

# Manufacturing of FeSiB high-temperature phase change material by silicothermic reduction

*Jianmeng Jiao<sup>1</sup>, Maria Wallin<sup>2</sup>, Wojciech Polkowski<sup>3</sup>, and Merete Tangstad<sup>4</sup>*

1. Postdoc researcher, Norwegian University of Science and Technology, Department of Materials Science and Engineering, 7491, Trondheim, Norway. Email: jian.m.jiao@ntnu.no
2. Researcher, Norwegian University of Science and Technology, Department of Materials Science and Engineering, 7491, Trondheim, Norway. Email: maria.wallin@ntnu.no
3. Researcher, Norwegian University of Science and Technology, Department of Materials Science and Engineering, 7491, Trondheim, Norway. Email: polkowski.wojciech@ntnu.no
4. Professor, Norwegian University of Science and Technology, Department of Materials Science and Engineering, 7491, Trondheim, Norway. Email: merete.tangstad@ntnu.no

Keywords: FeSiB, B<sub>2</sub>O<sub>3</sub>, silicothermic reduction, PCM, TES.

## ABSTRACT

Fe-26Si-9B (wt.%) alloy has been identified as a potential high-temperature phase change material (PCM) due to its attractive properties, such as its high latent heat of fusion and low volumetric change during solid/liquid transition. For the successful utilization of this alloy into thermal energy storage (TES) systems, the development of a cost-effective production method is essential. Presently, the Fe-26Si-9B alloy is produced by mixing FeSi alloys with either pure boron element or FeB alloys. However, the use of pure boron is financially prohibitive, and the carbothermic reduction results in high greenhouse gas emissions and high energy consumption in the production of FeB alloys. In this regard, our study proposes a silicothermic reduction method to produce Fe-26Si-9B PCM by using FeSi alloys and B<sub>2</sub>O<sub>3</sub>-based oxides. Accordingly, the influence of various parameters on the production process was investigated, including operating temperature, holding time, B<sub>2</sub>O<sub>3</sub> content in the added oxides, and initial Slag/Metal (S/M) ratio. Based on the experimental results, the optimal parameters for producing FeSiB alloys with over 9wt.% boron were determined. Consequently, it was documented that the FeSi alloys and the added oxides enriched with 50-65 wt.% B<sub>2</sub>O<sub>3</sub> should be subjected to temperatures ranging from 1550-1650 °C, maintain an initial S/M ratio exceeding 1, and ensure a holding duration beyond 1h. Moreover, the energy consumption of this process was estimated to be ~1.86 MWh/t metal and the mass loss was lower than 7 %. Therefore, silicothermic reduction offers a sustainable approach for producing FeSiB alloys with a boron content above 9 wt.%.

## 1. INTRODUCTION

Renewable energy sources, such as solar and wind, play an important role in achieving climate neutrality (EASE, 2023). However, a mismatch between energy supply and energy demand is the main challenge in their applications. By addressing this problem, thermal energy storage (TES) techniques have been developed, in which using of phase change materials (PCMs) has emerged as a particularly promising solution (IEA, 2019). Recently, Fe-26Si-9B (wt.%) eutectic alloy stood out due to its moderate melting temperature, high energy storage capacity, low volumetric change, and high thermal conductivity (Grorud, 2018; Sellevoll, 2018, 2019; Sindland, 2018; J. Jiao *et al.*, 2019; J. M. Jiao *et al.*, 2019; Jiao, 2020; Jiao, Safarian and Tangstad, 2022). However, the traditional methods of producing this alloy are based either on mixing pure iron (Fe), silicon (Si), and boron (B) elements, or on mixing ferroboration (FeB) and ferrosilicon (FeSi) alloys (Jiao *et al.*, 2023). The former approach is financially prohibited, particularly due to the cost of pure boron. The latter suffers from its high greenhouse gas emissions and a high energy consumption.

To tackle these challenges, a new route is proposed for manufacturing Fe-26Si-9B PCM from raw and waste materials in the Thermobat project funded by the European Commission. The strategy is to produce this alloy by using a metallothermic reduction, for which silicon is selected as the reducing agent. The primary reaction between silicon and  $B_2O_3$  is exothermic, and thus, this method has a low energy consumption. The process starts with mixing FeSi and  $B_2O_3$ -based oxides at high temperatures followed by a production of molten mixture of FeSiB alloys and  $SiO_2$ -based slags. Additionally, the chemical composition of FeSiB alloys can be adjusted at its molten state before tapping. Besides, the raw materials are abundant and cheap and can be obtained from iron and FeSi scraps, silicon kerf-loss waste and discarded FeSi and silicon fines. Thus, this process is expected to produce FeSiB alloys with a low cost.

