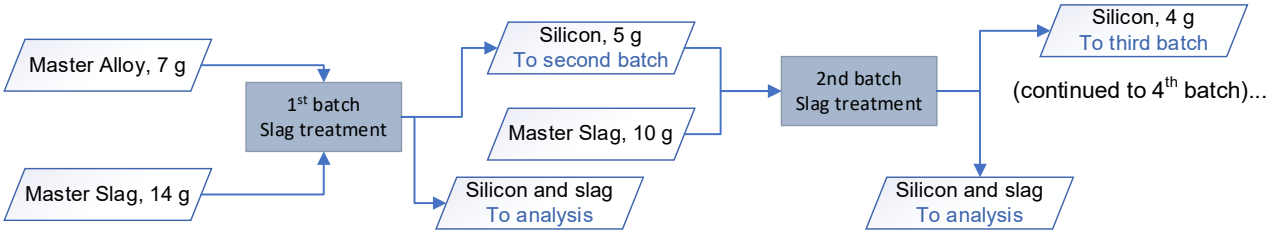

FIG 3 - Flow diagram for multi-stage slag treatment.

TABLE 2 - Weight measurements of silicon and slag after each stage.

Stage	Silicon, g	Slag, g
1	7.1667	14.3285
2	5.2020	10.4815
3	3.0813	6.1589
4	1.2851	2.6200

Analysis

Master silicon, master slag, and the slag from 1st to 4th stage of the experiment were crushed, and a fraction of the sample was taken for ICP-AES analyses conducted at Spectrometer Services PTY. LTD., Melbourne, Australia. Analysis preparation such as sample comminution, dilution, and standardisation were carried out at the company.

The multi-stage slag treatment samples were additionally analysed by laser ablation inductively coupled plasma spectrometer (LA-ICP-MS) at the Research School of Earth Sciences, Australian National University. Samples were mounted in epoxy resin and prepared by traditional wet metallographic techniques for microanalyses. The details on the equipment, analysis procedure and settings can be found in the other study (Avarmaa *et al.*, 2024).

RESULT

Thermodynamic calculations

The thermodynamic calculations in this study are divided into two scenarios: (1) the single-stage process and (2) multi-stage process. Figure 4 shows the boron distribution coefficient (L_B) of a single-stage slag treatment at temperatures from 1500 to 1600 ºC and slag to Si ratio from 1 to 20 (w/w). The line plot shows the boron distribution coefficient ranged between 1.84 to 1.96 for the observed temperatures. Slag to Si ratio had only marginal influence on the L_B and temperature influence was rather small as well.

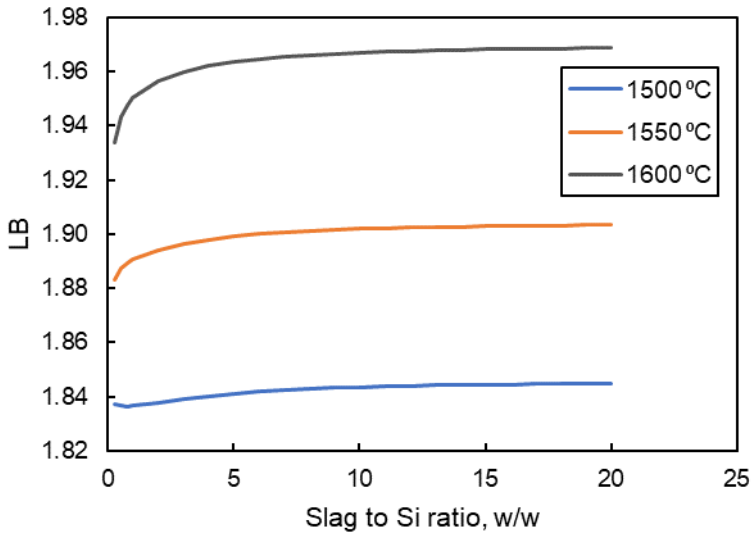

FIG 4 - Boron distribution coefficient of single-stage slag treatment at various temperatures and slag to Si ratios.

