# Upgrading of raw vanadium titanomagnetite concentrate

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## Synopsis

**In view of the continuous depletion of titanium, rutile, and ilmenite mineral deposits, the search for cost-effective practices for titanium production using vanadium titanomagnetite as raw material has become increasingly important. In this study, vanadium titanomagnetite concentrate obtained from the Panzhihua area in China was upgraded via fine grinding followed by magnetic separation to produce high-quality vanadium titanomagnetite concentrate. The potential utilization of this concentrate was investigated. Mineral liberation analysis and electron microprobe analysis were used to investigate the deportment of the impurities and major minerals. Experimental results showed that magnetic separation increased the Fe grade by 4% compared with the raw concentrate, while the total content of $Al_2O_3$, $SiO_2$, MgO, and CaO impurities decreased from 12.35% to 7.73%. Process mineralogy studies confirmed that the proportion of titanomagnetite in the high-quality vanadium titanomagnetite concentrate increased to 95.84%. Spinel and sphene were enclosed in the titanomagnetite particles at the nanometre scale, which would make them practically impossible to remove through mineral processing techniques.**

### Keywords

**beneficiation, vanadium titanomagnetite, fine grinding, concentration, magnetic separation, process mineralogy.**

## Introduction

Vanadium titanomagnetite deposits are widely distributed around the world. Vanadium titanomagnetite usually contains valuable elements such as Fe, V, and Ti; rare and noble metals such as Co, Tc, Ga, Pt, and Pd can also be present (Chen *et al.*, 2015; Zhu, Li, and Guan, 2016; Khomich and Boriskina,.2014; Luo *et al.*, 2013). Therefore, considerable value can be realized through comprehensive utilization of the ore. Currently, vanadium titanomagnetite concentrate and ilmenite concentrate can be obtained by traditional ore dressing methods such as magnetic separation, gravity separation, and flotation. Generally, low-intensity magnetic separation is first conducted to beneficiate the vanadium titanomagnetite concentrate after grinding of the raw ore. Then, flotation or electrostatic separation is used to recover the ilmenite concentrate from the magnetic separation tailings (Hukkanen and Walden, 1985; Chen *et al.*, 2011; Wang *et al.*, 2017; Chen *et al.*, 2013).

The Panzhihua area in China is very rich in vanadium titanomagnetite resources, with proven reserves of approximately 10 Gt (Wang *et al.*, 2016). The vanadium and titanium reserves rank as third and first in the world, respectively (Du, 1996). Ilmenite concentrate from Panzhihua has been concentrated by high-gradient magnetic separation and flotation. The ilmenite concentrate can be treated with sulphuric acid to obtain titanium dioxide (titanium white) or by high-temperature smelting to produce high-titanium slag. Approximately 30% of the titanium from raw vanadium titanomagnetite can be recovered into an ilmenite concentrate. Low-intensity magnetic separation can be used to obtain vanadium titanomagnetite concentrate, which is mainly used for producing steel and recovering vanadium through a conventional blast furnace-converter smelting process. Almost all the titanium remains in the titanium-bearing blast furnace slag, with 22–25% $TiO_2$ as the by-product. Owing to the low $TiO_2$ content and complex mineral phases in the slag, the $TiO_2$ is difficult to recover. As a result, approximately 7400 Mt of titaniferous slag has been stacked as solid waste, which has led to a considerable loss of titanium resources and environmental pollution (Huang *et al.*, 2013; Jiao *et al.*, 2018; Wang *et al.*, 2006; Wen and Zhang, 2011). Therefore, it is of considerable importance to develop effective methods for the comprehensive utilization of the titanium resource in vanadium titanomagnetite concentrate.

