1. Introduction

Vanadium-titanium magnetite (VTM) ore preserved in Panzhihua district (Sichuan, China) accounts for 95% of the total resource in China and a quarter of that in the world, which is a good source of the iron, vanadium, titanium. Currently, blast furnace (BF) is still the predominant technology for the utilization of VTM ore in China. However, some problems, such as the titanium cannot be effectively utilized, high-energy consumption, a large demand for the metallurgical coke and environmental pollution will become the major bottleneck to restrict the further development of the BF ironmaking technology. Therefore, two-step short process, namely direct reduction and electric furnace melting is developed, which can achieve the comprehensive utilization of Fe, V and Ti elements. Because the fluidized bed is favorable for the gases diffusion and can directly treat the ore powder without sintering. Therefore, it has been extensively applied in the direct reduction of iron ores. However, very limited literatures can be found on the direct reduction of VTM ore by means of fluidization technology.

The reduction of VTM ore is more difficult than the ordinary iron ore owing to its complex structure, requiring higher temperature and longer reduction time to achieve a relatively high metallization ratio. Destroying its dense structure and changing the phase by pre-oxidation can improve the reducibility, which has gained widespread attention. Chen treated the titanomagnetite concentrates pellets by oxidization in the range of 500–1 200°C for 30 min, then reduced the oxidized samples at 1 000–1 200°C by using pulverized coal as reducing agent. They found that pre-oxidation accelerated the reduction process by destroying the crystal lattice of the VTM and causing the formation of pores in the particles. Similarly, Park concluded that pre-oxidation of the iron sand increased its reducibility because of the structural transformation of spinel cubic TTM to rhombohedral TTH. However, different conclusion was obtained by Liu, the main phases of the VTM oxidized at 900°C for 6 min were Fe5TiO15 and Fe3O4. During the reduction process, pre-oxidation did not significantly improve the reducibility, under the same reduction conditions, the metallization rate only increased by 1% due to the formation of FeTi2O5 phase during the reduction process. The different conclusions might be attributed to the different phases such as Fe5TiO15, Fe3TiO4, FeTi2O5, FeTi2O3 generated in the oxidation-reduction process. In addition, different oxidation conditions caused some significant differences in the particle structure and element migration, which also

This paper contrastively investigated the reduction behavior of raw and oxidized vanadium titanium magnetite ore (RVTM and OVTM) in a bubbling fluidized bed at 800°C under 75%N2-25%H2, and systematically revealed the relationship between the structure, phases change, MgO migration behavior and the reduction performance. Microstructure observations suggest that oxidation conditions greatly affect structural evolution, phase transformation of Fe–Ti oxides, and migration of MgO in OVTM. The reduction results indicated that the average metallization degree enhanced by 8–10% as the OVTM ore had high oxidation degree. When the RVTM ore was treated at 800°C, the reduction rate increased with the increase of oxidation degree, no significant difference was observed among these oxidized samples due to their similar internal and surface structure. However, the reduction rate of OVTM oxidized at 950°C depended on the oxidation time. The reduction rate of OVTM oxidized at 950°C for 2 h was faster than the one for 1 h, which was attributed to the better exsolution of MgO and the formation of bigger pores, even though both samples were fully oxidized and Fe2TiO5 formed in the oxidation process.
caused the different reduction performance. However, no attention has been paid on the influence of the structure and the migration of the elements on the reduction. Moreover, the above mentioned references only referred to the reduction of the VTM pellets at high temperature by using solid carbon as reducing agent. As mentioned previously, the VTM ore powder without oxidation treatment was reduced in a fluidized bed by using CO as reducing gas. But, the reduction performance of the pre-oxidized VTM ore has not been reported.

Therefore, the focus of this paper was to investigate the reduction performance of the oxidized VTM powder at different conditions in a fluidized bed, and deeply clarify the structural evolution, phase transformation, and migration of MgO.

2. Experimental

2.1. Raw Materials

Vanadium-titanium magnetite ore powder supplied from Pan Xi area (Sichuan, China) was sieved to 100–150 μm by water, and then dried at 100°C for 24 h. The composition of the raw material was shown in Table 1.