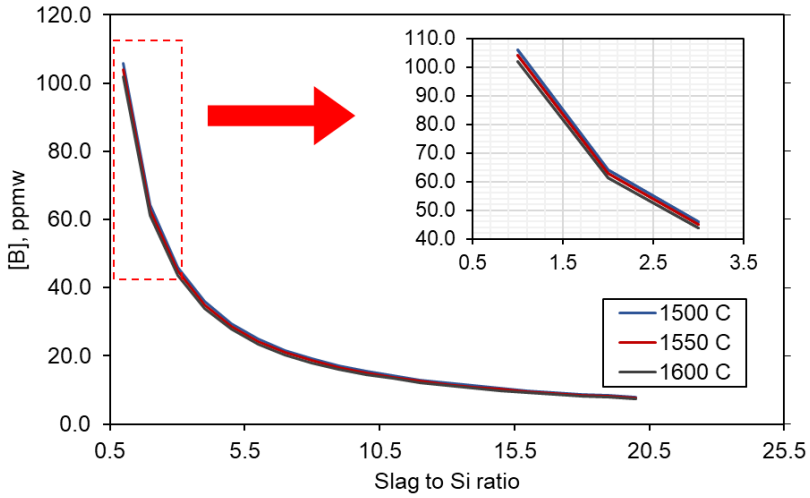

FIG 5 - Boron concentration in silicon after single-stage slag treatment at various slag to Si ratios at 1500 – 1600 ºC.

In terms of boron concentration in the silicon, [B], the result shows that it does not vary significantly between the different temperatures, although higher temperature generally yielded lower [B]. In addition, the result shows that the boron concentration decreased less significantly beyond slag to Si ratio of 5 (w/w). Boron concentration reached approximately 10 ppmw at slag to Si ratio of 20 (w/w). Boron concentration in the silicon for single-stage slag treatment at various temperatures and slag to Si ratios is presented in Figure 5.

The multi-stage simulation was carried out using two different slag to Si ratios, *i.e.* 2 and 1 (w/w) at 1550 ºC. The results of the boron concentration for each different scenario are presented in Figure 6. The Y-axis in this figure represents boron concentration in the silicon as ppmw and the X-axis represents the amount of slag consumed (g) for each gram of silicon, in order to compare effectiveness of a single and multi-stage slag treatments. The results show that the multi-stage process can significantly lower the boron concentration in the silicon compared to the single-stage slag treatment. For slag to Si ratio of 2 (w/w), 4 stages of slag treatment can already yield boron concentration in the silicon of 0.63 ppmw, or approximately 78 % reduction in each stage. At the 4th stage, the slag to Si consumed is equal to 8 (w/w) in the single-stage process. However, in the single-batch process, boron concentration in the silicon was at 19.06 ppmw, which is very high compared to the multistage result.

For slag to Si ratio of 1 (w/w), similar boron concentration was reached after 6 stages, or at 6 slag to Si consumed (w/w). Although one can achieve better boron removal using smaller slag to Si ratio in the multistage slag treatment, this scenario might have some issues in the applicability as numerous stages of slag treatment is also not favourable and a lot of slag would be produced. However, the result of this simulation generally demonstrates that multiple stage slag treatment is more effective to lower boron concentrations than a single-stage slag treatment.

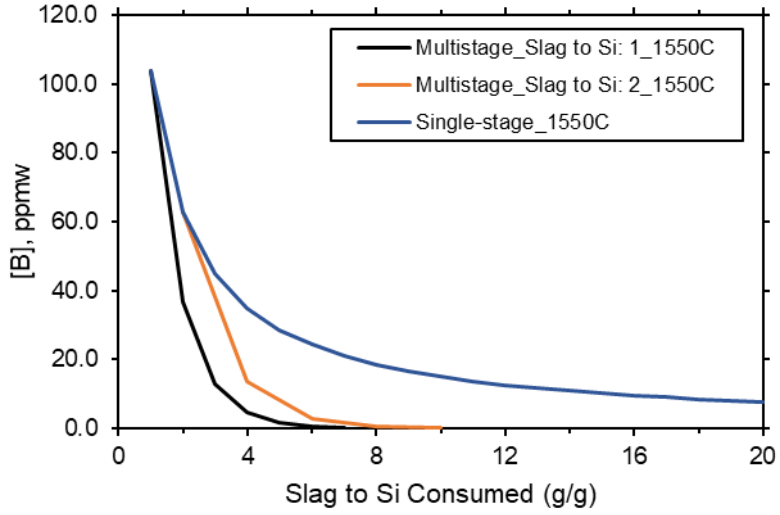

FIG 6 - Boron concentration in silicon after slag treatment for single-stage (blue), multi-stage with slag to Si ratio = 2:1 (orange), and multi-stage with slag to Si ratio = 1:1 (black) at 1550 ºC.