A direct reduction–electric furnace smelting process has been successfully applied in South Africa and New Zealand for comprehensive utilization of vanadium titanomagnetite concentrate (Samanta, Mukherjee, and Dey, 2015). However, alkaline flux must be added to the raw materials during the direct reduction process to improve the separation between iron and slag. This indirectly decreases the titanium grade of the raw materials. As a result, the smelting slag contained 30–33% $TiO_2$. The recovery of titanium from smelting slag through hydrometallurgical or pyrometallurgical methods is extremely difficult, owing to the amount of impurities and complex mineralogical characteristics. This kind of titaniferous slag (containing 30–33% $TiO_2$) has also been stacked as solid waste (Jena, Dresler, and Reilly, 1995). Hence, effective extraction of titanium from titanium-bearing electric furnace slag has become a significant problem. Research has indicated that when the $TiO_2$ content in titanium slag is above 50%, it can be recovered by acid leaching methods (Zheng *et al.*, 2016). Therefore, to improve the $TiO_2$ grade in titanium slag, a reduction-melting process was pursued by Panzhihua Iron and Steel Group. In this process, the reduction of vanadium titanomagnetite concentrate (containing approximately 54% Fe) was conducted in a rotary kiln or rotary hearth furnace, then the metallized pellets were melted in an electric arc furnace in the absence of a fluxing medium. The product titanium slag contained 45–50% $TiO_2$ (Liu, Wen, and Qie, 2015). However, the content of impurities, especially the $SiO_2$ content, was still high, making the extraction of $TiO_2$ from the slag very difficult owing to the low acidolysis of the slag. Thus, it is essential to decrease these impurities during the initial processing stage to obtain the desired titanium slag ($TiO_2 > 50\%$) and make the subsequent extraction of $TiO_2$ feasible and effective. However, minimal information is available regarding the preparation of titanium slag with a higher $TiO_2$ grade and the preparation of titanium white by treatment of the titanium slag with sulphuric acid. It is very important to solve the problem of efficient utilization of the titanium resource present in the vanadium titanomagnetite concentrate due to the large reserves of vanadium titanomagnetite in Panzhihua. Unfortunately, only a few studies have been conducted on the exploitation of titanium slag obtained from vanadium titanomagnetite concentrate in Panzhihua, mainly because of the complex phase composition and high content of impurities such as $SiO_2$, $Al_2O_3$, MgO, and CaO. Decreasing the impurities in the vanadium titanomagnetite concentrate to improve the mass ratio of $TiO_2$ to impurities is an effective strategy to obtain high-quality titanium slag through a reduction–melting process (Lv *et al.*, 2017).

The aim of this study was to upgrade raw vanadium titanomagnetite concentrate to obtain a high-quality concentrate by using a conventional and economical fine grinding and magnetic separation process. To predict the effectiveness of the technique, process mineralogy of the high-quality vanadium titanomagnetite concentrate sample was investigated.

## Experimental

### *Materials*

Raw vanadium titanomagnetite concentrate provided by Panzhihua Iron and Steel Group Ltd. in Sichuan Province was used in the experiments. The size analysis was about 87% below 75 µm. The chemical composition is shown in Table I. The content of Fe was 53.47%, and the grade of $TiO_2$ was 12.46%. The total content of the $SiO_2$, $Al_2O_3$, MgO, and CaO impurities was 12.35%. As shown in the XRD spectrum in Figure 1, the main crystalline phases present were titanomagnetite ($Fe_{2.75}Ti_{0.25}O_4$) and a small quantity of ilmenite ($FeTiO_3$).

## *Methods*

### *Fine grinding and magnetic separation*

To obtain a high-quality vanadium titanomagnetite concentrate, the raw vanadium titanomagnetite concentrate was upgraded by fine grinding in a ball mill (ZQMΦ250×100) and magnetic separation using a counter-rotation wet drum separator (Φ800×300 mm). The fine grinding was carried out by wet grinding at a solids to water ratio of 500:275 by mass). The effect of the fineness of grind and magnetic field intensity on the Fe grade and Fe recovery from the concentrate was investigated. The recovery from the concentrate was calculated by the following formula:

$$\eta = \frac{W_C}{W_T} \times \frac{\alpha}{\beta} \times 100\% \qquad [1]$$

where $\eta$ is the Fe recovery to the concentrate, $W_C$ is the weight of the concentrate, $W_T$ is the weight of the raw sample, $\alpha$ is the Fe grade of the concentrate, and $\beta$ is the Fe grade of the raw sample.

### *Mineral liberation analysis*

The process mineralogy of the high-quality concentrate was studied to determine the purification efficiency using a mineral liberation analyser (MLA, FEI 200) equipped with X-ray energy-dispersive spectroscopy (EDS). A total of 213 824 mineral particles from high-quality vanadium titanomagnetite concentrate were scanned.

