Effect of Mineral Elements Migration on Softening–melting Properties of Ti–bearing High Basicity Sinter

Tingle LI,^{1,2)} Changyu SUN,^{1,2)*} Dong LAN,^{1,2)} Jian SONG,³⁾ Sunny SONG⁴⁾ and Qi WANG^{1,2)}

1) Key Laboratory of Chemical Metallurgy Engineering Liaoning Province, University of Science and Technology Liaoning, Anshan, Liaoning, 114051 China.

2) School of Materials and Metallurgy, University of Science and Technology Liaoning, Anshan, Liaoning, 114051 China.

3) Panzhihua Steel Vanadium Ironmaking Plant, Panzhihua, 617024 China.

4) Technical Marketing, Rio Tinto Iron Ore, Perth, 6000 West Australia.

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The mineral elements migration behaviours of Ti–bearing high basicity sinter during softening–melting process, as well as its effect on softening–melting properties, are studied using scanning electron microscope and X–ray diffractometer. We can conclude that both MgO and Al₂O₃ content increase while TiO₂ content increases first and decreases later overall in the slag phase from the original state to melting end. At softening stage, some perovskite dissolves into the slag phase and part of TiO₂ and MgO in wustite phase transfer into the slag phase. The low melting temperature of the slag phase is the main factor for the low softening temperatures of Ti–bearing sinter. At melting stage, TiO₂ in the slag phase is tiny and changes little. The low liquidus temperature and viscosity of the slag phase result in the low melting start temperature of Ti–bearing sinter. The slag phase melts great earlier than metallic iron while the unfused sponge iron still bounds large amounts of perovskite crystals. And then, iron collapses and releases the restrained crystals at a higher temperature, resulting in the second rise of pressure drop.

KEY WORDS: Ti–bearing sinter; softening–melting; mineral elements migration.

1. Introduction

Titanium magnetite is a typical complex iron ore that is widely distributed in many countries, such as Australia, Russia and China.^{1–3)} It is usually made into Ti–bearing pellet and high basicity sinter used in the blast furnace (BF) for producing hot metal and slag.^{4–9)} High basicity sinter has been the most popular charging burden of BF due to its superiority on metallurgical properties, such as good reducibility and high softening–melting temperatures.¹⁰⁾ For BF smelting of Ti–bearing high basicity sinter, the softening–melting behaviours are not only important, but also very complex.

In the past, a large number of researches have been carried out to study softening–melting properties of sinter. It has been reported that softening–melting temperatures could be improved by increasing basicity.^{11,12)} The softening temperatures decreased^{13–15)} and dripping temperature increased¹⁶⁾ with increasing Al₂O₃ content. Increasing MgO content could improve softening–melting properties of sinter.^{17,18)} But, the reports about softening–melting behaviours of Ti–bearing high basicity sinter are few relatively. In the study of Bao *et al.*, the Ti–bearing sinter containing high TiO₂ content (11.30%) had higher softening–melting–dripping tem-

peratures and pressure drop compared to ordinary sinter.¹⁹⁾ Recently, TiO₂ content in sinter is decreased due to adding ordinary rich ore powder in sintering. The Ti–bearing high basicity sinter with 4.76%–6.03% TiO₂ had higher softening start temperature and lower softening end temperature and dripping temperature than ordinary sinter.²⁰⁾ Y. Z. Wang *et al.* claimed that the softening temperatures were increased with the increase of w(TiO₂) in sinter and were higher than that of ordinary sinter.²¹⁾ The results of softening–melting behaviours of Ti–bearing sinter have not been consensus. In addition, we think that they may not care mineral elements migration and structural changes during softening–melting process, which may be the important root cause for softening–melting properties of Ti–bearing sinter.

In this paper, the mineral elements migration behaviour and mineralogical and structural changes of Ti–bearing high basicity sinter during softening–melting process are investigated to clarify the softening–melting properties. We hope that the results will be helpful to better understand softening–melting behaviours of Ti–bearing sinter in blast furnace.

2. Experimental

2.1. Samples

One typical Ti–bearing high basicity sinter used in the study is taken from an actual iron and steel plant in China.