Experimental result and discussion

Physical appearance

Silicon and slag exist as two immiscible melts in the crucible. In this case, silicon has lower density than the used slag, therefore, silicon floats as a droplet above-inside the slag melt. After cooling, the crucible was shattered using a hammer, and some parts of the sample detached from the crucible were preserved for further experiment and analysis. Due to evaporation, the weight of the sample always reduced in each stage, as shown in Table 2.

On one hand, slag samples can be used in a destructive analysis, such as the ICP-AES because it is not used multiple times. On the other hand, silicon is not as flexible as the slag sample. The physical appearance of the shattered sample is provided in Figure 7 and the prepared sample for LA-ICP-MS is provided in Figure 8.

FIG 7 - Silicon-slag sample after 1st stage of slag treatment at 1550 ºC for 2 hours. A big portion of the silicon was taken for 2nd stage of slag treatment.

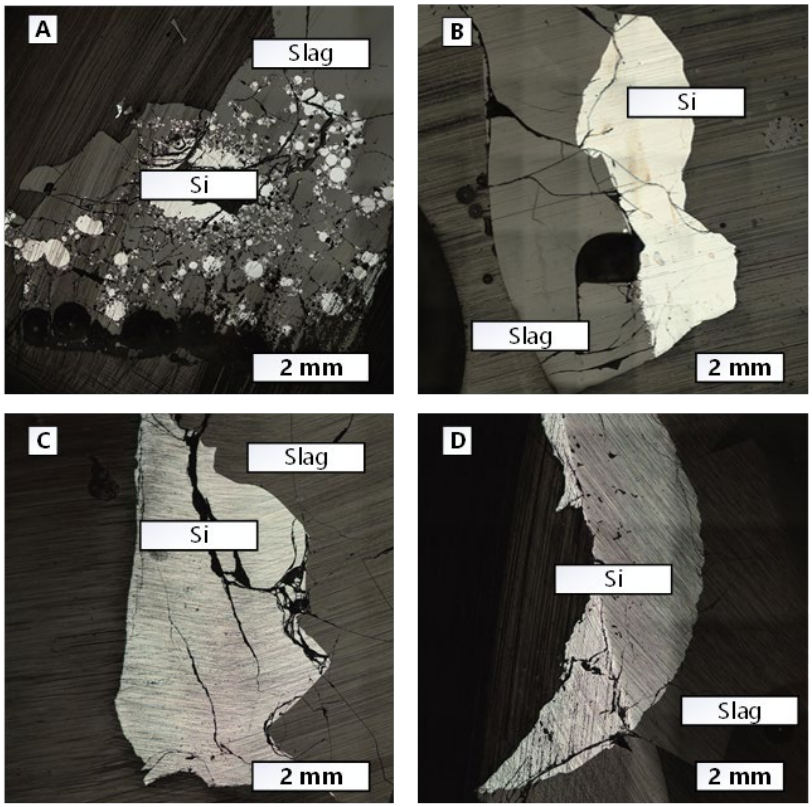

FIG 8 – Optical microscope images of silicon and slag samples from multistage slag treatment. (A) 1st stage, (B) 2nd stage, (C) 3rd stage, and (D) 4th stage.

Concentration results

The minor elements including boron, aluminium, calcium, and iron were measured by the LA-ICP-MS, whereas the major oxide concentrations were analysed by the ICP-AES. The results show that the boron concentration in the initial master silicon was 270 ppmw. This master silicon also contained traces of aluminium (810 ppmw) that primary have dissolved from the alumina crucible during the master alloy making and iron (930 ppmw) that may come from the comminution process or as an impurity from the chemicals used.

The boron concentration in the silicon from 1st to 4th stage were measured as 61.4; 12.0; 6.5; and 6.0 ppmw, respectively, showing a clear decrease of boron in the silicon alloy. In total, boron was successfully removed by approximately 97.8% from the silicon alloy. The results indicate that the multistage slag treatment was successful to remove boron from the silicon more effectively than the previous works using a single slag treatment. Previous study of a single stage slag treatment with similar slag composition, temperature and initial boron concentration reached approximately 110-120 ppmw of boron concentration in the silicon (Islam & Rhamdhani, 2018).

However, the decrease of the boron concentration in the silicon was less significant in the latter stages, which may be caused by contamination from the furnace or evaporation of both silicon and slag phases. Also, boron can be considered to vaporize from the system, as especially in the first 2 stages, its concentration decreased in both phases in much higher extent than initial concentration of B would predict. This occurs, even though, clearly alumina dissolved to the slag from the crucible and more slag mass/volume is expected to exist after the melting indicating to lower B, see slag results in Table 3. Figure 9 presents the boron removal from silicon and boron behaviour in the Si-slag system by the multi-stage treatment. The result show that the boron distribution coefficient is rather constant, between 1.4 and 2.4 which indicates that the boon concentration in both phases is at equilibrium (or close to equilibrium) at each stage. The achieved L_B results from the experiments also fit with the simulation results.

