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Effect of MgO content in sinter on the softening–melting behavior of mixed burden made from chromium-bearing vanadium–titanium magnetite

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Abstract: The effect of sinter with different MgO contents on the softening–melting behavior of mixed burden made from chromium-bearing vanadium–titanium magnetite was investigated. The results show that with increasing MgO content in the sinter, the softening interval and melting interval increased and the location of the cohesive zone shifted downward slightly and became moderately thicker. The softening–melting characteristic value was less pronounced when the MgO content in the sinter was 2.98wt%–3.40wt%. Increasing MgO content in the sinter reduced the content and recovery of V and Cr in the dripped iron. In addition, greater MgO contents in the sinter resulted in the generation of greater amounts of high-melting-point components, which adversely affected the permeability of the mixed burden. When the softening–melting behavior of the mixed burden and the recovery of valuable elements were taken into account, proper MgO contents in the sinter and slag ranged from 2.98wt% to 3.40wt% and from 11.46wt% to 12.72wt%, respectively, for the smelting of burden made from chromium-bearing vanadium–titanium magnetite in a blast furnace.

Keywords: ironmaking; magnetite; magnesia; softening; melting behavior; sinter; blast furnace practice

1. Introduction

Vanadium–titanium magnetite, a mineral resource rich in some valuable metals such as Fe, V, Ti, Cr, etc. has a significant utilization value [1–3]. This ore is mainly distributed in Russia, China, South Africa, Australia, New Zealand, Canada, and India, among other locations [4–6]. The blast furnace (BF) process is the main process for extracting Fe and V from vanadium–titanium magnetite [7]. Although great progress has been made in smelting burden made from vanadium–titanium magnetite in BFs in recent years, some problems remain to be solved; for example, the softening–melting behavior of mixed burden is not optimal, the recovery of vanadium is low, and the slag is viscous.

Under such circumstances, the addition MgO is recommended to obtain a fluid slag and an optimal burden structure. BF slag with an appropriate MgO content could exhibit good fluidity and desulfurization [8–10]. The mode of its addition to the BF is changed from raw flux to sinter. The effect of MgO content on the sintering process, sintered ore, and BF slag are fairly well established [11–13]; however, investigations of the effect of MgO content in sinter on the BF mixed burden, especially on the softening–melting behavior of BF mixed burden made from vanadium–titanium magnetite, are quite limited. Because of the interaction among different burden materials [14–15], the reduction behavior of BF mixed burden could more closely resemble that of actual production. Therefore, both the effect of MgO in sinter on the softening–melting behavior of BF mixed burden made from vanadium–titanium magnetite and the mechanism of its effect have been investigated to improve BF operation.

The chromium-bearing vanadium–titanium magnetite (Cr–V–Ti magnetite) used in this work, because of its complicated chemical composition and mineral phase structure, is an important complex mineral resource with a high comprehensive utilization value [16–18]. The literature contains few reports on the effect of MgO flux on the agglomeration of Cr–V–Ti magnetite [19–20]. Thus, we here investigate

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the effect of sinter with different MgO contents on the softening–melting behavior of the mixed burden by simulating the heating rule and atmosphere of BF. Furthermore, the effect of MgO in sinter on the content and recovery of V and Cr in dripped iron is discussed. We further analyzed the mechanism of the effect of MgO by performing chemical and X-ray diffraction analyses of the residual slag and dripped slag of mixed burden. The results provide a theoretical basis and technical support for the rational use of MgO in smelting Cr–V–Ti magnetite, improving BF operation and increasing the recovery of V and Cr.

2. Experimental

2.1. Experimental materials

Four kinds of Cr–V–Ti magnetite sinter and one type of Cr–V–Ti magnetite pellets were used in the tests. The chemical compositions of the sinter and pellets are listed in Tables 1 and 2, respectively. As evident from the results in Table 1, the MgO content in the sinter samples varied from 2.75wt% to 3.56wt%, whereas the basicity ($R = w(\text{CaO})/w(\text{SiO}_2)$) of all of the sinter samples was consistent at approximately 1.90.