The present study focused on optimizing the production process of FeSiB alloys. Firstly, the experimental process is established theoretically through thermodynamic analyses. Then, we explore the effect of process parameters on the boron content in the resulting FeSiB alloys. The investigated process variables include holding time,  $B_2O_3$  content in the added oxides, initial Slag/Metal (S/M) ratio, and operating temperature.

## 2. MATERIALS AND METHODS

In the production of FeSiB alloys, Fe-75Si and iron alloys, CaO,  $B_2O_3$ , and colemanite powders ( $CaO \cdot 2B_2O_3 \cdot 5H_2O$ ) were used in the experiments. Fe-75Si alloy was sourced from Finnjord AS, Norway, and the chemical composition of iron and silicon was analyzed to be 25.78 wt.% and 74.22 wt.% using Inductively Coupled Plasma Sector Field Mass Spectrometry (ICP-SFMS), respectively. Iron metal (99.99 wt.%) and CaO powder (99.95 wt.%) came from Alfa Aesar.  $B_2O_3$  powder (98 wt.%) obtained from Thermo Scientific, and commercial colemanite powder (particle size < 75 $\mu$ m) was provided by Eti Maden company (ETi Maden, 2015). It is important to note that prior to use colemanite, it must undergo calcination to eliminate its crystal water. This involved maintaining the colemanite powders in a muffle furnace at 600 °C for 180 min. The content of  $B_2O_3$  was analyzed to be ~ 55 wt.% in the calcinated colemanite using the ICP-SFMS technique.

The experiments were conducted in a resistance furnace under an argon (99.999% Ar) atmosphere. 7 to 24 g of  $B_2O_3$ -based oxides and 5 to 15 g of FeSi alloys were placed in the BN-coated SiC crucibles. The thin BN spray coating was applied to prevent the oxides creeping from the crucible due to its good wetting behavior with SiC. Notably, the  $B_2O_3$ -based oxides were either pre-produced by mixing pure  $B_2O_3$  and CaO powders or taken directly from the calcinated colemanite. The added oxides were placed at the bottom of crucible, and the Fe-41Si master alloy pieces were layered at the top of these oxides, where the Fe-41Si master alloy was produced by mixing Fe-75Si and iron metals in an induction furnace. This arrangement aimed to enhance mass transfer during the process, as after melting, the molten alloys would settle at the crucible's bottom, and the molten slags would ascend due to their density differentials. Subsequently, the holding temperature was set in the range of 1500-1650 °C, maintaining this for a duration of 5 to 180 min. The purpose was to find the optimal parameters to produce FeSiB alloy having the boron content above 9 wt.%. After experiments, the chemical composition of the produced alloys was analyzed using ICP-SFMS. Furthermore, thermodynamic analyses were conducted by FactSage 8.1, using databases such as FactPS, FTlite, FToxide, and Melts database within the viscosity module. The energy consumption

associated with the process was estimated using the Heat and Material Balance module of HSC Chemistry 9 software.

### 3. RESULTS AND DISCUSSION

#### 3.1. Silicothermic reduction process design

The Fe-26Si-9B eutectic alloy was initially designed by FactSage 7.2 using the FTlite database (J. Jiao *et al.*, 2019). However, the experimental results have revealed that the actual eutectic alloy's composition was near but not precisely at the eutectic point (Jiao, 2020). The Fe-29Si-10B alloy was further regarded as the eutectic alloy based on the phase distribution in the observed eutectic structures (Jiao, 2020). Then, when a new version of FactSage 8.1 was applied in the thermodynamic analyses, the composition of this eutectic FeSiB alloy was changed to Fe-24Si-11B. This led to the assumption that the eutectic point for this system was in a range of 61-65 wt.% iron, 24-29 wt.% silicon, and 9-11 wt.% boron. For this research, the silicothermic reduction process was designed based on the FactSage 8.1 using the FTlite and FToxide databases. The chemical reactions were expressed as:



Here, the parentheses indicate that the oxides are in a molten state, and the underline denotes that the metal elements are in their molten state.

