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Research Article

Experimental Study of the Effect of B_2O_3 on Vanadium-Titanium Magnetite Concentrates Pellets

Hao Liu,[1,2] **Ke Zhang**,[1] **Henrik Saxén**,[3] **Weiqiang Liu**,[3] **Yuelin Qin**,[1,2] **and Xiaoyan Xiang**[1,2]

[1]*School of Metallurgy and Materials Engineering, Chongqing University of Science and Technology, Chongqing 401331, China*
[2]*Value-Added Process and Clean Extraction of Complex Metal Mineral Resources, Chongqing Municipal Key Laboratory of Institutions of Higher Education, Chongqing 401331, China*
[3]*Process and Systems Engineering Laboratory, Faculty of Science and Engineering, Åbo Akademi University, Biskopsgatan 8, 20500 Åbo, Finland*

Correspondence should be addressed to Xiaoyan Xiang; xxycsu@163.com

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This paper aims to improve the metallurgical properties of vanadium-titanium magnetite (VTM) concentrates pellets by applying solid waste containing B_2O_3. Thus, the effects of adding B_2O_3 on the drop strength, compressive strength, pores area ratio, high-temperature metallurgical properties, and microstructure of VTM pellets were studied through pelletizing and roasting experiments. Results show that the addition of B_2O_3 reagent is not conducive to the increase of the drop strength of the green pellets. Nevertheless, the compressive strength and fracture toughness of the roasted pellets can be improved by adding more B_2O_3 during the pelletizing. The reduction degree of VTM pellets is firstly decreased and then increased with the added B_2O_3 amount. It is possible to improve the compaction degree and restrain the reduction-pulverization degree of the pellet by a low amount of additive (B_2O_3). The reduction-expansion performance of VTM pellets, in turn, can be raised by adding B_2O_3.

1. Introduction

Vanadium-titanium magnetite (VTM), which is found in rich reserves, has been widely used in the steel-making industry in recent years. Currently, VTM has been processed by the blast furnace-converter route, where the vanadium is extracted from the vanadium-rich slags [1]. Vanadium-titanium concentrates have been used as one of the raw materials for making pellets used for blast furnace iron-making in China since 2009 [2]. The vanadium-titanium pellets show less expansion performance than hematite pellets [3]. However, the high-temperature reduction performance of vanadium-titanium pellets is worse than for ordinary pellets [4]. The high-temperature metallurgical performance of pellets can be improved through different methods, such as grinding pretreatment and optimizing the ore matching structure.

Metallurgical properties of the pellets can be improved by the addition of solid fuels, basic fluxes, and boron-containing oxides. Wastes containing B_2O_3, such as boric mud and boric acid waste liquor (produced during the extraction process of boron from boron ore), can cause considerable environmental pollution. These byproducts are also enriched with magnesium, silicon, and boron elements. Therefore, using these materials in the pelleting process may enhance pellet quality and also eliminate related waste. Akberdin et al. [5] found that the addition of B_2O_3 or borate minerals to the pelletizing mix can improve the strength of the pellets, reduce the initial temperature of liquid phase formation, and accelerate the desulfurization process. Cheng et al. [6] investigated the production of high-chromium VTM oxide pellets by adopting B_2O_3 as an additive. The results showed that the compressive strength of high-chromium VTM pellets was significantly enhanced, the gangue phase and pores were greatly reduced, and a considerable amount of magnesium-aluminum spinel phase was formed with the increase of the B_2O_3 additive. The author of

the present paper has also studied the effects of adding B_2O_3 on the assimilation characteristics, softening temperature, the fluidity of liquid phase, compressive strength of bonding phase, and microstructure of the mixed powder of hematite and vanadium-titanium magnetite (H-VTM) [7]. Ren et al. [8, 9] found that B_2O_3 (mainly deposited in the $2CaO•xSiO_2•2/3\ (1-x)\ B_2O_3$ phase of high-titanium-type vanadium-titanium sinter) could effectively inhibit the precipitation of $CaTiO_3$ and $2CaO•xSiO_2•2/3\ (1-x)\ B_2O_3$, but promote the precipitation of iron-containing mineral phase. Zhang and Yang [10] analyzed the distribution law of the boron element in sinter-pot experiments and found that boron was mainly deposited in the glass phase of the sinter. It can be seen from the above research that boron oxide has played a certain role in the metallurgical properties of vanadium-titanium sinter. It is also necessary to study the effect of adding boron oxide on the metallurgical properties of vanadium-titanium pellets, which is another commonly used charge for pellet ore blast furnaces, but related studies are rarely reported.

Yu et al. [11] found that the compressive strength of the roasted pellets containing boron-bearing additive could reach almost 3100 N/pellet by preheating the green pellets (at 1000°C) 8 min, roasting them at 1280°C and then keeping this temperature for 15 minutes. This compressive strength is far beyond the standard requirement of good-quality pellets. Fu and Chu et al. [12] clarified that boron-bearing iron concentrate could increase the crushing strength of pellets and decrease the reduction swelling rate. Increasing the amount of the addition of boron-bearing iron concentrate from 0 to 7.5%, the crushing strength was raised from 2630 N to 3709 N, while the reduction swelling rate was decreased from 25.7% to 15.5%. When B_2O_3 entered into the pellet slag phase, the melting point and viscosity of the slag phase were both reduced [13]. As a result, the bonding effect was improved, and the strength of the pellets was increased [14]. The addition of B_2O_3 can improve not only the cold strength of the pellets but also their metallurgical performances [15]. Moreover, the roasting temperature of the pellet was reduced, and the range of the roasting temperature was also expanded by adding B_2O_3 in the pellet. Furthermore, the surface quality of the pellets was improved accordingly [10]. From the perspective of mechanical properties, fracture toughness can objectively measure the difficulty of crack propagation in minerals and reflect the fracture resistance of minerals. Among many fracture toughness testing methods, indentation technology is widely used because of its simple and economical preparation of samples. Ying et al. [16] used the Vickers indentation test to evaluate the generation and growth of cracks in sinters containing calcium ferrite with different morphology and examine the effect of calcium ferrite morphology on the antifracture ability of sinters. Wu et al. [17] investigated the microscopic mechanical properties of the three different forms of hematite in two kinds of pellets and analyzed the influence mechanism of hematite morphology on the strength of pellets. Therefore, this paper also uses such methods to study the micro-mechanical properties of pellets.