Table 1. Chemical composition of Cr–V–Ti magnetite sinter wt%

Elements	MgO	TFe	SiO_2	CaO	Al_2O_3	V_2O_5	TiO_2	Cr_2O_3
SA	2.75	53.46	5.40	10.26	2.51	0.368	1.68	0.240
SB	2.98	53.14	5.42	10.28	2.52	0.366	1.67	0.260
SC	3.40	52.86	5.44	10.33	2.52	0.364	1.66	0.262
SD	3.56	52.58	5.45	10.38	2.53	0.362	1.65	0.253

Table 2. Chemical composition of Cr–V–Ti magnetite pellets wt%

Elements	TFe	SiO_2	CaO	Al_2O_3	MgO	V_2O_5	TiO_2	Cr_2O_3
PA	59.43	5.64	1.75	1.39	1.57	0.373	2.53	0.22

2.2. Experimental method

Table 3 gives the structure of the mixed burden used in the tests. The results in Table 3 indicate that the ratio between sinter and pellets in the mixed burden samples was consistently 66:34. The softening–melting behavior of four kinds of mixed burden was investigated. In the four kinds of mixed burden, the MgO content in the sinter, from SA to SD, increased from 2.75wt% to 3.56wt%. We investigated the effect of MgO content in the sinter on the softening–melting behavior of Cr–V–Ti magnetite mixed burden as well as the migration of V and Cr using softening–melting experiments.

Table 3. Structure of the mixed burden used in the tests

No.	Burden structure	Sinter in mixed burden /wt%	Pellets in mixed burden / wt%
1#	SA + PA	66	34
2#	SB + PA	66	34
3#	SC + PA	66	34
4#	SD + PA	66	34

The experiments were carried out using softening–melting equipment. A schematic of the softening–melting equipment is illustrated in Fig. 1. A ϕ75 mm (inner diameter) graphite crucible with ϕ8 mm dripping holes in its bottom was used in the experiments. The graphite crucible was charged with 500 g of iron-bearing material to a height of approximately 60 mm. Coke was laid underneath and on the top of the iron ore samples to ensure molten materials and gas flow could easily pass through the bed. The particle sizes of the iron-bearing materials and coke were 10–12.5 mm and 8–10 mm, respectively. To simulate the heating and reduction process of the iron-bearing materials in a BF, the experimental conditions were fixed as reported in Table 4. After the experiments, the dripped substance and residual substance in the crucible were collected for further analysis.

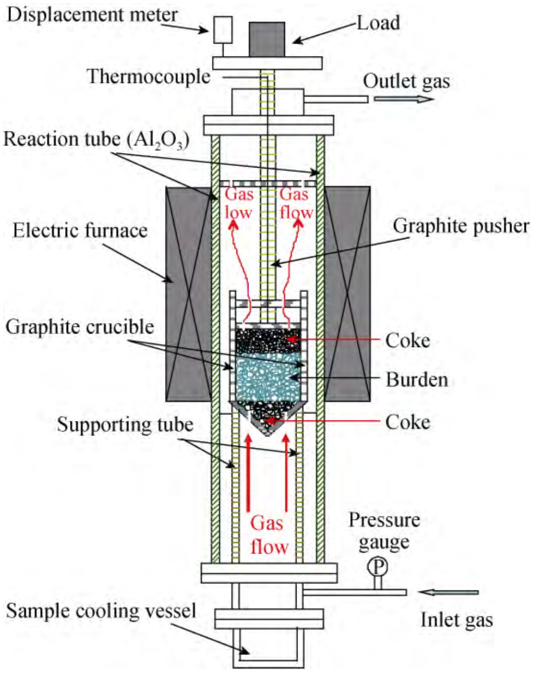

Fig. 1. Measurement system for softening–melting behavior of burden.