Figure 1 gave the phases and structure of the VTM ore powder. The particle exhibits irregular edges and the surface is dense. Titanomagnetite (light phase), ilmenite (FeTiO_3, dark grey phase) and hercynite spinel (dark black phase) coexist with each other. Magnesium aluminate spinel (MgAl_2O_4 spinel) closely embeds in the interior of the particles. According to Table 2, the light area is mainly titanium magnetite, and the gray area is mainly ilmenite (FeTiO_3) and ulvite (Fe_2TiO_3). Obviously, MgO mainly exists in the ilmenite and ulvite phase in the form of solid solution. High purity (99.99%) H_2 and N_2 were used as experimental gases.

2.2. Experimental Apparatus and Processes

As illustrated in Fig. 2, the fluidized bed reactor was made of quartz glass, with a height of 860 mm and an inner diameter of 15 mm. A punched quartz glass plate welded in the middle of the bed was used as gas distributor. Pre-oxidation was performed with an air stream (0.8 L/min) continuously blew into the furnace at 800°C and 950°C, respectively. For reduction experiments, the as-oxidized VTM ore (10 g) was first fluidized with pure N_2 before it reached the desired temperature as measured by the inserted thermocouple. Then, the reduction reaction was carried out with 75%N_2-25%H_2 gas mixture (0.8 L/min, STP) for different times. After the desired time ended, the reactor was removed from the hot zone of the furnace under the protection of N_2 and directly quenched by spraying water.

2.3. Characterization and Analysis

The phases analysis was carried out on a PANalytical

![Table 1](image)

| TFe | FeO  | Fe_2O_3 | TiO_2 | MgO  | SiO_2 | Al_2O_3 | Cr_2O_3 | V_2O_5 | others |
|-----|------|---------|-------|------|-------|---------|---------|--------|--------|
| 46.11 | 20.54 | 65.92 | 12.07 | 7.86 | 7.13 | 4.43 | 1.16 | 0.65 | 0.78 |

Note: nMgs/Fe stands for the molar of Mg element dissolved into the ilmenite (FeTiO_3) and ulvite (Fe_2TiO_3) phase, nFe stands for the molar of Fe element.

![Table 2](image)

| Points | Ti | Mg | Fe | Al | O | V | Cr | nMgs/Fe |
|--------|----|----|----|----|---|---|----|---------|
| 1      | 5.41 | 1.44 | 40.88 | 0.43 | 51.11 | 0.30 | 0.41 | 0.029 |
| 2      | 9.85 | 3.76 | 34.19 | 0.36 | 51.09 | 0.30 | 0.45 | 0.105 |

![Fig. 1](image)
X’pert diffractometer with Cu Ka radiation (k = 1.5408 Å). Surface and internal structure were observed on scanning electron microscopy (SEM, JSM-6700F)/energy dispersive spectrometry (EDS, Noran System six). The elements distribution of the raw VTM was examined by EPMA (JXA-8100R, JEOL, Japan) at 20.0 kV with pure elements as standard reference. Five spots were analyzed for each phase and standard deviation of the measured concentration were ±0.5 at.%. Pore size distribution was measured on a high pressure mercury porosimeter (AutoPore IV 9500). The oxidation degree (OD) was calculated as follows:

\[ OD = \frac{T_{Fe^{2+}} - R_{Fe^{2+}}}{T_{Fe^{2+}}} \times 100 \] ............................ (1)

Where, \( T_{Fe^{2+}} \) and \( R_{Fe^{2+}} \) represented the total Fe\(^{2+}\) and the un-reacted Fe\(^{2+}\), respectively, and the results were shown in Table 3.