For the previous investigations mentioned above, it was found that the performance of the pellets with magnetite can be improved by adding boron slime, boron acid, and boron-containing iron concentrates. Intending to further develop the vanadium-titanium pellets, the present study studied the effects of B_2O_3 on pellets (made of vanadium-titanium concentrates) by an experimental approach. The B_2O_3 analytical reagent is used to systematically study the mechanism of forming and roasting consolidation of VTM concentrates pellets. Before pelletizing, the VTM concentrates and bentonite were added to the raw materials at a particular proportion. The effects of the B_2O_3 addition on the green strength, the roasting process, phase changes, and microstructures of the pellets, as well as the strength of the roasted pellets, were studied with an experimental device. The results can provide theoretical guidance for the possible use of boron minerals in the production of pellets and a new solution for the recycling of boron solid waste.

2. Experiment

2.1. Raw Materials. The iron ore used in this study is vanadium-titanium concentrates obtained from southwest of China. This was used together with bentonite and B_2O_3 (≥98%). The chemical composition of the sintering raw materials is shown in Tables 1 and 2. The B_2O_3 was added in increments of 0.20% yielding 11 experiments with B_2O_3 ratio in the range 0–2%.

2.2. Methods

2.2.1. Experimental Process of Pellets. Pellets were made on a disc pelletizer following the experimental batching scheme. The pellets experiment process is shown in Figure 1. The mixture (4 kg) of VTM concentrates, bentonite, and B_2O_3 were weighed, mixed thoroughly at appropriate moisture, and placed on disc pelletizer ($d = 1$ m). The disc firstly rotated at 22 r/min for 2 min to create small pellets, followed by 10 min of rotation for creating the finished pellets, and 3 min more for compaction. After the pelletizing was completed, the drop strength of the green pellets was checked. A total of 20 green pellets were dropped from 50 cm on the ground one by one. The drop test of green pellets was repeated for each pellet until cracks appear, and the number of drops was recorded. The recorded values are used to calculate an average value for each batch of experiments. Next, preheating and roasting were conducted using three-tube heating furnaces; the green pellet quickly bursts if they are not dried and preheated properly. The first tube was used to preheat the sample to 200°C, and the temperature was gradually increased from 200°C to 600°C in the second tube. Then, the preheated and dried pellets were collected and placed into the muffle furnace where they were heated to 900°C. The temperature was gradually increased to 1200°C in 30 min. The roasted pellets after cooling were used for analysis.

Table 1: Chemical composition of bentonite (wt%).

Name	SiO_2	Al_2O_3	CaO	P	MgO	Fe_2O_3	K_2O	Na_2O	MnO
Bentonite	61.68	13.80	2.83	0.03	5.10	2.41	0.06	<0.01	0.01

Table 2: Chemical composition of VTM concentrates for the experiments (wt%).

Name	TFe	SiO_2	CaO	Al_2O_3	MgO	TiO_2	MnO	FeO	P	S
VTM	55.78	4.33	0.69	3.86	2.78	9.08	0.36	30.50	0.09	0.54

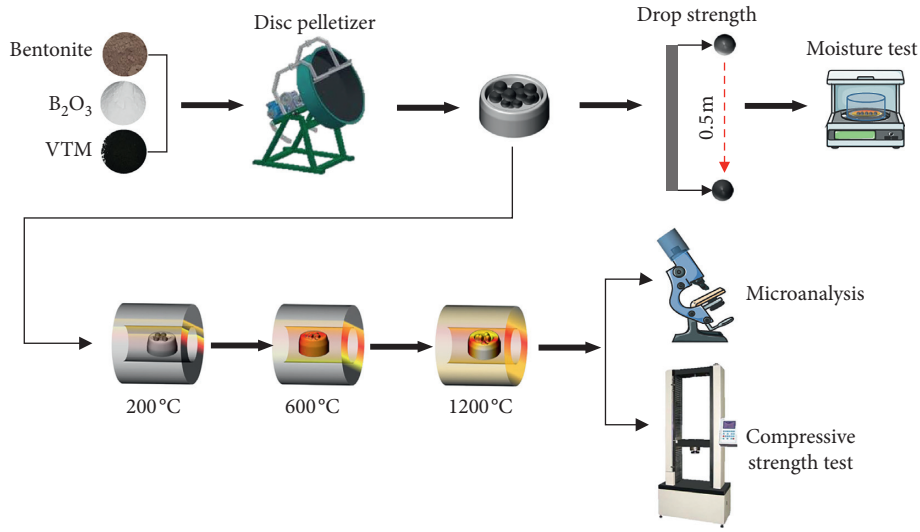

Figure 1: Flowchart of pellets experiment.

2.2.2. Pores Area Ratio and Fracture Toughness. The mineral phase of roasted pellets was studied by microscope and image analysis software to estimate the pore area ratio. HV-1000B Vickers hardness tester was used to conduct five groups of microhardness experiments on the roasted pellets, and the average values of the results were taken. The Vickers rhombic pressure cone was selected for the analysis. The objective lens of the Vickers hardness tester is ×40, the numerical aperture is 0.85, and the Vickers diamond cone is selected. The test load was 300 g and the time of action of the pressure cone was 10 s. Ten randomly measured points on the microscopic surface of each pellet were selected for the microhardness test and average value were taken after measuring the points. The fracture toughness results calculated by the microhardness test are shown in Figure 2. The elasticity modulus E and fracture toughness KIC of the roasted pellets were determined by

$$E = 0.45\frac{H}{1-n}, \quad (1)$$

$$\mathrm{KIC} = \delta\sqrt{\frac{E}{H}}\frac{P}{c^{3/2}}, \quad (2)$$

where H is the microhardness (GN/m^2), n is the ratio of the short diagonal to the long diagonal of the indentation, P is the pressure load, c is the average length of the diagonal of the indentation crack, and the constant δ is 0.0153. The modulus of elasticity E is 174.48 GPa [17].