### *Electron microprobe analysis (EPMA)*

A JEOL JXA-8100 electron probe microanalyser equipped with four wavelength-dispersive spectrometers was used to investigate the characteristics of the minerals in the high-quality concentrate sample.

## Results and discussion

### *Effect of the fineness of grind on the magnetic separation index*

Chemical analysis of the raw vanadium titanomagnetite concentrate indicated that the content of impurities was still

*Table I*

**Chemical composition of the raw vanadium titanomagnetite concentrate (wt%)**

| Fe (total) | $TiO_2$ | $SiO_2$ | CaO | MgO | $Al_2O_3$ | V | S |
|---|---|---|---|---|---|---|---|
| 53.47 | 12.46 | 3.68 | 1.24 | 3.25 | 4.23 | 0.39 | 0.66 |

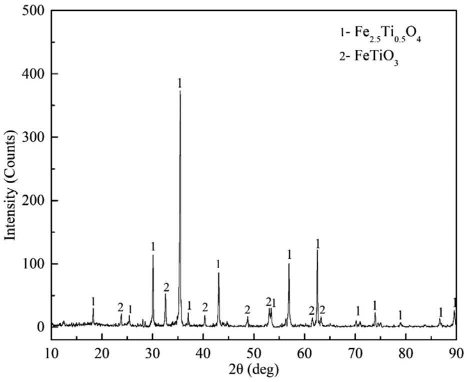

**Figure 1—XRD spectrum of raw vanadium titanomagnetite concentrate**

high, and the mass ratio of $TiO_2/(SiO_2+MgO+Al_2O_3+CaO+TiO_2)$ was 0.48. Theoretically, the $TiO_2$ grade of the titanium slag after the reduction and melting of the raw vanadium titanomagnetite concentrate should reach a maximum value of 48%. Therefore, a low-intensity magnetic separation was performed first on the milled raw concentrate. The magnetic field intensity was set at 1130 Gs. The relationship between the fineness of grind and magnetic separation index (the grade and percentage recovery of Fe from the concentrate) after one cleaning is shown in Figure 2.

From the results in Figure 2, it was evident that the Fe grade increased as the fineness of grind increased. Specifically, the Fe grade reached 55.56% when the grinding fineness was increased to 90% –45 µm. A further increase in the grinding fineness did not significantly improve the Fe grade. Therefore, a grinding fineness of 90% –45 µm was chosen for the subsequent tests.

### *Effect of magnetic field intensity on the magnetic separation index after secondary cleaning*

Since the Fe grade of the concentrate after one cleaning was low, a secondary cleaning was performed at a lower magnetic intensity. The magnetic field intensities used for the secondary cleaning were 377, 502, 628, 754, and 879 Gs. The effect of the magnetic field intensity on the Fe grade and Fe recovery of high-quality concentrate after the secondary cleaning is shown in Figure 3.

As shown in Figure 3, the Fe grade increased as the magnetic field intensity decreased. However, decreasing the magnetic field intensity below 628 Gs resulted in a little improvement in the Fe grade, while the Fe recovery sharply decreased. Therefore, a magnetic field intensity of 628 Gs was recommended. The chemical composition of the obtained high-quality concentrate is shown in Table II. The Fe grade reached 57.24%, with a $TiO_2$ content of 13.15%. The recoveries of Fe and $TiO_2$ to the high-quality concentrate were 81.28% and 80.35%, respectively. The total content of $SiO_2$, $Al_2O_3$, MgO, and CaO impurities decreased from 12.35% to 7.73%. The mass ratio of $TiO_2/(TiO_2+SiO_2+Al_2O_3+CaO+MgO)$ increased to 0.63, which indicated that the $TiO_2$ content of the titanium slag could theoretically reach approximately 63% after reduction-melting in the absence of additive. Even after adjusting the binary basicity to 0.6–1.0, the content of $TiO_2$ in the slag could still remain above 55%. It was demonstrated that by using a high-quality vanadium titanomagnetite concentrate in the reduction–melting process, the $TiO_2$ content in the titanium slag product reached 60.68% – a grade suitable for the preparation of titanium pigment by treatment with concentrated sulphuric acid (Jiao *et al*., 2018).