* Corresponding author: E-mail: sunchangyu1979@163.com
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The chemical compositions are shown in **Table 1**. The TiO₂ content is 6.61 mass%. The binary basicity R₂ (mass% CaO/mass% SiO₂) and Al₂O₃ content are 1.93 and 3.05 mass%, approximate to those of the ordinary high basicity sinter¹⁵⁾ (sample C in the literature) respectively.

2.2. Softening–melting Procedures

The softening–melting property testing equipment is employed to study mineral elements migration behaviours and mineralogical and structural changes of Ti-bearing high basicity sinter during softening–melting process. The schematic diagrams of the apparatus and experimental conditions are given detailed in elsewhere.¹⁵⁾ The sample is heated at 10°C/min below 950°C, and kept at 950°C for 1 hour, and then heated at 5°C/min over 950°C. The gas flow is changed from N₂ (5 L/min) to reducing gas composed of CO and N₂ with the ratio 30/70 (12 L/min) until the sample temperature reaches 400°C. The three main parameters including the sample temperature, bed shrinkage ratio (Δh) and gas pressure drop (ΔP) during softening–melting process are continuously recorded.

The whole softening–melting process is divided two stages, softening stage and melting stage. The following indices are adopted to represent softening and melting characteristics of sinter. The sample temperature when Δh reaches 10%, T_{10%}, is defined as softening start temperature; T_{40%} is defined as softening end temperature when Δh reaches 40%; melting start temperature, T_m, is the temperature at which ΔP first begins to steeply rise; and melting end temperature T_f is the temperature that Δh does not change greatly.

2.3. Observation of Mineral Elements Migration

When the whole softening–melting experiment is completed, the graphite crucible is removed from the apparatus and broken to gain residual materials. Several parallel softening–melting experiments are conducted under the same conditions described in section 2.2. As soon as the sample temperature reaches desired temperature (T_{10%}, T_{40%} and T_m), the samples are quenched under high N₂ flow by turning off the furnace, obtaining interrupted samples at softening stage and melting stage. And then, interrupted samples and residual materials are photographed and separated for further investigation.

The microstructures and minerals of original sinter sample, interrupted samples and residual materials from softening–melting tests are examined using scanning electron microscope (SEM: ZEISS, SIGMA HD) and X-ray diffractometer (XRD: PANalytical, X'pert Powder) respectively. Energy dispersive spectroscopy (EDS: Oxford, X-Max 50 mm²) spot and map scanning are also used to observe distribution and migration of mineral elements. By these means, mineral elements migration behaviours of

Ti-bearing high basicity sinter during softening–melting process could be analyzed.

3. Results

3.1. Softening–melting Experiment

Figure 1 shows the result of softening–melting experiment of Ti-bearing sinter. The shrinkage (Δh) curve in first test is little different from that in repeated test, as is the pressure drop (ΔP) curve. It is proved that the experimental reliability is acceptable. As the temperature increases up to a specific value, the appearance of some liquid slag in sinter particles results in sinter layer softening under a constant load. Once the molten slag accumulates to some extent, the gas channels are plugged up and ΔP begins to rise abruptly. When ΔP reaches a maximum value, representing the worst permeability. It is noted that the ΔP curve of Ti-bearing sinter exhibits two steeply increases in both first test and second test. In contrast, the ΔP curve of ordinary high basicity sinter has just one steeply increase.¹⁵⁾ Until the end of the experiment, no dripping phenomenon is found. For Ti-bearing sinter, the sample temperature when ΔP second starts to steeply rise, is recorded as T_{m2} and defined as the second melting temperature. Compared to the ordinary sinter, Ti-bearing sinter has lower T_{10%}, T_{40%} and T_m.

3.2. Composition and Structure in Softening–melting Process

XRD analysis of Ti-bearing sinter before reduction is shown in **Fig. 2**. The diffraction peaks due to magnesioferrite, hematite, perovskite and calciumferrite are observed. Compared with relative peak intensity of the main minerals in ordinary sinter, magnesioferrite and perovskite of Ti-bearing are present in comparatively large amounts except for calciumferrite which exists just a little. It is suggested that Ti-bearing sinter would have a relatively lower reducibility.