FIG 1 shows the calculated iso-boron lines of FeSiB alloys within the CaO-SiO<sub>2</sub>-B<sub>2</sub>O<sub>3</sub> liquidus region at 1500 °C, fixing a constant Fe content at 65 wt.%. It is observed that an increase in the boron content in FeSiB alloys is directly proportional to the increased content of B<sub>2</sub>O<sub>3</sub> in the equilibrium slags. Here, the 11 % boron line shows the composition of the equilibrium CaO-SiO<sub>2</sub>-B<sub>2</sub>O<sub>3</sub> slags corresponding to the production of Fe-24Si-11B alloy. So, to produce an FeSiB alloy with a boron content over 11 %, the added CaO-SiO<sub>2</sub>-B<sub>2</sub>O<sub>3</sub> oxides should ideally be situated within the light green area in the diagram, where the B<sub>2</sub>O<sub>3</sub> content exceeds 25 wt.% in the equilibrium CaO-SiO<sub>2</sub>-B<sub>2</sub>O<sub>3</sub> slags.

FIG 2 presents the predicted effects of B<sub>2</sub>O<sub>3</sub> content in the added oxides on both the mass of CaO-B<sub>2</sub>O<sub>3</sub> oxides added and the viscosity of the equilibrium CaO-SiO<sub>2</sub>-B<sub>2</sub>O<sub>3</sub> slags. In the production of 1 t Fe-24Si-11B alloy, it is found that an increase of B<sub>2</sub>O<sub>3</sub> content and consequently a decrease in the CaO content, less slag will be produced and hence it leads to a decrease of the requested mass of CaO-B<sub>2</sub>O<sub>3</sub> oxides. Conversely, the viscosity of the equilibrium slags increases with higher B<sub>2</sub>O<sub>3</sub> content in the added CaO-B<sub>2</sub>O<sub>3</sub> oxides. In the production process, the goal is to use as little of the added CaO-B<sub>2</sub>O<sub>3</sub> oxides as possible while keeping the slags fluid enough. However, it's hard to achieve both low mass of the CaO-B<sub>2</sub>O<sub>3</sub> oxides added and its low viscosity at the same time. According to FIG 2, a critical intersection point of these parameters is ~ 55 wt.% B<sub>2</sub>O<sub>3</sub>. Hence, the added CaO-B<sub>2</sub>O<sub>3</sub> oxides containing the B<sub>2</sub>O<sub>3</sub> in the range of 50-65 wt.% are investigated in the experiments. Ideally, this B<sub>2</sub>O<sub>3</sub> composition range corresponds to an input mass for the added oxides between 1.6-2.7 t and a viscosity range for the equilibrium slags of 1-1.8 poise in the production of 1 t Fe-24Si-11B alloy.

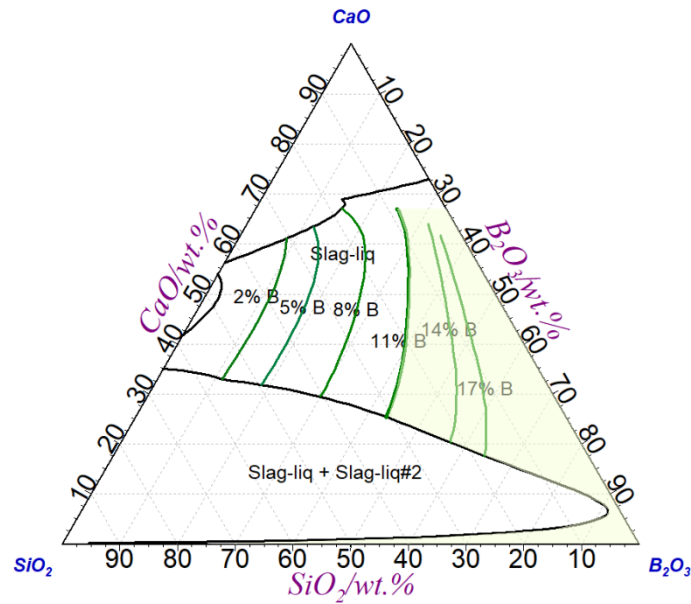


FIG 1 – CaO-SiO<sub>2</sub>-B<sub>2</sub>O<sub>3</sub> phase diagram at 1500 °C, showing iso-boron lines (in green) for Fe-Si-B alloys (65 wt.% iron) with their equilibrium slags. The light green region indicates the compositional range of the added oxides to produce the Fe-24Si-11B alloys (FTlite + FToxide databases).