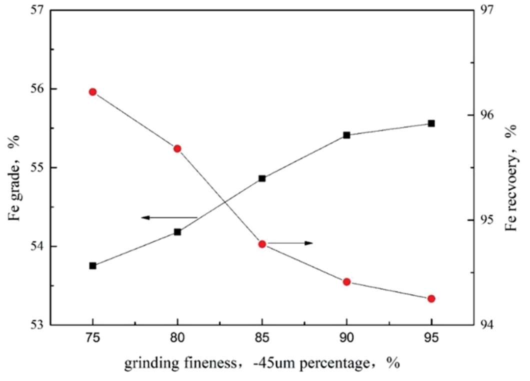

**Figure 2—Effect of fineness of grind on the Fe grade and recovery after the first cleaning magnetic separation stage**

Bench-scale test work showed that more than 95% of the $TiO_2$ in this slag could be leached, and a titanium pigment product containing 99.6% $TiO_2$ was produced successfully.

### *Production of iron concentrate via one-stage scavenging*

In order to reduce resource losses as much as possible, an iron concentrate with Fe grade 54.06% was obtained *via* one-stage scavenging at a magnetic field strength of 879 Gs, which could be used for blast furnace ironmaking. The complete flow sheet is shown in Figure 4, and the test results in Table III. From Table III, it can be seen that the total recovery of Fe in the high-quality concentrate plus iron concentrate was above 93%, and $TiO_2$ in the high-quality concentrate could be recovered to produce titanium pigment, since the grade is acceptable for the Panzhihua Iron and Steel Group. Moreover, the value of the high-quality vanadium titanomagnetite concentrate achieved by above mentioned technology can be increased by 10 dollars per ton with a preliminary estimate. More importantly, the titanium slag with a $TiO_2$ content of 60.68% has great potential values.

### *Mineral composition and content*

The results of the MLA analysis and the mineral composition and content of the high-quality concentrate are shown in Figure 5 and Table IV.

As shown in Figure 5 and Table IV, the proportion of valuable minerals such as titanomagnetite, ilmenite, and haematite in

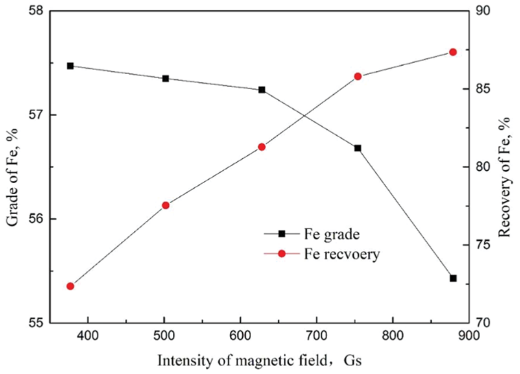

**Figure 3—Effect of magnetic field strength on Fe grade and recovery**

*Table II*

**Major chemical components in the high-quality vanadium titanomagnetite concentrate (%)**

| Fe (total) | $TiO_2$ | $SiO_2$ | CaO | MgO | $Al_2O_3$ | V | S |
|---|---|---|---|---|---|---|---|
| 57.24 | 13.15 | 1.41 | 0.40 | 2.36 | 3.56 | 0.40 | 0.22 |

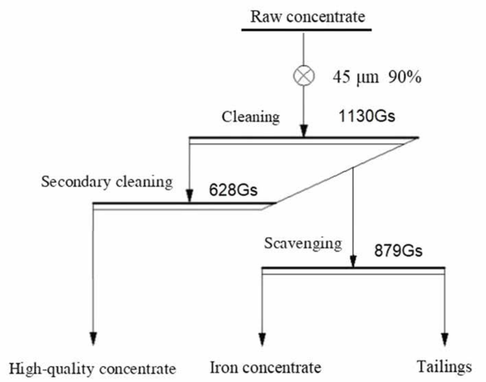

**Figure 4—Flow sheet for upgrading of raw concentrate**

*Table III*

**Test results from the flow sheet in Figure 4**

| Product | Yield % | Grade % | | Recovery % | |
|---|---|---|---|---|---|
| | | Fe (total) | $TiO_2$ | Fe | $TiO_2$ |
| High-quality concentrate | 76.13 | 57.24 | 13.15 | 81.28 | 80.35 |
| Iron concentrate | 12.46 | 54.06 | 10.32 | 12.53 | 10.36 |
| Tailings | 11.41 | 29.01 | 10.15 | 6.19 | 9.29 |
| Raw concentrate | 100 | 53.47 | 12.46 | 100 | 100 |