Other minor elements, such as aluminium and calcium appeared at higher concentrations in some of the silicon alloy samples, which may be caused by the reactions within the silicon-slag-crucible system. Iron also was measured at higher concentration in some stages, which may be caused by multiple comminution process. However, the fluctuating values of the mentioned minor elements show that the multistage slag treatment does not make any great impurity accumulation in the silicon sample.

TABLE 3 – ICP-AES and LA-ICP-MS analysis result of the multistage experiments.

Sample	B, ppmw	Al, ppmw	Ca, ppmw	Fe, ppmw	SiO_2, %	CaO, %	Al_2O_3, %
Master Si	270	810	<20	930			
Si-1st stage	61.4	143.1	68.8	1563.5	-	-	-
Si-2nd stage	11.9	439.9	78.8	11.0	-	-	-
Si-3rd stage	6.5	4664.8	254.7	233.9	-	-	-
Si-4th stage	6.0	421.6	64.3	117.2	-	-	-
Master slag	-	-	-	-	43.9	47.6	8.34
Slag-1st stage	89.0	-	-	104.5	45.8	37.1	16.0
Slag-2nd stage	30.9	-	-	110.6	46.5	33.9	18.5
Slag-3rd stage	13.7	-	-	97.2	52.1	28.7	23.0
Slag-4th stage	8.6	-	-	97.6	45.8	30.2	23.0

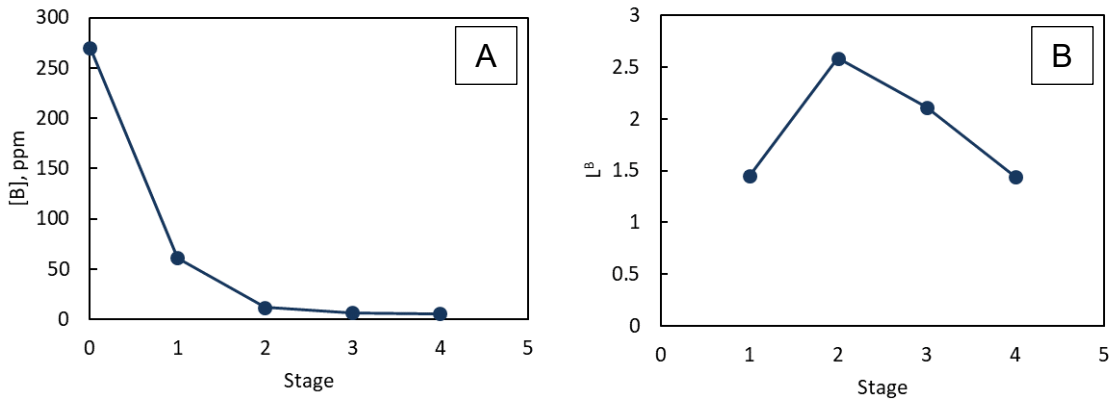

FIG 9. Boron concentration in silicon (A) and boron distribution coefficient (B) in each stage of the multistage silicon slag treatment.

CONCLUSIONS

Boron removal from silicon using multistage slag treatment was successfully simulated and experimentally investigated. Using the FactSage ® 8.2 (FactPS, FTOxid and FSupsi databases), it was demonstrated that the boron distribution coefficient is independent on slag to Si ratio. This feature was harnessed by applying multistage slag treatment process and the boron can be consistently reduced by approximately 78% in each stage using slag to Si mass ratio of 2.

The laboratory experimentation was carried out by reacting master silicon (with 270 ppmw of boron) and $45SiO_2$-$45CaO$-$10Al_2O_3$ (wt. pct.) slag at 1550 ºC and Ar gas atmosphere for 2 hours. The experiment was carried out using slag to Si mass ratio of 2, in 4 stages by adding a new slag in each stage. Boron concentrations in the master silicon, 1st to 4th stage, can be reduced from 270 to 61.4; 12.0; 6.5 and 6.0 ppmw, respectively. The current study demonstrated that the multistage slag treatment can be utilised to gradually remove boron from silicon. Other minor element such as Al, Ca, and Fe contamination in the silicon showed no radical accumulation during the multiple slag treatment that was carried out.

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