The softening–melting behavior of iron ore plays a decisive role in the location, shape, and thickness of the cohesive zone, although the BF operating parameters also have some effect on the cohesive zone [21–22]. A typical set of softening–melting behavior test results are shown in Fig. 2, in which the temperature of the burden, shrinkage ratio, pressure drop, and the dripping mass were measured to evaluate the softening–melting behavior of iron ore samples [23–25]. Observing the behavior directly is difficult because the interior of the softening–melting equipment is not viewable. Thus, some indexes were used to evaluate the softening–melting behavior of the iron ores. The burden temperature when the shrinkage ratio reaches 4%, T_4, is defined as the temperature at which softening starts; T_{40} is defined as the softening end temperature when the shrinkage ratio reaches 40%; the melting start temperature, T_S, is the temperature at which the burden shrinking percentage obviously increases or at which the pressure drop begins to dramatically increase; and T_D is the temperature at which molten materials start to drip. The temperature interval T_{40}–T_4 represents the softening interval of the iron ore samples, and the temperature interval T_D–T_S reflects the melting interval or cohesive zone.

Table 4. Experimental conditions for softening–melting behavior tests

Heating time / min	40	50	40	>120
Load / (kg·cm^{-2})	0.5	0.5	1.0	1.0
Gas composition and flow rate	N_2: 100%, 3 L/min	N_2: 9 L/min CO: 3.9 L/min CO_2: 2.1 L/min	N_2: 10.5 L/min CO: 4.5 L/min	N_2: 10.5 L/min CO: 4.5 L/min
Heating rate and target temperature	10°C/min to 900°C	10°C/min to 900°C	3°C / min, to 1020°C	5°C / min, to dripping

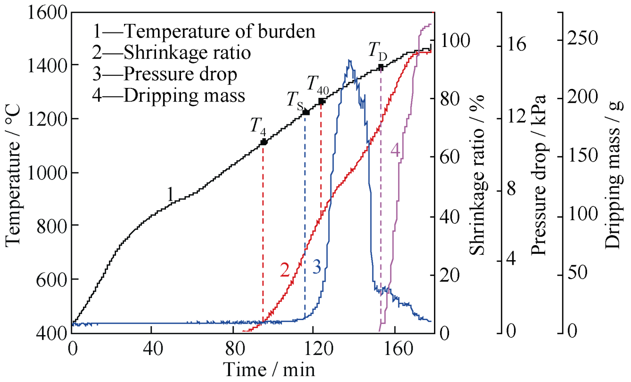

Fig. 2. Data curve showing softening–melting characteristics.

3. Results and Discussion

3.1. Softening behavior of the mixed burden

Fig. 3 shows the effect of the MgO content in the sinter on the softening behavior of the mixed burden. As shown in Fig. 3, with increasing MgO content in the sinter, the softening start temperature T_4 remains steady at 1104°C, whereas the softening end temperature T_{40} increases from 1252.3°C to 1261.7°C; the softening interval T_{40}–T_4 therefore increases from 142.5°C to 153.5°C. Thus, the softening behavior of the mixed burden slightly improved with increasing MgO content in the sinter in the experimental range for the burden made from Cr–V–Ti magnetite.

There are two reasons for the increase of the softening end temperature T_{40}. First, magnesium ferrite is generated when the MgO content in the sinter is increased. An increase in the magnesium ferrite content results in a lower calcium ferrite (SFCA). Both of these effects result in diminished reducibility and a lower shrinkage rate of the mixed burden. Second, the high-melting-point substance containing MgO increases in the primary slag phase with increasing MgO content in the sinter. On the basis of these two effects, the softening end temperature T_{40} is expected to increase with increasing MgO content in the sinter, which would facilitate the gas–solid reactions of sinter in the burden.

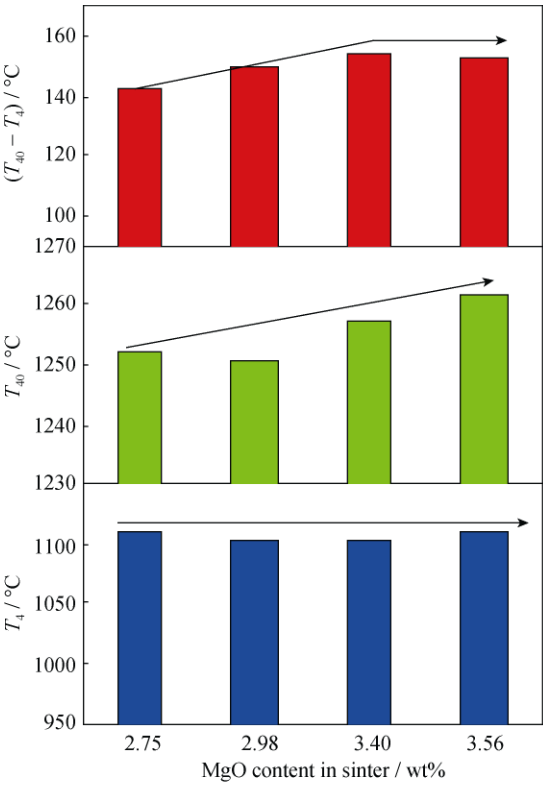

Fig. 3. Effect of MgO content in the sinter on the softening behavior of the mixed burden.