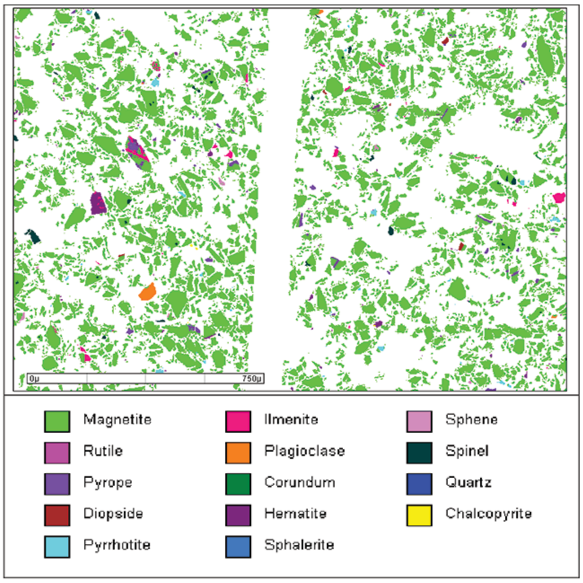

Figure 5—MLA image of the high-quality concentrate

*Table IV*

**Mineral composition and content of the high-quality concentrate (wt%)**

| Mineral | Chemical formula | % |
|---|---|---|
| Titanomagnetite | $Fe_{2.75}Ti_{0.25}O_4$ | 95.84 |
| Ilmenite | $FeTiO_3$ | 0.66 |
| Sphene | $CaTiSiO_5$ | 0.18 |
| Plagioclase | $CaSi_3AlO_8$ | 0.06 |
| Spinel | $(Mg_{0.5},Fe_{0.5})Al$ | 0.53 |
| Pyrope | $Mg_3Al_2(SiO_4)_3$ | 0.98 |
| Corundum | $Al_2O_3$ | 0.02 |
| Quartz | $SiO_2$ | 0.02 |
| Diopside | $CaMg(Si_2O_6)$ | 0.12 |
| Haematite | $Fe_2O_3/FeOOH$ | 0.98 |
| Pyrrhotite | $Fe_{1-x}S$ | 0.54 |
| Total | | 100.00 |

the high-quality vanadium titanomagnetite concentrate was approximately 97.48%. The total content of gangue minerals was less than 3%, with sphene (0.18%), spinel (0.53%), and pyrope (0.98%) being the major gangue constituents.

### *EMPA analysis of titanomagnetite*

As shown in Table II, the total impurity content in the high-quality vanadium titanomagnetite concentrate was approximately 7%. The reason for this was elucidated from the results of EMPA analysis of some representative titanomagnetite grains, as shown in Figure 6.

From Figure 6, it can be seen that the titanomagnetite was closely associated with spinel ($MgAl_2O_4$), pyrope ($Mg_3Al_2(SiO_4)_3$), and sphene ($CaTiSiO_5$). Fine spinel and sphene were distributed at the nanometre scale in titanomagnetite, and pyrope was embedded in titanomagnetite at a scale of several micrometres. This indicates that a further upgrading of the high-quality vanadium titanomagnetite concentrate by mineral processing techniques would be extreme difficult.

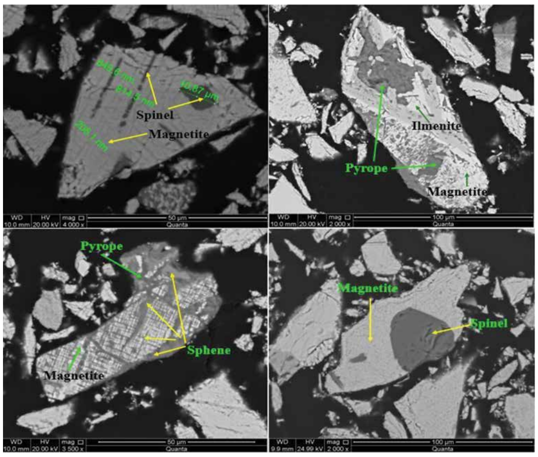

Figure 6—EPMA images illustrating textural relationships between titanomagnetite and gangue minerals