Figure 3 shows SEM back-scattered electron images of typical structures of the original Ti-bearing sinter. The sinter contains the main structure composed of magnesioferrite, perovskite and amorphous glass phase and a relatively smaller amount of hematite and calciumferrite. EDS maps

Table 1. The chemical compositions of Ti-bearing high basicity sinter (mass%).

	TFe	FeO	SiO ₂	CaO	MgO	Al ₂ O ₃	TiO ₂	R ₂
Ti-bearing sinter	49.33	7.65	5.91	11.43	2.25	3.05	6.61	1.93
Ordinary sinter ¹⁵⁾	54.70	9.40	5.53	10.26	2.84	3.08	—	1.86

Fig. 1. The softening–melting curves of Ti-bearing sinter.

of image (a) in Fig. 3 are presented in Fig. 4. And, Table 2 shows the mineral elements contents in each phase of the original sinter. Hereafter in all the tables of this style, every mineral element content is the average of ten values analyzed at different ten positions. Fe concentration in each phase is calculated as FeO (not Fe_2O_3) to make it easier to compare the mineral elements content values among interrupted samples. According to the results, Mg is mainly

distributed in magnetite forming magnesioferrite because of isomorphism replacement between Fe^{2+} and Mg^{2+} . Al is mainly in the glass phase and calciumferrite. Most of Ti are picked up by perovskite. The TiO_2 content in calciumferrite and glass phase are 2.96 mass% and 3.79 mass%, greatly in lower quantities than that in perovskite (about 55 mass%). This is due to the greater binding affinity between CaO and TiO_2 . Large amounts of CaO are seized by TiO_2 so that no enough CaO reacts with Fe_2O_3 forming calciumferrite, which is corresponding to XRD analysis.

When sinter layer undergoes deformation under load, the softening stage is considered as the process from 10%–shrinkage to 40%–shrinkage. The sinter samples interrupted at 10% and 40%–shrinkage, symbolized as $S_{T10\%}$ and $S_{T40\%}$, representing sinter at softening stage. As shown

Fig. 2. X-ray diffraction analysis of the original sinter.

Table 2. The mineral elements contents in each phase of the original sinter (mass%).

Identified phases	CaO	MgO	Al_2O_3	SiO_2	TiO_2	FeO
Magnesioferrite	0.67	5.04	1.67	0.13	0.67	91.82
Hematite	1.35	0.63	0.48	0.03	1.86	95.65
Calciumferrite	12.79	1.69	4.71	5.52	2.96	72.33
Perovskite	40.99	0.02	0.43	0.73	55.27	2.56
Glass phase	36.68	0.62	8.03	38.79	3.79	12.09

H: hematite M: magnesioferrite CF: calcium ferrite CT: perovskite G: glass phase

Fig. 3. SEM-BSE images of typical microstructures for original Ti-bearing sinter: (a) the main structure composed of M, P and G, (b) small amounts of structure composed of H and CF.

Fig. 4. EDS maps of image (a) in Fig. 3.

in **Table 3**, the chemical compositions of $S_{T10\%}$ and $S_{T40\%}$ are observed. At 10%–shrinkage, the reducibility degree of $S_{T10\%}$ is calculated to be 78.55% which is lower than that of ordinary sinter (86.37%), indicating Ti-bearing sinter is bad reducible at low temperature. This coincides with the mineral microstructure of the original sinter sample mentioned above.

Figure 5 shows XRD analysis of $S_{T10\%}$ and $S_{T40\%}$. The main minerals of Ti-bearing sinter interrupted at softening stage are Fe, FeO, perovskite and melilite. SEM–BSE images of $S_{T10\%}$ and $S_{T40\%}$ are shown in **Fig. 6**. Obviously, from 10%–shrinkage to 40%–shrinkage, the amount of metallic iron increases. There exists a four-phase coexistent microstructure composed mainly of metallic iron, wustite, perovskite and slag phase. Some dot-like substance is found in wustite phase of $S_{T10\%}$ and it disappears in $S_{T40\%}$.

The corresponding EDS maps of the images of $S_{T10\%}$ and $S_{T40\%}$ are shown in **Fig. 7**. Ti mainly exists in perovskite while part of Ti is also picked up by wustite and the slag phase. Mg is not only distributed in FeO but also abundantly

Table 3. The chemical compositions of $S_{T10\%}$ and $S_{T40\%}$ (mass%).