2.2.3. High-Temperature Metallurgical Performance. According to the national standards of the People's Republic of China (GB/T 13241-2017 Iron Ores-Determination of Reducibility) [18], pellets of the size 10–12.5 mm were placed in the fixed chamber and reduced at 900°C. The reducing gas was composed of 70% N_2 and 30% CO. The reduction degree of the pellets was measured by the weight-loss method.

According to the national standards of the People's Republic of China (GB/T 13242-2017 Iron Ores-Low-Temperature Disintegration Test-method Using Cold Tumbling after Static Reduction) [19], the gas included CO, N_2, and CO_2 in the (volume) proportions 20:60:20 in the low-temperature reduction pulverization experiment. The gas ratios were allowed to fluctuate ±0.5%. During the experiment, the flow rate of the reducing gas mixture should be maintained at 15 L/min, allowing for fluctuations of ±0.5 L/min. The reduction temperature was 500°C. After the reduction for 60 min, drum sieve experiments were performed. The ratio of the sample (>3.15 mm) weight to the original sample weight $R_{+3.15}$ is called the low-temperature reduction pulverization index.

According to the national standards of the People's Republic of China (GB/T 13240-91 Iron Ore Pellets-Method for Measuring Relative Free Swelling Index) [20], reduction swelling experiment process was shown in Figure 3. A total of 18 pellets of the size 10–12.5 mm were selected. The reaction tube where the samples were placed had a diameter of 75 mm and a height of 800 mm. The pellets samples were firstly heated

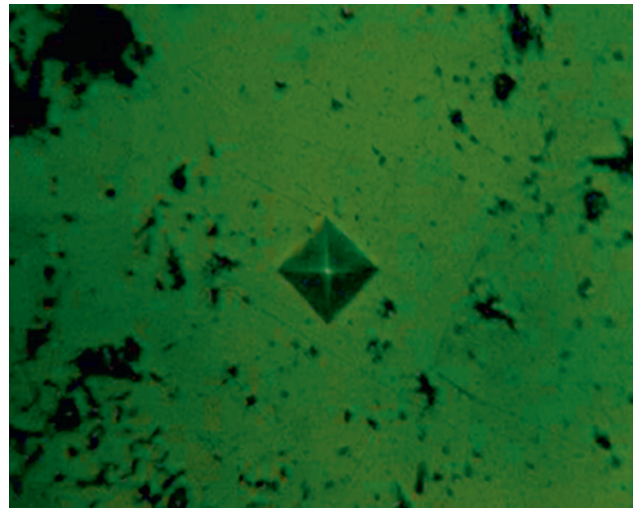

Figure 2: Microhardness indentation of pellet.

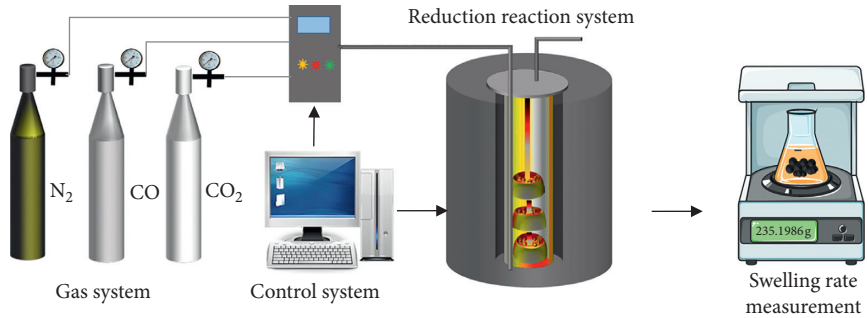

Figure 3: Flowchart of pellets reduction swelling experiment.

(at a rate $\leq 10^{\circ}$C/min), and when the temperature reached 200°C, 5 L/min of N_2 was passed into the reduction tube and the heating continued. When pellets samples reached 900°C, the flow rate of inert reducing gas (30% CO and 70% N_2) was increased to 15 L/min for 30 min. After the reduction, the volume of pellets was measured by the water immersion method. The ratio of the volume increase of the pellets before and after reduction to the original volume is recorded as the pellets swelling index. The temperature and atmosphere of this experiment were set to resemble the conditions of the upper and middle of the blast furnace. The reduction swelling index of pellets is an indicator of the volume expansion of pellets in the upper part of the blast furnace.

3. Results and Discussion

3.1. Changes in the Strength of Vanadium-Titanium Pellets under Different B_2O_3 Addition. The main mechanical properties of pellets are presented in Table 3. Drop-strength tests of green pellets with the addition of B_2O_3 in different proportions are shown in Figure 4. The compressive strength test of green pellets uses pressure testing machine QTG-1, where the maximum force is 500 N. It is seen that the vanadium-titanium pellets without added B_2O_3 show the highest drop strength, with an average number of drops of 15.40 until cracks are detected. With the increase of B_2O_3 amount in the mixture, the drop strength of pellets gradually decreases down to 4.10 for the green pellets with 2.0% added B_2O_3. Many factors can affect the quality of green pellets, such as the moisture of the balls, the particle size composition and surface characteristics of the raw materials, and the types of additives. The B_2O_3 reagent particles were added to the vanadium-titanium pellets as raw material. This may weaken the capillary bond between iron ore powder particles, then decreasing the drop strength of the green pellets.