### *Size distribution of the main gangue minerals*

The size distribution of the main gangue minerals in the high-quality concentrate is shown in Table V. Almost of the sphene was smaller than 38 µm, mostly in the –27 µm +6.8 µm fraction. More than 90% of the spinel was less than 38 µm and mainly between –38 µm and +4.8 µm. More than 90% of the pyrope was below 45 µm and mainly distributed in the range of –48 µm to +6.8 µm. Most of the gangue minerals were in the –38 µm to +4.8 µm fraction. It was difficult to remove these impurities by using physical mineral processing methods; this was the main reason why the content of Al, Mg, and Si in the high-quality concentrate was relatively high.

## Conclusions and recommendation

1. The content of impurities in raw concentrate produced at the Panzhihua ore dressing plant was high. The total content of $Al_2O_3$, $SiO_2$, MgO, and CaO was 12.35%. The mass ratio of $TiO_2/(SiO_2+Al_2O_3+CaO+MgO+TiO_2)$ was 0.48. The comprehensive utilization of titanium and iron via reduction–melting process from this raw concentrate may be difficult.
2. After the upgrading process with fine grinding and a two-stage low-intensity magnetic cleaning, a high-quality vanadium titanomagnetite concentrate was obtained. The total content of $Al_2O_3$, $SiO_2$, MgO, and CaO decreased to 7.73%. The mass ratio of $TiO_2/(SiO_2+Al_2O_3+CaO+MgO+TiO_2)$ was 0.63, which makes the concentrate suitable for the production of titanium slag.
3. Process mineralogy of the high-quality vanadium titanomagnetite concentrate showed that main gangue minerals, such as sphene, spinel, and pyrope, were finely distributed in the titanomagnetite particles, and their removal by physical mineral processing methods would be extremely difficult.
4. In practice, the upgrading process could be carried out directly to treat raw concentrate without changing the original process. Fine grinding to 90% -45 µm would be achieved through

*Table V*

**Size distributions of major gangue minerals (wt %)**

| Fineness (µm) | Sphene | | Spinel | | Pyrope | |
|---|---|---|---|---|---|---|
| | % | Cum. % | % | Cum. % | % | Cum. % |
| +75 | - | - | - | - | - | - |
| -75+45 | - | - | 8.78 | 8.78 | 6.69 | 6.69 |
| -45+38 | - | - | 0.00 | 8.78 | 9.54 | 16.23 |
| -38+27 | 6.00 | 6.00 | 12.28 | 21.07 | 12.48 | 28.71 |
| -27+19 | 15.30 | 21.32 | 15.05 | 36.12 | 18.49 | 47.20 |
| -19+13.5 | 27.72 | 49.04 | 9.66 | 45.78 | 18.88 | 66.08 |
| -13.5+9.6 | 24.54 | 73.58 | 18.14 | 63.91 | 17.30 | 83.38 |
| -9.6+6.8 | 16.91 | 90.48 | 19.39 | 83.30 | 9.45 | 92.83 |
| -6.8+4.8 | 7.13 | 97.61 | 10.98 | 94.27 | 4.72 | 97.54 |
| -4.8+3.4 | 1.56 | 99.17 | 3.76 | 98.03 | 1.60 | 99.14 |
| -3.4+2.4 | 0.46 | 99.63 | 1.37 | 99.40 | 0.55 | 99.69 |
| -2.4+1.75 | 0.15 | 99.77 | 0.43 | 99.83 | 0.22 | 99.91 |
| -1.75 | 0.22 | 100.00 | 0.17 | 100.00 | 0.10 | 100.00 |

IsaMill or Tower mill technology, and a drum magnetic separator or magnetic separation column could be applied to obtain the high-quality concentrate and iron concentrate. The tailings should be beneficiated to recover ilmenite. Although the process outlined would involve some capital costs and increase operating costs, the majority of $TiO_2$ in the high-quality concentrate would be extracted and used to produce titanium pigment. Due to the huge resource and high value of titanium dioxide, this would add considerable value. Thus, this technique utilizes cost-effective practices and a sufficiently flexible procedure for the comprehensive utilization of vanadium titanomagnetite.

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