Samples at softening stage	TFe	MFe	FeO	CaO	MgO	Al ₂ O ₃	SiO ₂	TiO ₂
$S_{T10\%}$	57.44	42.42	18.77	15.17	2.99	4.05	7.84	8.77
$S_{T40\%}$	59.12	53.16	7.43	15.40	3.03	4.11	7.96	8.91

Fig. 5. X-ray diffraction analysis of $S_{T10\%}$ and $S_{T40\%}$.

picked up by the slag phase. Al is mainly distributed in the slag phase. The mineral elements contents in each phase of $S_{T10\%}$ and $S_{T40\%}$ measured by EDS, are presented in **Table 4**. Fe concentration in wustite, perovskite and slag phase is calculated as FeO. From $S_{T10\%}$ to $S_{T40\%}$, TiO₂ content in wustite decreases from 7.05% to 3.16% and MgO content decreases from 6.60% to 5.16%. Meanwhile, both TiO₂ and MgO content in the slag phase has an increase trend, from 4.41% to 6.71% and from 4.86% to 6.48%, respectively. The binary basicity changes from 1.03 to 1.13. The average compositions of the dot-like substance is analyzed to be 0.04(mass%)CaO–7.14MgO–0.13Al₂O₃–0.03SiO₂–18.53TiO₂–74.13FeO, which has solidus temperature of about 1350°C and liquidus temperature of about 1380°C based on FeO–MgO–TiO₂ system.²²⁾ This suggests there exist localized areas in wustite containing high TiO₂ and MgO which pass from being into nonbeing at softening range of 1245°C–1374°C.

After softening, sinter layer enters melting stage. The sinter samples interrupted at T_m , T_{m2} and the residual materials (near T_f), are symbolized as S_{Tm} , S_{Tm2} and S_{Tf} , representing the samples at melting stage. The chemical compositions except reduced metallic iron are shown **Table 5**. As can be seen, there is still much FeO remaining in S_{Tm} . The composition of S_{Tm2} is similar to that of S_{Tf} , showing low FeO content and indicating little composition evolution from T_{m2} to T_f . **Figure 8** shows X-ray diffraction results of S_{Tm} , S_{Tm2} and S_{Tf} . The main minerals are perovskite and melilite, of which the amounts are obviously increased from softening stage to melting stage. It could believe that, as FeO is reduced, the release of Ti and Mg from wustite strengthens formation conditions of perovskite and melilite.

The photos of S_{Tm} , S_{Tm2} and S_{Tf} are shown in **Fig. 9**. At T_m , the sample still has granular sinter particles and some small iron beads are observed, indicating that slag/iron separation is not completed. When the temperature reaches T_{m2} , it is apparent that a large iron bulk generates, which shows slag/iron separation is finished. S_{Tf} has a massive structure composed of coke particles, slag and iron. **Figure 10** shows SEM–BSE images of S_{Tm} , S_{Tm2} and S_{Tf} (except for bulk iron). As can be seen, there is a little iron in images of S_{Tm2} and S_{Tf} , supporting that slag and iron have been separated. Some wustite phase is retained in S_{Tm} while no wustite is observed in S_{Tm2} and S_{Tf} , which are corresponding to the compositions in **Table 5**. As listed in **Table 6**, the mineral

Fig. 6. SEM–BSE images of $S_{T10\%}$ and $S_{T40\%}$.

 Fig. 7. EDS maps of the images of (a) $S_{T10\%}$ (b) $S_{T40\%}$.

Table 4. The mineral elements contents in each phase of $S_{T10\%}$ and $S_{T40\%}$ (mass%).

Samples at softening stage	Identified phases	Composition					
		CaO	MgO	Al ₂ O ₃	SiO ₂	TiO ₂	FeO
$S_{T10\%}$	Wustite	0.44	6.60	0.39	0.04	7.05	85.47
	Slag phase	35.11	4.86	15.21	33.87	4.41	6.54
	Perovskite	40.09	0.15	0.63	0.43	53.83	4.87
$S_{T40\%}$	Wustite	0.07	5.16	0.39	0.05	3.16	91.17
	Slag phase	34.01	6.48	16.10	30.04	6.71	6.66
	Perovskite	40.76	0.31	1.61	1.68	52.05	3.60

Table 5. The chemical compositions of S_{Tm} , S_{Tm2} and S_{Tr} except reduced metallic iron (mass%).