The compressive strength of the vanadium-titanium roasted pellets without the addition of B_2O_3 was 1100 N at 1200°C. The compressive strength test of roasted pellets used pressure testing machine WDS-10QT; With a maximum force is 10000 N. When 0.6% of B_2O_3 was added (lower panel of Figure 4), the compressive strength of the pellets had decreased to the smallest value of all tested samples, about 850 N. When the blending amount of B_2O_3 was increased to 1.2–1.4%, the compressive strength increased to 1500–1700 N, but the addition of B_2O_3 above 1.4% yielded a decrease in the compressive strength pellets.

Thus, an appropriate amount of added B_2O_3 may improve the compressive strength of vanadium-titanium pellets. Since the B_2O_3 has a low melting point, it can form low-melting binding phases in the pellets which increases the

Table 3: Main mechanical properties of pellets (wt%).

B_2O_3% (wt%)	Drop strength of green pellets (T/P)	Compressive strength of green pellets (N)	Compressive strength of roasted pellets (N)
0	15.0	7.2	1087
0.2	7.5	7.2	1136
0.4	9.8	7.1	1033
0.6	5.5	7.5	849
0.8	5.6	7.0	903
1.0	6.4	4.7	1302
1.2	4.8	5.6	1501
1.4	5.6	5.7	1701
1.6	5.8	5.3	1095
1.8	4.5	5.5	1181
2.0	4.1	7.2	1045

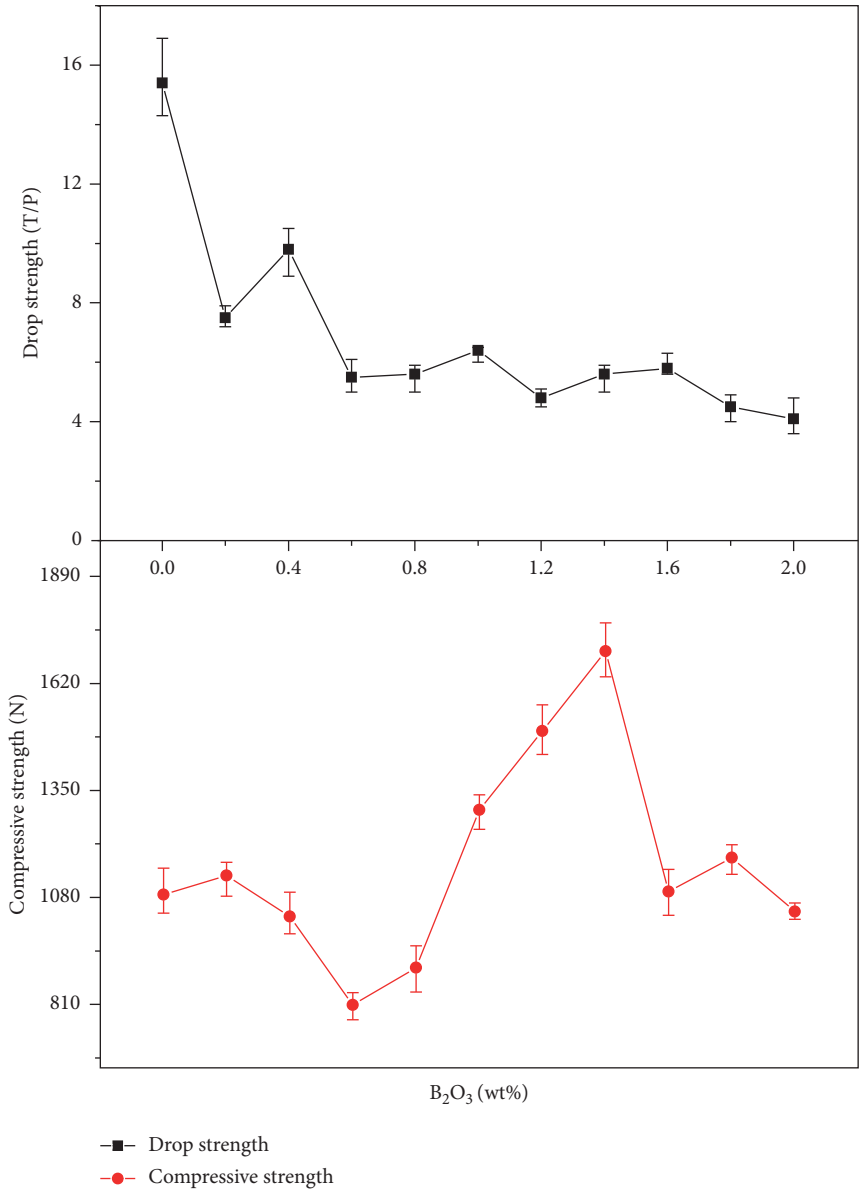

Figure 4: Drop strength and compressive strength of vanadium-titanium pellets with B_2O_3 addition.

strength of the pellets. The pellets bonding force produced by B_2O_3 can be complementary to the oxidative recrystallization and bonding force of Fe_3O_4. However, if more B_2O_3 was added (than the optimal amount), the binder phases become excessive. This prevents the direct connection of solid-phase particles and also the permeation of the liquid phase along