3.2. Melting behavior of the mixed burden

The effect of MgO content in the sinter on the melting behavior of the mixed burden is shown in Fig. 4. As evident in the figure, with increasing MgO content in the sinter, the melting start temperature T_S is increased from 1234.1°C to 1245.3°C and the dripping temperature T_D is increased from 1424.2°C to 1461.7°C. The melting interval (cohesive zone) T_D–T_S is widened from 190.1°C to 216.4°C. In addition, as shown in Fig. 5, the location of the cohesive zone moves down slightly and the cohesive zone becomes mildly thicker. Therefore, the melting behavior of the mixed burden is worsened to some extent with increasing MgO content in the sinter.

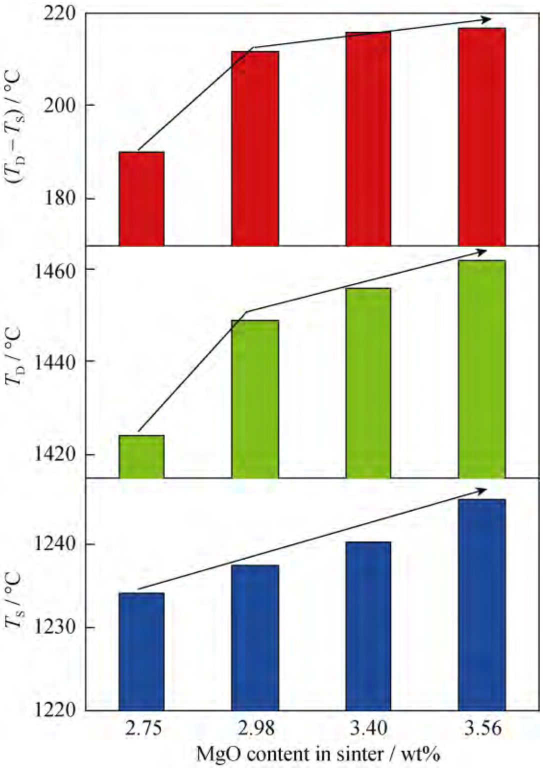

Fig. 4. Effect of MgO content in the sinter on the melting behavior of he mixed burden.

The generation of a high-melting-point substance containing MgO in the slag is the main reason for the increase of T_S and T_D, which can be explained on the basis of the slag phase diagram. Fig. 6 gives the CaO–SiO$_2$–MgO–Al$_2$O$_3$–TiO$_2$ isotherm-primary crystal region phase diagram, in which the mass fractions of Al$_2$O$_3$ and TiO$_2$ are 11.4% and 11.1%, respectively. The slag of the mixed burden is in the primary crystal region of merwinite ($Ca_3MgSi_2O_8$), close to the primary crystal region of periclase (MgO), where the isothermal lines are dense and the melting point of the slag phase is obviously increased with increasing MgO content in the sinter. Thus, the melting start temperature T_S and dripping temperature T_D increase to some extent.

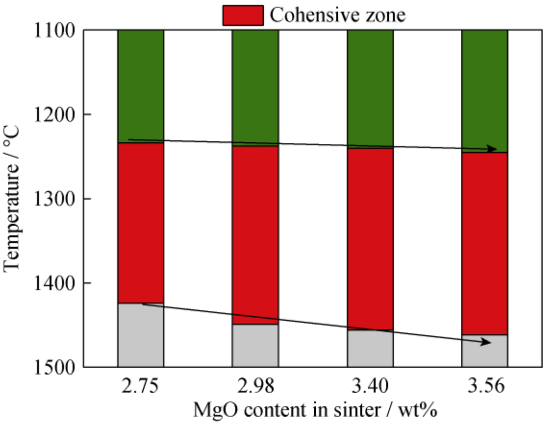

Fig. 5. Effect of MgO content in the sinter on the location of the cohesive zone.