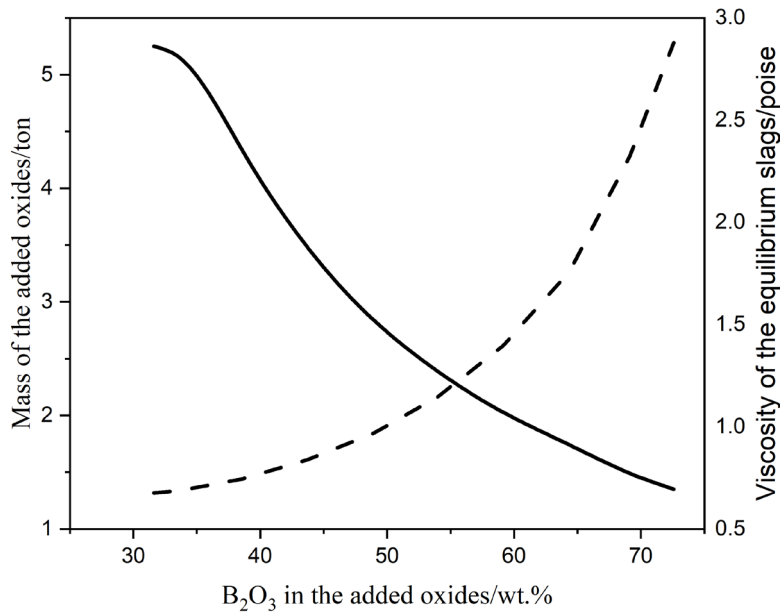


FIG 2 – The solid line shows the relationship between the content of B<sub>2</sub>O<sub>3</sub> in CaO-B<sub>2</sub>O<sub>3</sub> added oxides and the mass for producing 1ton Fe-24Si-11B alloy at 1500 °C, and the dashed line shows its corresponding viscosity of the equilibrium CaO-SiO<sub>2</sub>-B<sub>2</sub>O<sub>3</sub> slags (Melts, FTlite, and FToxide databases).

### 3.2. Effect of holding time

FIG 3 shows the influence of holding time on the boron content in the produced FeSiB alloys, where the Fe-41Si master alloy and CaO-65B<sub>2</sub>O<sub>3</sub> oxides were subjected to 1650 °C for the holding time ranging from 5 to 120 min with the fixed initial S/M ratio of 1.6. It is observed that the boron content increased with the increase in holding time, tending towards the equilibrium concentration of 10.3 wt.%, as shown in the dashed line. It reveals a rapid initiation of the slag/metal reaction within the initial 5 min, achieving a boron content ranging from 2.6 to 5 wt.%. Then, a more gradual increase was observed over time. The system was close to its equilibrium state after 60 min holding time, where the boron content was increased to ~ 9 wt.%. Interestingly, the boron content was higher than the predicted equilibrium content after 120 min holding time, which was impossible. This deviation might be caused by the inaccuracy in the FactSage database. These results show that a holding time of 60 min is necessary to achieve a boron content over 9 wt.% in the FeSiB alloys.

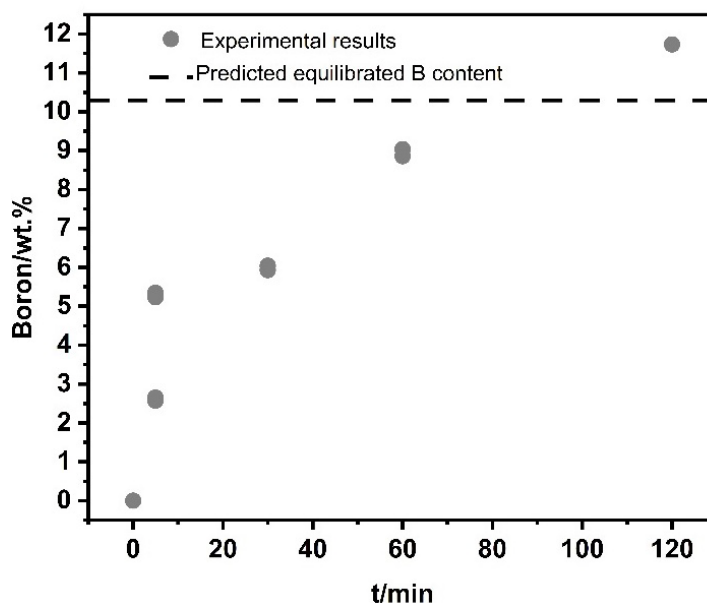


FIG 3 – The effect of holding time on boron content in the produced FeSiB alloys. Fe-41Si and CaO-65B<sub>2</sub>O<sub>3</sub> oxides is reacted at 1650 °C with the initial S/M ratio of 1.6. The dashed line represents the predicted equilibrium B content (FTlite, and FToxide databases).