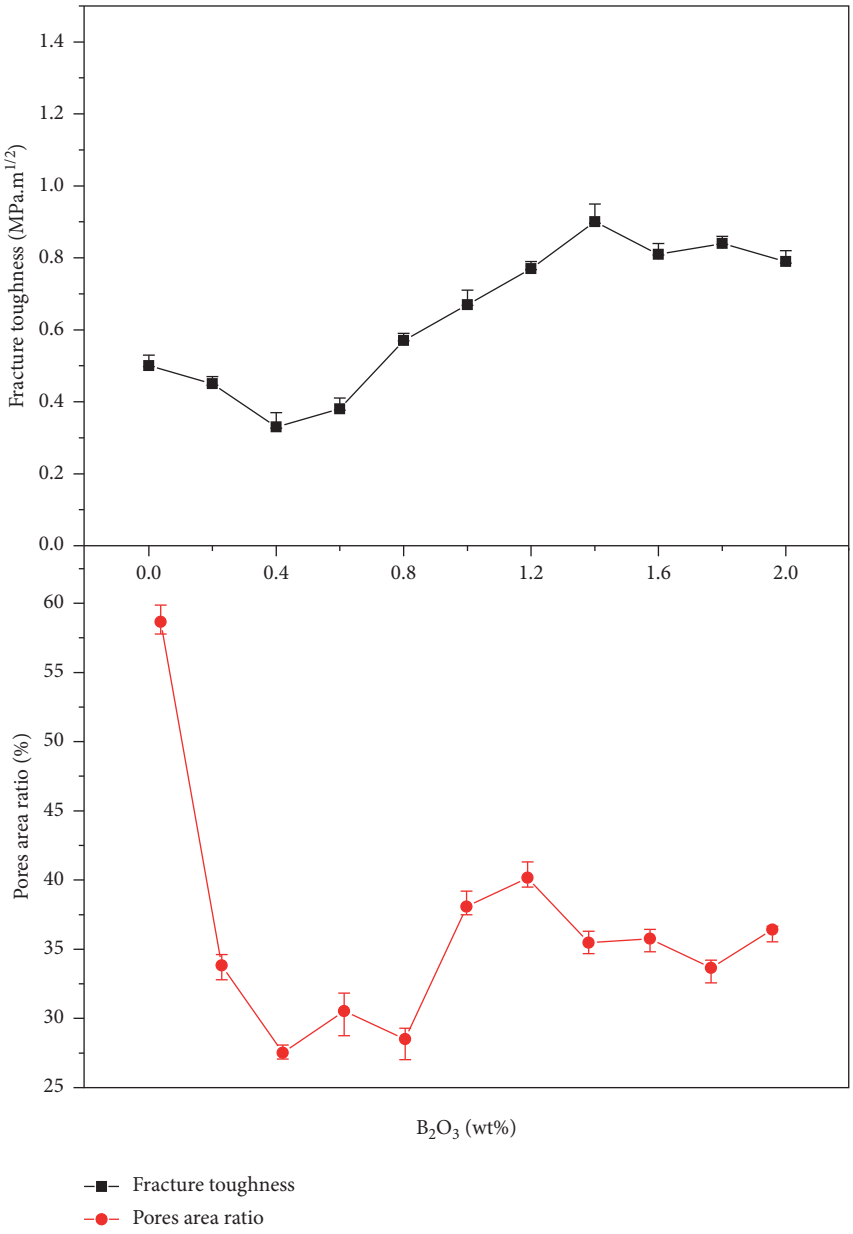

Figure 5: Pores area ratio and average fracture toughness of pellets with different B_2O_3 addition.

the grain boundaries. As a result, the consolidation of the large crystal clusters is weakened. The glass phase is also brittle, which reduces the strength of the pellets.

With the addition of B_2O_3, the area of the pores of the pellets has decreased (Figure 5) significantly, which means that the internal structure of the pellets has become dense, so the compressive strength of the pellets has increased. When the amount of added B_2O_3 is increased, pores formed by internal liquid phase cooling shrinkage are increased, as shown in Figure 6. These changes affect the strength of the pellets. The fracture toughness of pellets was slightly improved with the addition of boron oxide (Figure 2), but only in a very small range; and it had little effect on the structural strength of the pellets.

3.2. Changes in Microstructure of VTM Pellets with Different B_2O_3 Addition. For the pellets used in the blast furnace, the reducing agent (CO, H_2, and carbon) makes them undergo a stepwise reduction reaction, finally yielding metallic iron. During this reduction process, many complex physical and chemical changes occur in the pellets, including an increase of the volume of the pellets. High-temperature reduction properties of pellets with different B_2O_3 addition are shown in Figure 7.

The reasons for the volume expansion of different pellets during the reduction process depend on differences in mineral and chemical composition and internal structure. Along with the addition of B_2O_3 to the raw material, the reduction swelling rate is gradually decreased. The reason may be that the B_2O_3 activates the consolidation reaction

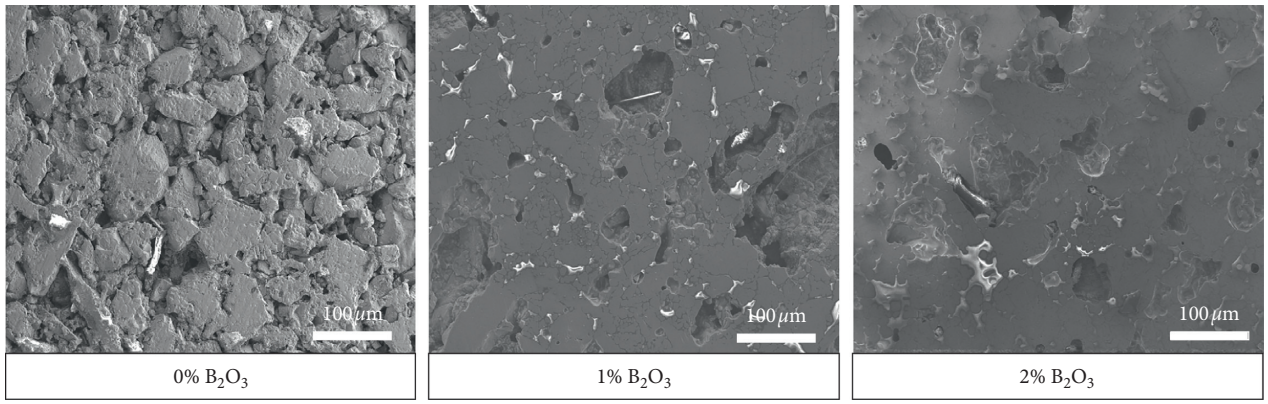

Figure 6: SEM images of pellets with different B_2O_3 addition.