Table 3 Oxidation degree of the treated samples at different conditions

| Conditions              | 800°C-30 min | 800°C-120 min | 800°C-240 min | 950°C-10 min | 950°C-30 min | 950°C-60 min | 950°C-120 min |
|-------------------------|-------------|--------------|--------------|-------------|-------------|-------------|--------------|
| OD (%)                  | 63.87       | 74.63        | 83.15        | 78.77       | 92.50       | 98.75       | 98.97        |

3. Results

3.1. Reduction Performance of the Raw and Oxidized Samples

Figure 3 shows the reduction curves of the raw and the oxidized VTM ore. Compared with the untreated one, the average MD of the oxidized samples increased by 10–15%. The volume increases by 25% as the magnetite transforms to hematite, which ascribes to the arrangement of oxygen atoms in the close-packed hexagonal structure to form a face-centered cubic structure.\(^{10}\) It is well known that reduction rate of the magnetite ore is slower than the hematite ore. Therefore, the reducibility of magnetite can be greatly improved by oxidation. In addition, the formation of pores was beneficial for the diffusion of the reducing gas.

Figure 3 shows that the effect of oxidation temperature on the trend of reduction curves is distinct. On one hand, when the oxidation temperature is 800°C, the reduction rate of OVTM increases with increasing OD. As the value of OD exceeds 74.63%, the influence of oxidization on the MD of OVTM is marginal.

On the other hand, when the oxidation temperature is 950°C, the reduction rate could be divided into three kinds of circumstances. When the VTM powder was oxidized at 950°C for a short time (≤ 30 min), the reduction rate gradually increased with the increase of the oxidization degree. Similarly, when the raw VTM ore was oxidized for 30 min, the metallization degree of the sample was higher about 10% than the untreated one. However, the reduction rate decreased as the oxidation time was prolonged to 60 min, which was equivalent to that of the sample treated for 10 min.

As shown in Table 3, the oxidation degree were 98.75% and 98.97% when the VTM were oxidized for 60 min and 120 min, respectively, which indicated the VTM almost has been fully oxidized. However, the metallization degree of the sample oxidized for 120 min was average higher 8% than that of the sample treated for 60 min. In addition, the sample treated for 120 min had the highest reduction rate among the tested samples.

![Fig. 2. Schematic diagram of the fluidized bed reduction system.](image1)

1-Computer; 2-Pressure sensor; 3-Fluidized bed; 4-Furnace; 5-Mass flow controller; 6-Valve; 7-Gas sources; 8-Thermocouple; 9-Temperature controller system.

![Table 3. Oxidation degree of the treated samples at different conditions.](image2)

![Fig. 3. The reduction curves of the sample oxidized at 800°C and 950°C.](image3)
3.2. Phase Transformation during the Reduction Process

To analyze the reduction process of the raw and the oxidized VTM ore, the phase transformation of Fe–Ti oxides in RVTM and OVTM was investigated. For both the raw and oxidized VTM, FeTiO$_3$ still could be detected when the metallization degree was as high as 88% (as shown in Fig. 4), indicating the FeTiO$_3$ phase is difficult to be reduced. As reported in some researches,$^{13,14}$ the new phase of Fe$_2$TiO$_4$ was generated in the initial stage of the VTM reduction, which was further reduced to FeTiO$_3$, and finally was reduced to Fe and TiO$_2$. However, no peaks belongs to Fe$_2$TiO$_4$ could be detected due to its low content. Moreover, there might be a FeTiO$_3$ regenerated reaction by consuming Fe$_2$O$_3$ and TiO$_2$. Correspondingly, the reduction process of the oxidized sample at 800°C could be described as follows:

\[
\begin{align*}
\text{Fe}_2\text{O}_3 & \rightarrow \text{Fe}_3\text{O}_4 \rightarrow \text{Fe}_{1.8}\text{O} \rightarrow \text{Fe} \quad \ldots \quad (3) \\
\text{Fe}_2\text{O}_3 + \text{TiO}_2 & \rightarrow \text{FeTiO}_3 \rightarrow \text{Fe} + \text{TiO}_2 \quad \ldots \quad (4)
\end{align*}
\]

For the sample oxidized at 950°C for 2 h, the reduction path for the Fe$_2$O$_3$ phase was similar with the sample oxidized at 800°C for 4 h Fig. 4(c). Fe$_2$TiO$_4$ was gradually generated accompanied with the disappearance of the Fe$_2$TiO$_5$. The diffraction peaks belong to FeTiO$_3$ that were detected as the sample was reduced for 30 min, which attributed to the reduction of Fe$_2$TiO$_4$. Therefore, the reduction process of sample oxidized at 950°C for 2 h included reaction (3) and (5).