### 3.3. Effect of B<sub>2</sub>O<sub>3</sub> content in the added oxides

FIG 4 presents a comparison of the experimental results and thermodynamic predictions regarding the impact of B<sub>2</sub>O<sub>3</sub> content in the added oxides on the boron content in the produced FeSiB alloys. In the experiments, Fe-41Si master alloys reacted with a range of B<sub>2</sub>O<sub>3</sub>-based oxides at an initial S/M ratio of 1.6 during 60 min holding time at 1650 °C. It is noted that 55 wt.% B<sub>2</sub>O<sub>3</sub> based oxides were used directly from the calcinated colemanite, whereas 50 wt.% and 65 wt.% B<sub>2</sub>O<sub>3</sub> based oxides were synthesized by blending pure CaO and B<sub>2</sub>O<sub>3</sub> powders. It is seen from the modelling results that the boron content increased slightly in the produced FeSiB alloys with a higher B<sub>2</sub>O<sub>3</sub> content in the added oxides. Experimental results showed that the boron content in the alloys had a good agreement with the thermodynamic predictions by using 50 wt.% and 55 wt.% B<sub>2</sub>O<sub>3</sub> based oxides. Conversely, when employing 65 wt.% B<sub>2</sub>O<sub>3</sub> based oxides, the analyzed boron content was lower than the predicted equilibrium boron content, indicating that the reaction did not reach equilibrium within a 60 min holding time. It implicates that the mass transfer was likely the limiting step in this process. Therefore, an increase in the B<sub>2</sub>O<sub>3</sub> content in the added oxides from 50 wt.% to 65 wt.%, a longer holding time is expected to achieve equilibrium.

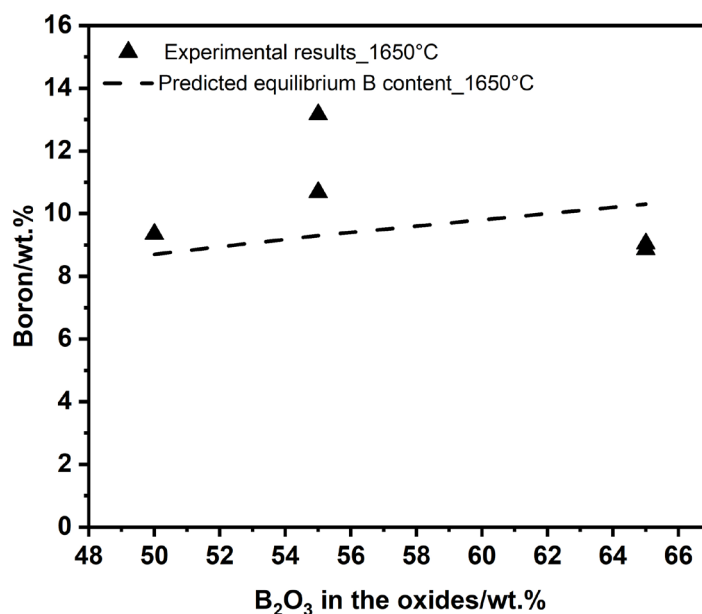


FIG 4 – The effect of  $B_2O_3$  content in the added oxides on boron content in the produced FeSiB alloys. The points represent the experimental results, and the dotted line represents the predicted results based on FactSage 8.1 using FToxide and FTlite databases. The calcinated colemanite was used as the 55 wt.%  $B_2O_3$  based oxides.

### 3.4. Effect of the initial S/M ratio

FIG 5 shows the relationship between the initial S/M ratio and boron content in the produced FeSiB alloys. These experiments involved a reaction of Fe-41Si master alloys with the calcined colemanite (~55 wt.%) at different initial S/M ratios, maintaining the reaction for 60 min at 1650 °C. The analyzed boron content in the produced alloys was summarized in the figure, accompanied by the predicted equilibrium boron content represented by the dashed line. There was a clear trend indicating that the boron content increased with an increasing initial S/M ratio, as a higher initial S/M ratio would give a higher  $B_2O_3$  for a given boron content, and hence the driving force would be higher. Significantly, the boron content was higher than 9 wt.% in the FeSiB alloys when the initial S/M ratio was over 1. In the comparison of the experimental results and the predicted equilibrium boron content, it is found that the experimental results were close to or higher than that from the thermodynamic modelling, as mentioned before, this deviation might be caused by an inaccurate FactSage database. According to the experimental results, to achieve a boron content higher than 9 wt.% in our target FeSiB alloys, the initial S/M ratio should be greater than 1.