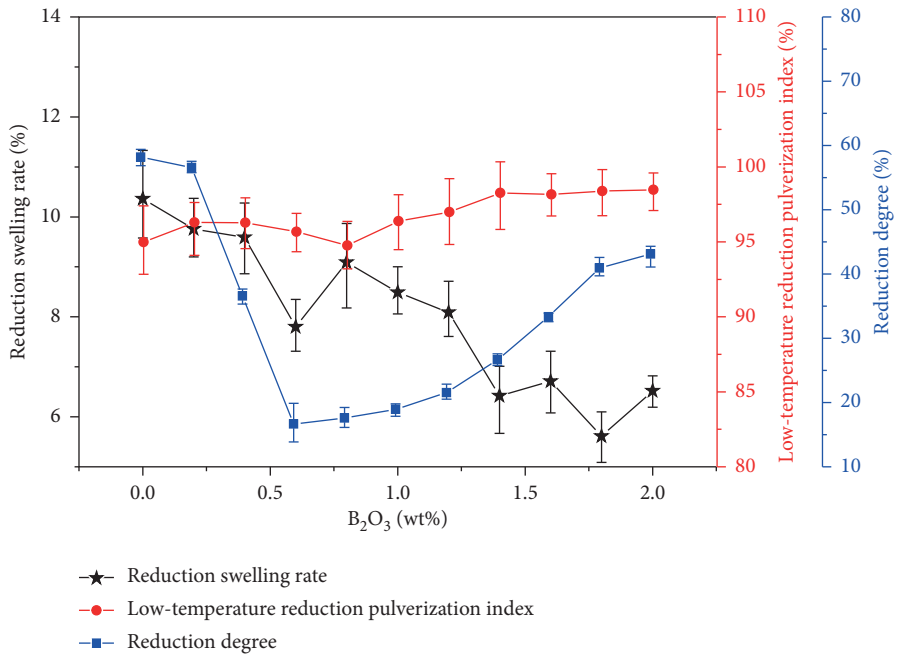

Figure 7: Metallurgical properties of pellets with different B_2O_3 addition.

during the roasting process. This also can strengthen the intermolecular forces, which suppresses volume changes of the pellets.

Possible chemical reactions by which B_2O_3 reacts with some oxides in VTM are listed in Table 4. Five main subsystems can be seen: B_2O_3-CaO, B_2O_3-CaO-SiO_2, B_2O_3-MgO, B_2O_3-Al_2O_3, and B_2O_3-CaO-Al_2O_3. Thermodynamic analysis of the five reaction systems was performed. The changes of the Gibbs free energy for the reactions of B_2O_3 and the oxide at different temperatures are depicted in Figure 8. Among the components studied, $CaO{\cdot}2B_2O_3$, $CaO{\cdot}B_2O_3$, $CaO{\cdot}B_2O_3{\cdot}2SiO_2$, $2CaO{\cdot}B_2O_3{\cdot}SiO_2$, $MgO{\cdot}2B_2O_3$, $2Al_2O_3{\cdot}B_2O_3$, $CaO{\cdot}B_2O_3{\cdot}Al_2O_3$, and $2CaO{\cdot}B_2O_3{\cdot}Al_2O_3$ have melting points below 1200°C. The lowest melting point, about 900°C, is observed for $2CaO{\cdot}B_2O_3{\cdot}SiO_2$. These low-melting intermediates enable the formation of primary liquid phases during the sintering process [16]. It also leads to the increase of the amount of liquid phase of the pellets as the amount of the added B_2O_3 increases, which is consistent with the results of thermodynamic calculations by FactSage 7.1, as shown in Figure 8.

Adding B_2O_3 promotes the rearrangement of the particles of the pellets during the roasting process. As a result, the compactness of the particles is increased. Adding a small amount of B_2O_3 can promote the recrystallization development of hematite, increase the liquid phase formation, and reduce the area of the pores. As the amount gradually increases, large-diameter pores appear in the pellets. The reason is the liquid phase, the appearance of which is promoted by B_2O_3 addition, and that the pellets shrink during the cooling process, which is illustrated in Figure 9. The B_2O_3 can also reduce the activation energy of pellets thus increasing the rate of the chemical reactions. Furthermore,

Table 4: Main reactions of B_2O_3 and oxides.

Number	Chemical reaction equation
(3-1)	$2Al_2O_3 + B_2O_3 = 2Al_2O_3{\cdot}B_2O_3$
(3-2)	$MgO + 2B_2O_3 = MgO{\cdot}2B_2O_3$
(3-3)	$9Al_2O_3 + 2B_2O_3 = 9Al_2O_3{\cdot}2B_2O_3$
(3-4)	$2MgO + B_2O_3 = 2MgO{\cdot}B_2O_3$
(3-5)	$CaO + B_2O_3 = CaO{\cdot}B_2O_3$
(3-6)	$CaO + B_2O_3 + 2SiO_2 = CaO{\cdot}B_2O_3{\cdot}2SiO_2$
(3-7)	$CaO + Al_2O_3 + B_2O_3 = CaO{\cdot}B_2O_3{\cdot}Al_2O_3$
(3-8)	$3MgO + B_2O_3 = 3MgO{\cdot}B_2O_3$
(3-9)	$CaO + 2B_2O_3 = CaO{\cdot}2B_2O_3$
(3-10)	$2CaO + B_2O_3 = 2CaO{\cdot}B_2O_3$
(3-11)	$2CaO + B_2O_3 + SiO_2 = 2CaO{\cdot}B_2O_3{\cdot}SiO_2$
(3-12)	$2CaO + Al_2O_3 + B_2O_3 = 2CaO{\cdot}B_2O_3{\cdot}Al_2O_3$
(3-13)	$3CaO + B_2O_3 = 3CaO{\cdot}B_2O_3$
(3-14)	$11CaO + B_2O_3 + 4SiO_2 = 11CaO{\cdot}B_2O_3{\cdot}4SiO_2$