3.3. Permeability of the mixed burden

To quantify the softening–melting behavior of the mixed burden, a softening–melting characteristic value (referred to as the *S*-value) is introduced. A smaller *S*-value indicates a better permeability of the mixed burden. The current *S*-value is defined as the integral of the pressure-drop function over the melting and dripping temperature interval, which represents the area below the pressure-drop curve. The formula for the *S*-value is shown as:

$$S = \int_{T_S}^{T_D} (P_m - \Delta P_S)\mathrm{d}T \qquad (1)$$

where P_m is the pressure drop at a certain temperature between T_S and T_D, and ΔP_S is the pressure drop at the melting start temperature T_S.

The effect of the MgO content in the sinter on the mixed burden permeability is shown in Fig. 7. This figure indicates that the *S*-value first decreased and then increased with increasing MgO content in the sinter, indicating that the permeability of the mixed burden first increased and then decreased. Therefore, the permeability of the mixed burden is better when the content of MgO in the sinter is between 2.98wt% and 3.40wt% because the liquidity of the slag phase is improved to a certain extent with increasing MgO content.

3.4. Dripping behavior of the mixed burden

Fig. 8 shows the effect of the MgO content in the sinter on the dripping behavior of the mixed burden. The dripping ratio decreased, whereas the dripping pressure-drop slightly changed with increasing MgO content in the sinter. The worsened reducibility of the mixed burden and the generation of a high-melting-point substance dispersed in the slag

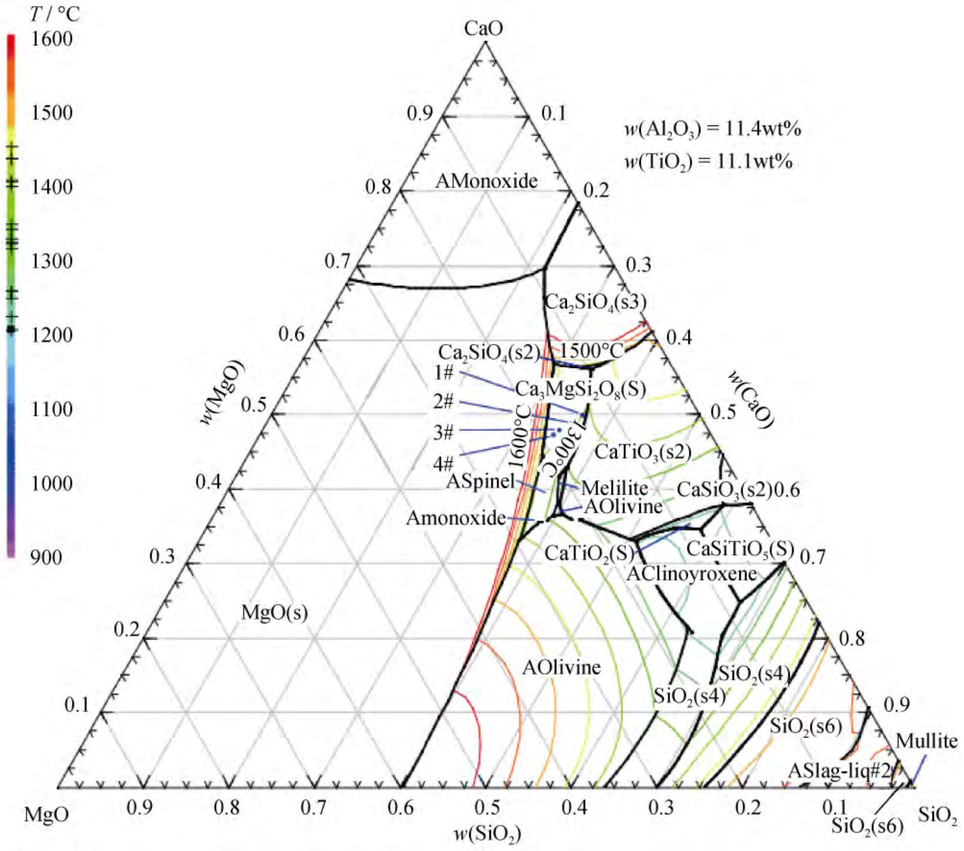

Fig. 6. CaO–SiO$_2$–MgO–Al$_2$O$_3$–TiO$_2$ isotherm-primary crystal region phase diagram.