\[
\begin{align*}
\text{Fe}_2\text{TiO}_3 & \rightarrow \text{Fe}_3\text{TiO}_4 \rightarrow \text{FeTiO}_3 \rightarrow \text{Fe} + \text{TiO}_2 \quad \ldots \quad (5)
\end{align*}
\]

In some reported documents,$^9,16$ the reduction sequence of titanium magnetite was:

\[
\begin{align*}
\text{Fe}_3\text{Ti}_4\text{O}_9 & \rightarrow \text{FeO} + \text{Fe}_2\text{TiO}_4 \rightarrow \text{Fe} + \text{Fe}_2\text{TiO}_4 \quad \ldots \quad (6) \\
& \rightarrow \text{Fe} + \text{FeTiO}_3 \rightarrow \text{Fe} + \text{FeTi}_2\text{O}_5 \rightarrow \text{Fe} + \text{TiO}_2
\end{align*}
\]

It was reported that the formation temperature of FeTi$_2$O$_5$ was as high as 1 050°C.$^9$ Therefore, FeTi$_2$O$_5$ phase could not be detected under the current experimental conditions. Clearly, the final reduction process mainly referred to the reduction of the original or newly generated ilmenite. It was worth to noting that some crystal information of the regenerated FeTiO$_3$ has changed. Compared with the FeTiO$_3$ in the raw VTM, the lattice plane $a$ and $c$ of the newly formed FeTiO$_3$ increased by ca. 0.062% and 0.087%, respectively. According to the crystal structure theory, the increase of the crystal cell parameters means the crystal stability decrease and the activity increase. Thus, the regenerated FeTiO$_3$ had high activity due to the existence of some defects, which was beneficial for the reduction.

3.3. Morphology Evolution and MgO Migration Behavior

For the OVTM, see Fig. 5, in the initial reduction stage, the average size of the generated Fe particles was about 500 nm, and some un-reacted cores could be found in the particle interior. Most of the metallic iron existed as dot, linear distribution between the grain boundary and the un-reacted core area. The Fe particles presented as monodisperse state gradually grew up to ca. 2 μm as prolonged the reduction time.

In contrast, the Fe particles formed by the reduction of the untreated VTM were relatively large, some particles connected with each other (Fig. 6). As stated previously, oxid-
zation improved the diffusivity of reducing gas. As a result, smaller crystal of particles were obtained via increasing the number of the crystal nucleus and the nucleation rate.\textsuperscript{17)}

When the iron ore contains MgO, it is difficult for iron oxides to be reduced due to the existence of the stable solid solution.\textsuperscript{1,18)} According to Table 1, the content of MgO and Al\textsubscript{2}O\textsubscript{3} is 7.86% and 4.43% in the raw VTM ore, respectively. The amount of MgO in the form of magnesium aluminate spinel (MgO-Al\textsubscript{2}O\textsubscript{3}) is about 1.74%, accounting for 39.28% of the total amount of MgO. Therefore, 60.72% of the MgO combines with FeO that contained in the titanium magnetite, ilmenite and ulvite to form solid solution, which can significantly influence the reduction performance of VTM ore.

According to Table 4, both $n_{\text{MgS}}/n_{\text{Fe}}$ in the dark area of the raw VTM ore and the oxidized sample gradually increased as prolonged the reduction time was prolonged, indicating the MgO migrated into the FeTiO\textsubscript{3} and enriched in the form of solid solution. Therefore, it was difficult for the raw or newly formed FeTiO\textsubscript{3} to be reduced, that was why the diffraction peaks belong to FeTiO\textsubscript{3} could be still detected when the products had high metallization degree. However, the $n_{\text{MgS}}/n_{\text{Fe}}$ of the oxidized sample increased faster than that of the raw VTM during the reduction process, indicating the iron ions and the MgO in the oxidized sample migrated more rapidly than the raw VTM one. Therefore, the reduction rate of the oxidized sample was much faster than the raw VTM ore.