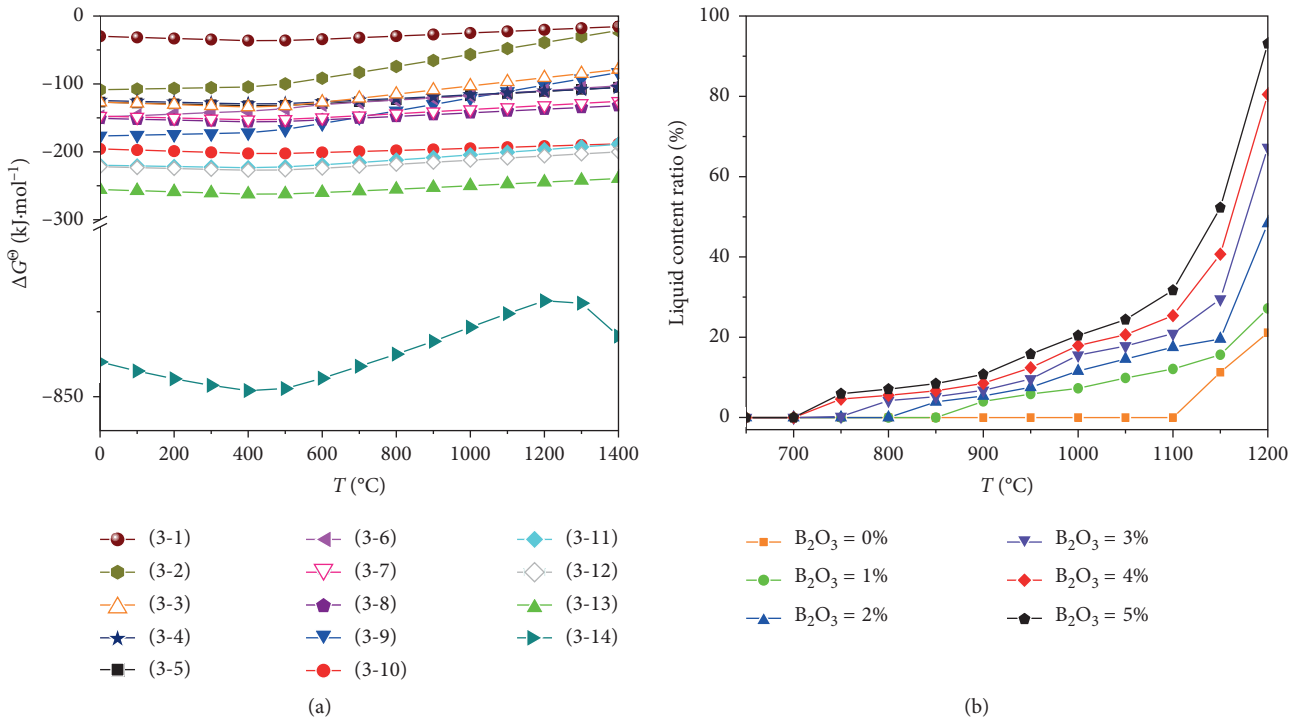

Figure 8: Gibbs free energy of B_2O_3 and oxides and liquid content of the pellets at different B_2O_3 addition.

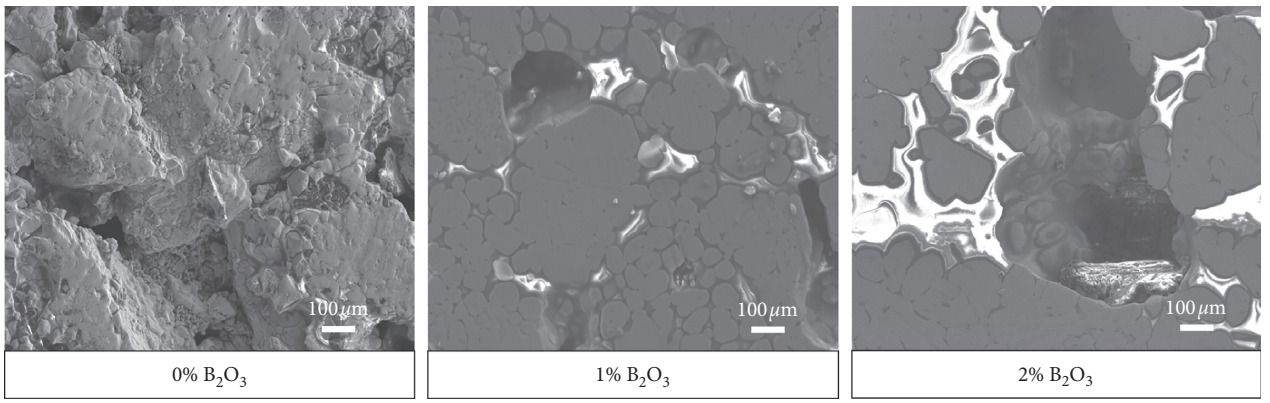

Figure 9: SEM images of pellets with different B_2O_3 addition.