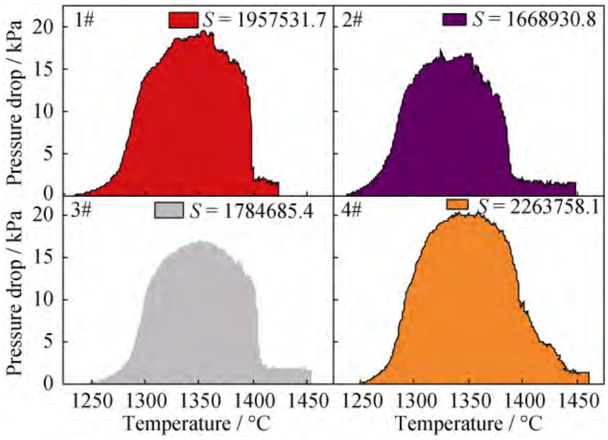

Fig. 7. Effect of MgO content in the sinter on the permeability of the mixed burden.

phase are the main reasons for the decrease of the dripping ratio. Given the permeability and the dripping behavior of the mixed burden, the proper MgO content in the sinter is suggested to be between 2.98wt% and 3.40wt%, whereas the proper content of MgO in the BF slag is 11.46wt%–12.72wt%.

3.5. Migration of V and Cr in initial iron and in slag

During the softening–melting–dripping process, some minerals containing V or Cr could be reduced and a certain amount of V and Cr would be reduced into molten iron and subsequently form dripped iron. As greater amounts of V and Cr are reduced, the amounts of V and Cr recovered from the iron ore increase.

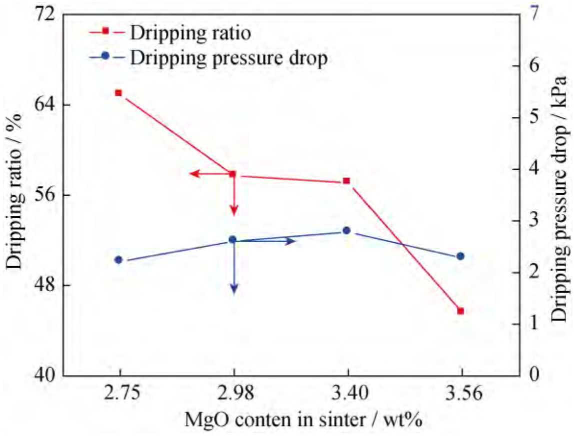

Fig. 8. Effect of MgO content in the sinter on the dripping behavior of the mixed burden.

The effects of MgO content in the sinter on the content and recovery ratio of V and Cr in the dripped iron are shown in Figs. 9 and 10, respectively. With increasing MgO content in the sinter, the content and recovery of V and Cr in the dripped iron decreased. The recovery of V and Cr decreased slightly when the MgO content in the sinter was less than 3.40wt%, whereas the recovery obviously decreased when the MgO content in the sinter was greater than 3.40wt%. When the MgO content in the sinter was 3.56wt%, the V and Cr contents in the dripped iron were 0.165wt% and 0.174wt%, respectively; the corresponding recoveries of V and Cr were 22.482% and 17.160%, which were the lowest observed values.

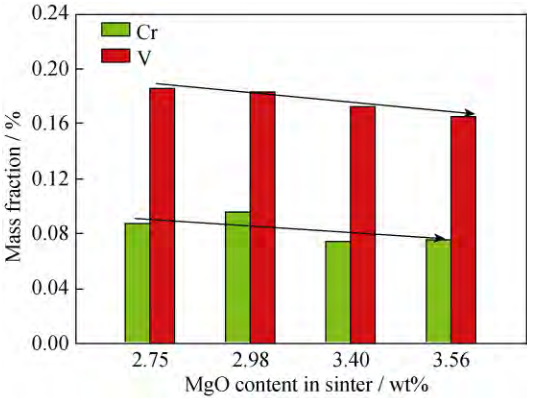

Fig. 9. Effect of MgO content in the sinter on the content of V and Cr in the dripped iron.