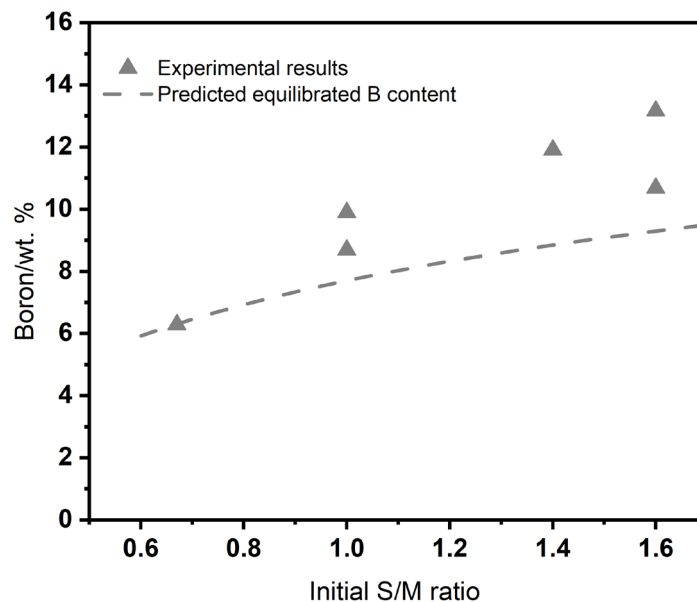


FIG 5 – The effect of initial Slag/Metal (S/M) ratio on boron content in the produced FeSiB alloys. The points represent the experimental results, and the dash line represents the predicted result by FactSage 8.1 using FToxide and FTlite databases.

### 3.5. Effect of operating temperature

FIG 6 shows the impact of operating temperature on the boron content in the produced FeSiB alloys. These alloys were produced by reacting Fe-41Si master alloys with the calcined colemanite at the initial S/M ratio of 1 and 1.6, within a 60 min holding time at temperatures range of 1500-1650 °C. The figure summarized the experimental results, with the dashed lines indicating the predicted equilibrium boron content for these initial S/M ratios. The experimental data reveals that an increase in temperature at a constant initial S/M ratio led to a higher boron content. In contrast, the predicted equilibrium boron content decreases with increasing temperature, indicating that equilibrium was not achieved at lower temperatures (1550 °C and 1500 °C) within 60 min holding time. It is noted that a lower temperature was preferable for the silicothermic reduction process. However, a lower temperature also resulted in a higher viscosity of the molten slag, which impeded mass transfer during the process. Thus, at a holding time of 60 min, mass transfer became the limiting factor.

The two red lines in the figure shows the trend of B content with increasing temperature at initial S/M ratios of 1 and 1.6, respectively. These two lines were drawn based on the experimental data. It was

observed that a higher initial S/M ratio resulted in a higher boron content at a given temperature and holding time, due to a stronger driving force in the slag/metal systems with a higher initial S/M ratio. Consequently, the system with a higher S/M ratio was likely to reach equilibrium in a shorter holding time at a constant temperature. Therefore, for a 60 min holding time and an initial S/M ratio greater than 1, the optimal operating temperature is approximately 1650 °C for the calcinated colemanite. To achieve equilibrium at temperatures below 1650 °C, a longer holding time would be necessary.

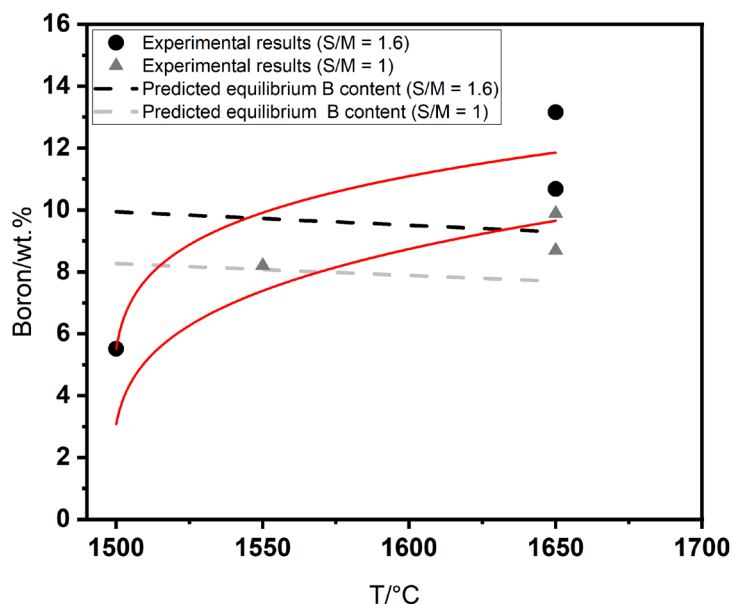


FIG 6 – The effect of temperature on boron content in the produced FeSiB alloys. The points represent the experimental results, and the dash lines represent the predicted results based on FactSage 8.1 using FToxide and FTlite databases.