4. Discussions

It is well known that some factors such as reaction conditions, mineral structure, reactants, reactor, etc. can influence the the reduction performance of the iron ore. As described previously, for the VTM ore oxidized at 800°C, the reduction rate insignificantly increased with the increase of the oxidation degree.

As shown in Fig. 7, the diffraction peaks representing the pseudobrookite phase (Fe\textsubscript{2}TiO\textsubscript{5}) by consuming hematite and rutile began to appear after being roasted at 950°C for 30 min. Moreover, the particle surface became dense because the nano-sized sheets were sintered together, as shown in Figs. 8(b) and 8(c). It suggests that the Fe\textsubscript{2}TiO\textsubscript{5} content in the sample oxidized for 1 h was higher than the sample oxidized for 30 min. Consequently, the reduction rate still decreased though more pores were formed.

When the raw VTM was oxidized for 120 min, only Fe\textsubscript{2}O\textsubscript{3} and Fe\textsubscript{2}TiO\textsubscript{5} could be detected. It is reasonable to believe that most of the TiO\textsubscript{2} have converted to Fe\textsubscript{2}TiO\textsubscript{5} because of the high content of Fe\textsubscript{2}O\textsubscript{3}. Obviously, the Fe\textsubscript{2}TiO\textsubscript{5} content in OVTM oxidized for 2 h was equal to or higher than that oxidized for 1 h. As illustrated in Fig. 8, with the extension of the oxidation time, the nano-sized particles on the surface became molten state, connected with each other and formed a compact layer. Therefore, from the perspective of

\begin{table}
\centering
\caption{EDS results of the raw VTM and oxidized at 950°C for 2 h after reduced for different times.}
\begin{tabular}{cccccccc}
\hline
Points & Ti & Mg & Fe & Al & O & V & $n_{\text{MgS}}/n_{\text{Fe}}$ \\
\hline
1 & 7.36 & 2.92 & 33.08 & 1.17 & 55.30 & 0.18 & 0.07 \\
2 & 6.17 & 2.41 & 34.88 & 2.37 & 53.81 & 0.37 & 0.035 \\
3 & 11.95 & 4.04 & 29.21 & 3.99 & 49.96 & 0.85 & 0.07 \\
4 & 16.43 & 5.10 & 25.39 & 2.25 & 49.90 & 0.92 & 0.156 \\
5 & 16.91 & 5.67 & 16.34 & 3.02 & 54.73 & 0.97 & 0.269 \\
6 & 6.02 & 2.07 & 33.24 & 0.41 & 58.00 & 0.25 & 0.056 \\
7 & 9.59 & 2.46 & 35.81 & 0.97 & 49.96 & 0.29 & 0.026 \\
8 & 12.41 & 3.34 & 33.05 & 2.23 & 47.30 & 0.59 & 0.063 \\
9 & 21.37 & 5.23 & 33.43 & 2.29 & 36.94 & 0.71 & 0.122 \\
10 & 15.84 & 7.66 & 21.86 & 3.59 & 50.01 & 0.57 & 0.268 \\
\hline
\end{tabular}
\end{table}
diffusion, the compact layer might be unfavorable for the diffusion of the reducing gas and the gaseous products.

However, the internal structure exhibited obvious distinctions, as shown in Fig. 9. Light and dark phase with irregular structure gradually separated and grew up when the VTM ore was treated at 950°C for 2 h. According to the EDS results (Table 5), the dark phase was mainly Fe₂TiO₅. Therefore, the Ti element was separated from the Fe₂O₃ and enriched in the form of Fe₂TiO₅. It was worth noting that the content of MgO gradually decreased in the light phase while increased in the dark phase, indicating the MgO migrated from the solid solution and enriched in the dark phase. Sun16) and She’s19) work indicated that it was difficult for the titanitic magnetite and ilmenite contained MgO to be reduced due to the barrier effect of MgO. Therefore, the migration of MgO from the solid solution was beneficial for the reduction. Moreover, the pore of the VTM treated for 2 h was bigger than that treated for 1 h, which might be ascribed to the MgO migration. Therefore, the formation of bigger pores was favorable for the reducing gas to contact with the raw material and the diffuse out from the pores.