Samples at melting stage	FeO	SiO ₂	CaO	MgO	Al ₂ O ₃	TiO ₂
S_{Tm}	9.59	18.27	35.33	6.95	9.43	20.43
S_{Tm2}	1.79	20.09	35.42	7.29	10.02	25.40
S_{Tr}	2.40	20.11	35.23	7.64	9.83	24.79

 Fig. 8. X-ray diffraction results of S_{Tm} , S_{Tm2} and S_{Tr} .

elements contents in each phase of S_{Tm} , S_{Tm2} and S_{Tr} are revealed. In S_{Tm} , TiO₂ content in wustite and the slag phase is tiny, indicating that TiO₂ has moved into perovskite after melting start. TiO₂ content in the slag phase changes little.

Fig. 9. The photos of S_{Tm} , S_{Tm2} and S_{Tf} .

M: metallic iron W: wustite CT: perovskite S: slag phase

Fig. 10. SEM-BSE images of S_{Tm} , S_{Tm2} and S_{Tf} .**Table 6.** The mineral elements contents in each phase of S_{Tm} , S_{Tm2} and S_{Tf} (mass%).

Samples at melting stage	Identified phases	Composition					
		CaO	MgO	Al ₂ O ₃	SiO ₂	TiO ₂	FeO
S_{Tm}	Wustite	0.09	11.93	0.36	0.07	1.68	85.87
	Slag phase	35.96	7.21	18.01	32.41	0.63	5.78
	Perovskite	40.49	0.12	1.28	1.18	52.34	4.59
S_{Tm2}	Slag phase	36.03	8.19	19.77	33.23	0.66	2.12
	Perovskite	39.97	0.78	2.28	0.02	55.43	1.52
S_{Tf}	Slag phase	37.82	10.13	19.24	30.02	0.46	2.33
	Perovskite	41.85	0.23	2.25	0.83	54.19	0.65

Fig. 11. The change of mineral elements contents in glass/slag phase.

4. Discussion

4.1. Mineral Elements Migration Behaviours

When Ti-bearing sinter undergoes softening and melting, glass phase in original sinter turns into slag phase, and then the slag phase goes through a development. As shown in Fig. 11, the change of mineral elements contents in glass/slag phase from the original state to melting end is observed. Both MgO and Al₂O₃ content increase while TiO₂ content increases first and decreases later. At softening stage, the extent of the increase of TiO₂, MgO and C/S is larger than that of Al₂O₃ and FeO. Combined with the result of Table 4, it could be believed that part of TiO₂ and MgO in wustite phase transfer into the slag phase at softening stage. As evidence of XRD results, perovskite is always existing in Ti-bearing sinter during the whole softening–melting pro-

cess. Part of perovskite would dissolve into the slag phase, changing binary basicity and TiO₂ content. At melting stage, TiO₂ content in the slag phase keeps almost constant. The slag phase of S_{Tm} still has a certain amount of FeO that is close the FeO content in the slag phase during softening. The elements migration behaviours would change the slag phase property, affecting softening–melting properties of sinter.

4.2. Effect of Mineral Elements Migration on Softening–melting Properties

MgO content in wustite of Ti-bearing sinter at softening stage is about 6 mass%, lower than that (about 14 mass%) of ordinary sinter. The diffraction intensity of melilite

Fig. 12. The phase diagrams of CaO–SiO₂–5%MgO–Al₂O₃–TiO₂ with 10%Al₂O₃²³⁾ and 20%Al₂O₃²⁴⁾

mentioned in the XRD pattern of $S_{T10\%}$ and $S_{T40\%}$ is much stronger than that in ordinary sinter, although Ti-bearing sinter has similar Al₂O₃ content and lower MgO content compared to ordinary sinter. It is suggested that TiO₂ existing in wustite could suppress MgO content in wustite and promote MgO entering into the slag phase, favoring the formation of melilite. According to the phase diagrams of FeO–TiO₂ system and FeO–MgO system,²²⁾ TiO₂ and MgO make inverse influences on the melting temperature of wustite when TiO₂ or MgO content is less than 10 mass%. MgO in wustite increases the melting temperature of wustite while TiO₂ decreases that. In other words, TiO₂ would make FeO easy to dissolve into the slag phase at softening stage, which indirectly leads to low softening temperatures.