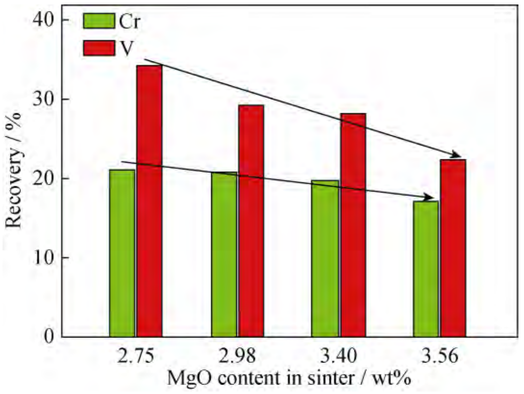

Fig. 10. Effect of MgO content in the sinter on the recovery ratio of V and Cr in the dripped iron.

Therefore, to improve the recovery of V and Cr for smelting Cr–V–Ti magnetite in a BF, the proper MgO content in the sinter is 3.40wt% or less. The reason for the lower recovery of V and Cr in the dripped iron is mainly the worsened reducibility of the mixed burden. Simultaneously, with increasing MgO content in the sinter, the dripped mass is lower and the recovery ratio of V and Cr are obviously decreased.

3.6. Mechanism of MgO function

In the reduction process, iron-bearing materials undergo reduction, softening, and melting stages, in sequence. Some molten materials are then dripped, forming the dripped substance, whereas the remainder of the materials in the crucible transform into a residual substance [26–27]. Fig. 11 shows photos of the dripped and residual substances in the tests. The dripped substances contain an iron phase and a relatively smaller amount of slag phase, whereas the dripped substances melt completely to form a sheet structure. In contrast, the residual substances are composed of iron mineral particles and relatively more slag mineral particles.

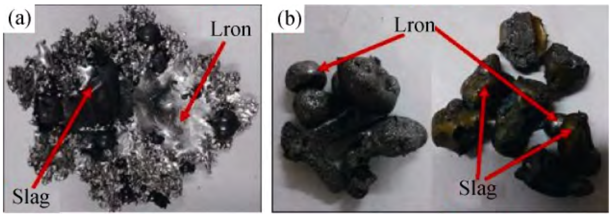

Fig. 11. Photos of the dripped (a) and residual substances (b).

To clarify the mechanism by which the MgO content in the sinter affects the softening–melting behavior of the mixed burden, we used chemical analysis and X-ray diffraction to analyze the residual slag and dripped slag of the mixed burden. The chemical compositions of the dripped slag and the residual slag are listed in Tables 5 and 6, respectively. As evident from the results in the tables, (a) the MgO content in the dripped slag has no direct relation with the initial MgO content in the sinter; greater MgO content in the sinter results in less MgO content in the dripped slag; (b) the MgO content in the residual slag is obviously increased with increasing MgO content in the sinter, and the MgO content in the residual slag is greater than that in the dripped slag.

Table 5. Chemical composition of the dripped slag wt%

No.	CaO	SiO_2	MgO	Al_2O_3	TiO_2
1#	31.44	29.95	13.29	14.59	10.72
2#	31.52	29.27	11.35	17.88	9.99
3#	32.73	29.43	8.78	17.83	10.23
4#	33.66	29.72	7.84	18.13	10.66

Table 6. Chemical composition of the residual slag wt%

No.	CaO	SiO_2	MgO	Al_2O_3	TiO_2
1#	31.04	32.79	13.14	12.57	10.47
2#	28.93	31.71	16.35	13.09	9.92
3#	26.21	27.97	23.27	12.31	10.23
4#	27.34	26.27	25.15	11.45	9.79

The X-ray diffraction results for the dripped slag and residual slag are shown in Figs. 12 and 13, respectively. The results indicate that (a) the main components of the dripped slag are melilite ($Ca_2(Al,Mg)[(Si,Al)SiO_7]$), perovskite