As described previously, MgO gradually migrated from the solid solution by exsolution as extended the oxidization time, this phenomenon was attributed to the limited solution content of MgO in Fe₂O₃.20) Therefore, pre-oxidization could effectively decrease the content of MgO in the iron oxides. Moreover, the results also demonstrated that it was beneficial for the MgO exsolution as the raw VTM was treated for a long time, which could well explain the phenomenon that the reduction performance of the VTM oxidized at 950°C for 2 h was more excellent than that of the sample oxidized for 1 h.

We analyzed the phase evolution of the samples pre-oxidized at 800°C, as shown in Fig. 7(a). Fe₃O₄ and FeTiO₃ disappeared gradually as prolonged the oxidization time, the final oxidation products were rutile (TiO₂), hematite (Fe₂O₃) and little titanitic magnetite after being oxidized at 800°C for 120 min and 240 min. The results meant that the reduction process mainly referred to the reduction of Fe₂O₃. Because no Fe₂O₃ was consumed to form Fe₂TiO₅, as calculated the from the oxidization degree and the raw VTM composition, the Fe₂O₃ content were 81.56% and 84.54% when the oxidization degree were 74.63% and 83.15%, respectively. So, the sample oxidized for 1 h and 2 h had the similar reduction performance from the viewpoint of Fe₂O₃ content.

Figure 10 shows the internal structure of the particles, compared with the RVTM, no obvious change appeared in the internal structure when it was oxidized for 30 min. With the extension of the oxidization time, the as-oxidized samples had similar internal structure with some holes. Therefore, the reduction rate was close to each other as the VTM was treated at 800°C for 2 h and 4 h. It could be speculated that MgO did not significantly migrate and enrich. Thus, the exsolution content of MgO was low compared with the sample treated at 950°C.

Table 5. EDS results of the oxidized VTM at 950°C for 1.0 h and 2.0 h.

| Points | Element Content (atom.%) | \( n_{Mg}/n_{Fe} \) |
|--------|--------------------------|---------------------|
|        | Ti          | Mg   | Fe   | Al | O | V |       |
| 1      | 10.15       | 0.91 | 27.08 | 0.77 | 60.24 | 0.27 | 0.019 |
| 2      | 5.40        | 1.56 | 32.03 | 0.34 | 60.03 | 0.13 | 0.043 |
| 3      | 10.37       | 1.99 | 28.92 | –    | 58.04 | 0.35 | 0.069 |
| 4      | 4.21        | 0.41 | 35.37 | 0.70 | 58.86 | 0.20 | 0.0017 |

Fig. 8. Surface morphology evolution of the VTM ore oxidized at 950°C for (a) 10 min; (b) 30 min; (c) 60 min; (d) 120 min.

Fig. 9. Back-scattered electron images of the VTM ore oxidized at 950°C for (a) 10 min; (b) 30 min; (c) 60 min; (d) 120 min.

Fig. 10. Internal structure of the sample oxidized at 800°C for (a) 30 min; (b) 120 min; (c) 240 min.
of large pores. Therefore, the reduction rate of the sample treated at 800°C for 4 h was similar to the one treated at 950°C for 2 h. In fact, the sample treated at higher temperature for a long time also was uneconomical for the industrial procedure. In short, it should reasonably estimate the energy consumption in the oxidation step and the improvement of the reduction rate, and judge the economical efficiency of the oxidation treatment.

5. Conclusions

(1) Pre-oxidization was beneficial for the fluidization reduction. The average metallization degree could be enhanced 8-10% as the raw VTM ore was oxidized at 800°C and 950°C.

(2) When the sample was treated at 800°C, the oxidation degree and the reduction rate increased gradually with the reduction time. However, no significant difference about the reduction rate could be found due to the similar internal and external structure.

(3) Fe₂TiO₅ phase that was unfavorable for the reduction generated during the oxidation as the raw VTM was treated at 950°C. When the VTM has been fully oxidized at 950°C, the sample treated for longer time was advantageous for the exsolution of the MgO, the formation of big pores, and showed faster reduction rate than that oxidized for shorter time.

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