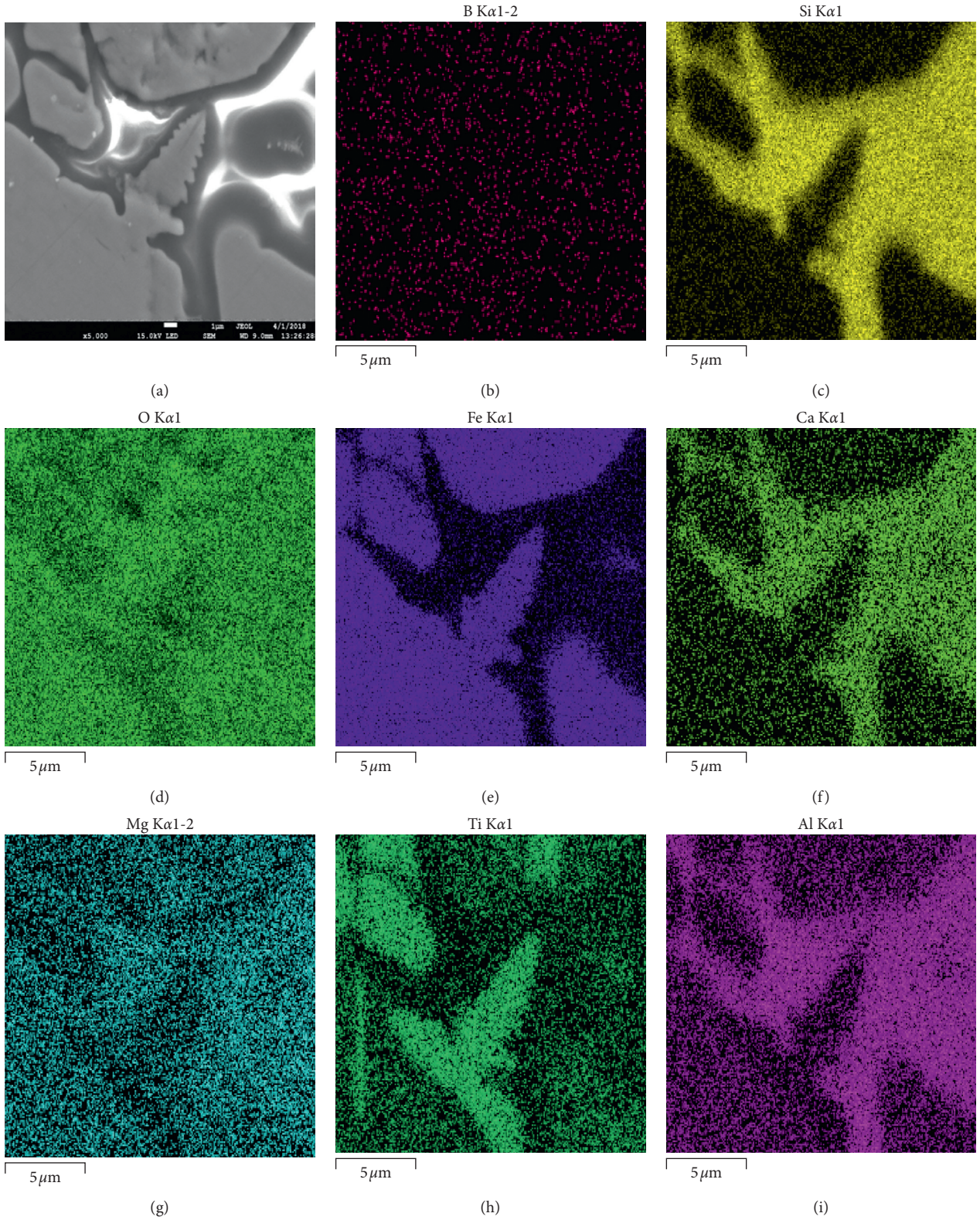

Figure 10: SEM-EDS images of pellet at 2% B_2O_3 addition.

after adding B_2O_3, the reduction pulverization rate for small particles, especially those below 3.15 mm, decreases, since the mix is prone to produce liquid phases, which increases the compactness inside the pellets.

When more than 2% B_2O_3 was added to the raw material mix, the pores in the pellets gradually disappeared. The shape of the hematite was changed from bone-like to tightly connected granular, as shown in Figure 10. During the roasting of the pellets, the compressive strength is determined by the microstructure inside the pellets. Inside the vanadium-titanium pellets with B_2O_3, the main lattice was distorted and activated since B^{3+} has a small radius with strong polarization ability. This promotes the recrystallization of hematite into pieces and its growth into blocks [17, 18]. The blocks were filled with a cohesive silicate phase. With the increase of the B_2O_3 addition, the binder phase in the pellets was increased and ferrite was replaced by silicate. The melting point of silicate is low. As a result, the roasting temperature of boron-added pellets decreased. The B_2O_3 could be uniformly dispersed among the hematite grains. Thus, it strengthened the bonding effect of the pellets and improved the strength of the pellets. However, the positive effect was only achieved within a specific range of added B_2O_3. When too much B_2O_3 was added, the bonding phase was increased, which negatively affects the solid-phase contact of Fe_2O_3 and reduces the strength.

4. Conclusions

The effects of adding B_2O_3 to the raw material mix before pelletizing on the drop strength, compressive strength, pores area ratio, high-temperature reduction properties, and microstructure of vanadium-titanium magnetite (VTM) concentrates pellets have been studied through pelletizing and roasting experiments. The following main conclusions can be drawn based on the findings:

(1) Although the addition of the B_2O_3 reagent did not increase the drop strength of the green pellets, the compressive strength and fracture toughness of the roasted pellets were improved to some extent with the increase of B_2O_3 addition. Under the conditions of the experiments undertaken in this work, the optimal amount of B_2O_3 added was found to be between 1.4% and 1.6%.

(2) The reduction degree of VTM pellets firstly decreased and then increased with the addition of B_2O_3. The addition can increase the amount of liquid phase in the pellets, enhance the compactness, and reduce the reduction pulverization if the added amount is low. Also, the reduction expansion performance of the VTM pellets may improve by adding B_2O_3, but gradually yielding holes with growing size in the pellets, which reduces the strength of pellets.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

Authors' Contributions

Hao Liu, Ke Zhang, and Xiaoyan Xiang contributed to performing the experiments, material characterization, data analysis, and paper writing; Henrik Saxén, Weiqiang Liu, and Yuelin Qin revised the paper and refined the language; and Hao Liu and Yuelin Qin contributed to the design of the experiment.

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