### 3.6. Mass loss

FIG 7 presents a plot of mass loss as a function of holding time under various temperatures, in the production of FeSiB alloys using B<sub>2</sub>O<sub>3</sub>-based oxides. The measurement of mass loss was conducted by comparing the total weight of raw materials before and after conducting the experiments. It reveals that the mass loss was below 7 %. In the experiments using CaO-65B<sub>2</sub>O<sub>3</sub> at 1500 °C, the mass loss increased proportionally with holding time, from 0.4 % after 5 min to 3.5 % after 180 min. Additionally, when examining the impact of temperature on mass loss with a constant holding time of 60 min using the CaO-65B<sub>2</sub>O<sub>3</sub>, an increase in temperature led to an increase in mass loss, from 0.1 % at 1400 °C to 4.1 % at 1650°C. In contrast, when it came to the calcinated colemanite, the behavior was less predictable. It varied in the range of 2-7 % after 60 min holding time at 1650 °C. A review of the data indicates that the use of calcinated colemanite led to a higher mass loss compared to CaO-65B<sub>2</sub>O<sub>3</sub> master oxides. FIG 8 shows the partial pressure of potential species in the CaO-SiO<sub>2</sub>-B<sub>2</sub>O<sub>3</sub> system across a temperature range of 1200-1700 °C. This suggests that the mass loss was primarily due to the evaporation of B<sub>2</sub>O<sub>3</sub> in the system. Given these findings, it is suggested to compensate for this loss by adding an additional B<sub>2</sub>O<sub>3</sub> to the oxides charged in the FeSiB alloy production process.

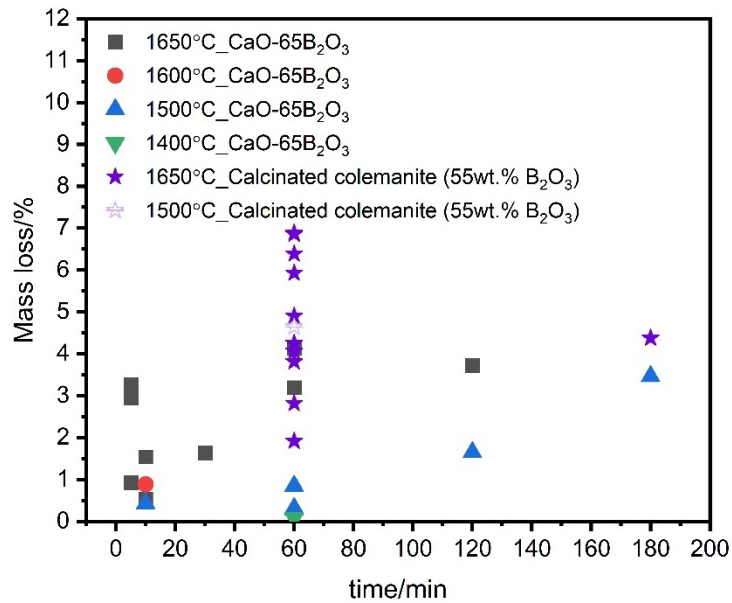


FIG 7 – Mass loss vs. holding time in the silicothermic reduction process.

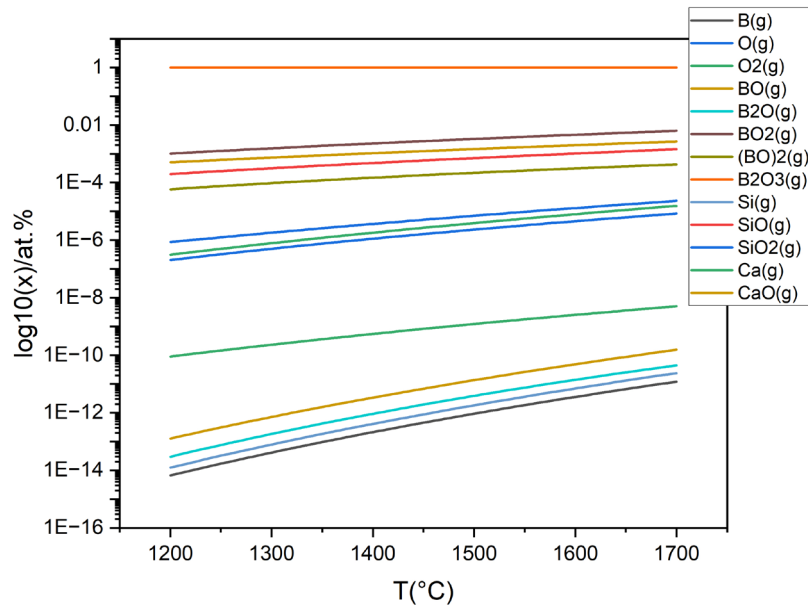


FIG 8 – The partial pressure of possible species in the CaO-SiO<sub>2</sub>-B<sub>2</sub>O<sub>3</sub> system at a standard atmosphere. It was calculated by FactSage 8.1 using FactPS database.