($CaTiO_3$), magnesium aluminate spinel ($MgAl_2O_4$), monticellite ($CaMgSiO_4$); in addition, with increasing MgO content in the sinter, the amount of perovskite tends to decrease, whereas the amounts of magnesium aluminate spinel and monticellite tend to increase; (b) the main components of the residual slag are melilite, perovskite, magnesium aluminate spinel, monticellite, diopside ($CaMgSi_2O_6$), and periclase (MgO); with increasing MgO content in the sinter, the amounts of perovskite, magnesium aluminate spinel, monticellite, diopside, and periclase tend to increase or appear; and (c) compared with the dripped slag, the residual slag contains two additional components: diopside and periclase.

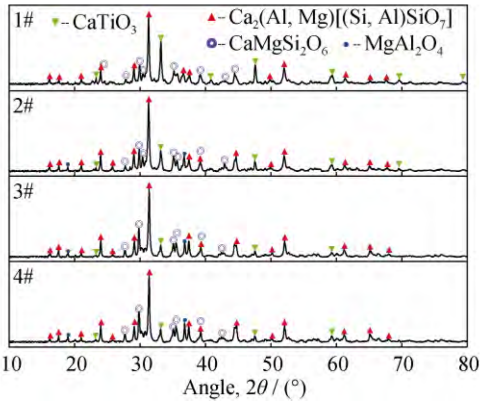

Fig. 12. X-ray diffraction patterns of the dripped slag.

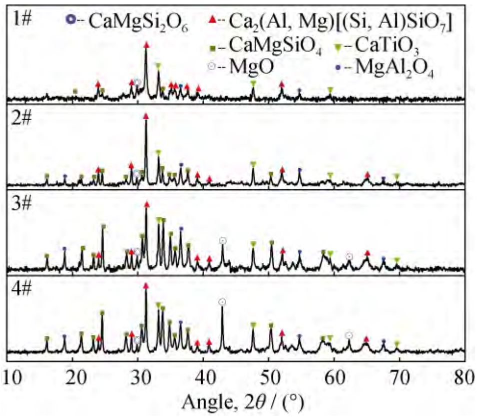

Fig. 13. X-ray diffraction patterns of the residual slag.

On the basis of the chemical analysis and X-ray diffraction results for the dripped and residual slag, a higher MgO content in the sinter results in the formation of greater amounts of the high-melting-point component. We deduce that the slag containing large concentrations of MgO cannot drip easily, resulting in an increase of the temperature range of the cohesive zone. The permeability of the BF mixed burden would be worsened, which is not favorable for BF operation. Thus, taking softening–melting behavior of the mixed burden and the recovery of valuable elements into account, we determined that the proper MgO content in the sinter is 2.98wt%–3.40wt%, whereas that in slag is 11.46wt%–12.72wt% for smelting mixed burden made from Cr–V–Ti magnetite in BF.

4. Conclusions

(1) With increasing MgO content in the sinter, the softening interval T_{40}–T_4 increases from 142.5°C to 153.5°C, which facilitates the gas–solid reaction for the burden made from Cr–V–Ti magnetite. The melting interval T_D–T_S is increased from 190.1°C to 216.4°C. The location of the cohesive zone is shifted down slightly, and the cohesive zone becomes moderately thicker. The softening–melting characteristic value is smaller when the content of MgO in the sinter is between 2.98wt% and 3.40wt%.

(2) With increasing MgO content in the sinter, the content and recovery of V and Cr in the dripped iron decrease. When the MgO content in the sinter is 3.56wt%, the content of V and Cr in the dripped iron are 0.165wt% and 0.174wt%, respectively, and the recoveries of V and Cr are 22.482wt% and 17.160wt%, respectively, which are all the lowest corresponding values obtained in tests performed in this work.

(3) A higher MgO content in the sinter results in the generation of more components with high melting points, which decreases the permeability of the BF mixed burden and is not favorable for BF operation.

(4) With the softening–melting behavior of the mixed burden and the yield of valuable elements taken into account, the recommended proper MgO contents in the sinter and slag are 2.98wt%–3.40wt% and 11.46wt%–12.72wt%, respectively, for smelting Cr–V–Ti magnetite in a BF.

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