From 10%–shrinkage to 40%–shrinkage, evolution of the slag phase at softening stage is described using phase diagrams of CaO–SiO₂–5%MgO–Al₂O₃–TiO₂ system with different Al₂O₃ content level. The phase diagrams with 10%Al₂O₃²³⁾ and 20%Al₂O₃²⁴⁾ are shown in Fig. 12. The estimated liquidus temperature of the slag phase is in the range of 1 270°C–1 340°C and 1 320°C–1 370°C, respectively. What's more, there is some FeO existing in the slag phase so that the melting temperature would be much lower. In CaO–FeO–TiO₂ system,²²⁾ there is a very low melting point eutectic of 1 137°C. The low melting temperature of the slag phase at softening stage would be the main factor for the low softening temperatures of Ti-bearing sinter.

When Ti-bearing sinter melts, TiO₂ in the slag phase is tiny and changes little. As shown in Fig. 13, evolution of the slag phase at melting stage is described in CaO–SiO₂–MgO–20%Al₂O₃ phase diagram.²²⁾ The estimated liquidus temperature of the slag phase in S_{Tm} is about 1 420°C. As mentioned above, a certain amount of FeO retains in the slag phase of S_{Tm} , decreasing the melting temperature. In addition, some viscosity measurements showed that the slag containing TiO₂ and FeO had a low viscosity at 1 450°C. The viscosity of CaO–SiO₂–11.26wt%Al₂O₃–9.13wt%MgO–5wt% TiO₂–5wt% FeO with CaO/SiO₂=1.32 was 0.263 Pa·s²⁵⁾ and that of CaO–SiO₂–14wt%Al₂O₃–7wt%MgO–5wt%FeO with CaO/SiO₂=1.35 was 0.36 Pa·s.²⁶⁾ This could also result in the low T_m of Ti-bearing sinter.

The low melting temperature and low viscosity of the slag phase of S_{Tm} may lead to the slag phase melting great earlier than metallic iron. The slag phase flows into coke bed and blocks gas channels, resulting the first steeply increase of ΔP and little FeO content in S_{Tm} and S_{Tm2} . But, the unfused

Fig. 13. The phase diagrams of CaO–SiO₂–MgO–20%Al₂O₃.²²⁾

sponge iron still bounds large amounts of perovskite crystals. After reaching a higher temperature, iron collapses and releases the restrained crystals, resulting in the second rise of ΔP and the sudden increase of Δh . This may be the reason for two increases of the ΔP curve of Ti-bearing sinter.

From Fig. 13, the liquidus temperature of the slag phase in S_{Tf} is estimated to be 1 450°C approximately, lower than the T_f (1 529°C). Although the slag phase is liquid, large amounts of perovskite crystals disperse in the slag phase, causing difficult coalescence of the molten iron. Therefore, no dripping behaviour is observed.

5. Conclusions

The mineral elements migration behaviours and its effect on the softening–melting properties of Ti-bearing high basicity sinter are investigated and analyzed during softening–melting process. Major findings and conclusions have been obtained.

(1) From the original state to melting end, both MgO and Al₂O₃ content increase while TiO₂ content increases first and decreases later overall in the slag phase.

(2) At softening stage, TiO₂ existing in wustite could suppress MgO content and decreases the melting temperature of wustite, which indirectly leads to low softening temperatures. Some perovskite dissolves into the slag phase and part of TiO₂ and MgO in wustite phase transfer into the slag phase. The low melting temperature of the slag phase could be the main factor for the low softening temperatures of Ti-bearing sinter.

(3) At melting stage, TiO₂ in the slag phase is tiny and changes little. The low liquidus temperature and viscosity

of the slag phase of S_{Tm} result in the low melting start temperature of Ti-bearing sinter.

(4) The slag phase melts great earlier than metallic iron while the unfused sponge iron still bounds large amounts of perovskite crystals. Iron collapses and releases the restrained crystals at a higher temperature, resulting in the second rise of pressure drop.

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