### 3.7. Energy balance

In the production of 1 ton Fe-24Si-11B alloy, the estimated theoretical energy consumption was 1860kWh/t metal. This estimation was calculated based on a mass balance achieved at 1650°C under thermodynamic equilibrium. The relevant stoichiometry equation is as follows:



Further analysis of the electrical energy consumption was carried out by using HSC Chemistry software (version 9). This analysis was based on the principle of enthalpy conservation, as shown in the equation:

$$\text{enthalpy in input materials} + \text{electrical energy} = \text{enthalpy in output products} \quad (3)$$

Subsequently, the energy flow involved in the production of 1ton Fe-24Si-11B alloy was illustrated in the Sankey diagram, as it is presented in FIG 9. It is observed that the energy from the input metal was slightly higher than the energy from the output metal, attributed to the exothermic reaction between silicon and B<sub>2</sub>O<sub>3</sub>. The electrical energy (1860kWh/t of metal) was directly transformed to the



heat in the output metal and slag. Given these observations, the challenge was to develop an efficient method to reuse the heat in the products. This is crucial for improving energy efficiency and sustainability of the proposed production process.

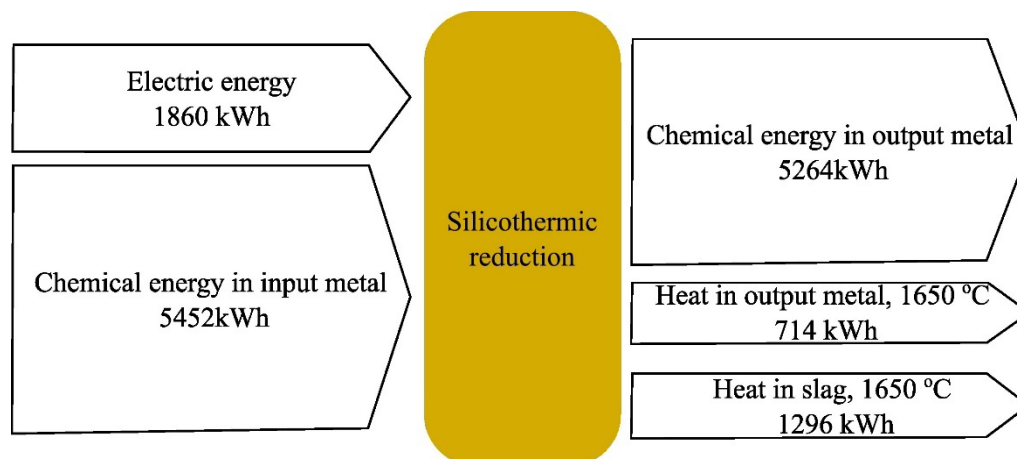


FIG 9 – Sankey diagram, illustrating the energy flow for the production of 1t Fe-24Si-11B alloy. Fe-41Si and CaO-65B<sub>2</sub>O<sub>3</sub> oxides reacted at 1650°C with an initial S/M ratio of 1.6. Thermal loss was not included.

#### 4. CONCLUSIONS

This research aimed to develop a method to produce FeSiB alloys, targeting a composition of 61-65 wt.% iron, 24-29 wt.% silicon, and 9-11 wt.% boron. This method was based on the silicothermic reduction between silicon and B<sub>2</sub>O<sub>3</sub>. In this regard, the experiments were conducted in a resistance furnace under an argon atmosphere. The parameters in the influence of the boron production in the alloys were investigated. According to the experimental results, the optimal procedure involved reacting an Fe-41Si master alloy with oxides containing over 50 wt.% B<sub>2</sub>O<sub>3</sub> by maintaining an initial Slag/Metal (S/M) ratio of 1-1.6. The mixture was then subjected to the temperatures above 1550 °C for a duration over 60 min. The mass loss was observed to be below 7 %, and the energy consumption was estimated to be ~ 1860 kWh/t metal. These findings provided a basis to develop reliable and efficient method for producing the targeted FeSiB alloys with the boron content above 9 wt.%.

#### ACKNOWLEDGEMENTS

The THERMOBAT project received funds from European Commission under grant agreement 10105754. The sole responsibility for the content of this publication lies with the authors. It does not necessarily reflect the opinion of the European Union. Neither the REA nor the European Commission is responsible for any use that may be made of the information contained therein.

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