Selective Separation of Perovskite (CaTiO_3) from Titanium Bearing Slag Melt by Super Gravity

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Based on the precipitation experiment results of titanium bearing molten slag during cooling liquation, perovskite (CaTiO_3) was the first precipitated phase, and 1 593–1 563 K was the advantageous precipitation temperature of perovskite from the molten simulated slag with a basicity of 1.30. So if perovskite could be separated from the slag melt at this temperature range, at which perovskite becomes a solid state while other minerals form into the molten slag, it would be beneficial for the solid-liquid separation between them. Hence, selective separation experiments of perovskite from titanium bearing slag melt with a basicity of 1.30 at 1 578 K by super gravity were carried out in this study, and the results confirmed that it was an effective method. In this process, the molten slag moved along the super gravity direction and went through the filter and then concentrated as the slag phase in the bottom crucible, in which it was practically impossible to find any perovskite grains. In contrast, all the perovskite grains were intercepted by the filter and concentrated as the perovskite phase on the filter, which appeared as the typical dendrite structure. Consequently, after super gravity separation with gravity coefficient of $G = 660$ to $G = 840$ at 1 578 K for 10 minutes, the mass fraction of TiO_2 in the perovskite phase was up to 46.36 wt%, whereas that of the slag phase was only 8.77 wt%. In this case, the recovery ratio of Ti in the perovskite phase was up to 78.17%.

KEY WORDS: super gravity; solid-liquid separation; perovskite (CaTiO_3); titanium bearing slag melt; filter.

1. Introduction

There are multitudes of vanadium-titanium magnetite resources in Panxi region of China, which is a typical multiple-element symbiotic composite ore consist of vanadium, titanium and iron.^{1,2)} Nevertheless, this kind of ore resource has been mainly adopting Blast Furnace iron making process for refining of iron, but the extractions of vanadium and titanium are inadequate. During Blast Furnace iron making process, the iron and vanadium will be reduced by carbon and transform into the vanadium bearing hot metal, whereas the titanium migrates to the titanium bearing blast furnace slag.^{3,4)} As a result, the titanium bearing slag containing about 20 wt% of TiO_2 has been accumulating 70 million tons, and it is increasing at a rate of 3 million tons per year. However, the conventional beneficiation methods currently face great difficulties in recovering titanium from the slag due to the complicated mineralogy and very fine mineral dissemination of the titanium mineral phases.⁵⁾ So far, many technical routes have been proposed across the world for titanium separation and comprehensive utilization of the titanium bearing slag, including the acid leaching process,⁶⁾ the alkaline leaching process⁷⁾ and the high temperature carbonization process.⁸⁾

The precipitation first and then physical separation process of the titanium phases from titanium bearing slag was first proposed by Sui.⁹⁾ As for the precipitation process of titanium phases, the effects of slag basicity,^{10,11)} oxidation,¹²⁾ and different additives^{13–15)} on the precipitation behavior of perovskite from titanium bearing slag melt were investigated by different authors. They found that appropriate increase of the slag basicity, oxidize the slag and add some CaF_2 , MnO , Fe_2O_3 and Si-Fe powders into the slag were beneficial for the precipitation of perovskite from the titanium bearing slag melt.

Currently, precipitation of perovskite from titanium bearing slag melt carried out at high temperature is always combined with some physical methods to further separate perovskite from the slag at room temperature, such as the fine grinding process, flotation and gravity separation process.^{15,16)} However, it ran into one problem that the perovskite and other mineral phases could not be effectively separated from the slag by the conventional beneficiation methods. So if the separation temperature could be increased to the precipitation temperature of perovskite, at which the perovskite changes to a solid state while the other minerals form into the molten slag, it would be beneficial for the separation of solid-particle and liquid melt. Nevertheless, it was impossible to accomplish the solid-liquid separation at the conventional conditions, which was proven in this study.

Inspired by the successful application of super gravity

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technology in preparation of functionally gradient materials,^{18,19)} removing inclusion from alloy melt,^{20,21)} and iron-slag separation at low temperature,^{22,23)} it is possible to separate perovskite from the titanium bearing slag melt by super gravity. Hence, the precipitation behavior of the titanium bearing molten slag during cooling liquation was first investigated in this study, and the advantageous temperature range for precipitation of perovskite (CaTiO_3) was determined according to the variations of diffraction peak intensity and microstructure at different temperatures. On this basis, the experiments on selective separation of perovskite from the slag melt by super gravity were carried out at this temperature range, and the feasibility of the process was verified. Simultaneously, the effect of super gravity field on the microstructures, mineral compositions, components and recovery ratio of Ti in the separated products were also investigated.

2. Experimental

2.1. Material

The chemical compositions and mineralogical compositions of the titanium bearing blast furnace slag after natural cooling process from the Panzhihua Iron and Steel Cor-

poration of China are shown in **Table 1** and **Fig. 1**. The mass fractions of TiO_2 and CaO were 23.35 wt% and 28.64 wt%, and the mineral phases mainly included perovskite (CaTiO_3), pyrope ($\text{Mg}_3\text{Al}_2\text{Si}_3\text{O}_{12}$) and diopside ($\text{Ca}(\text{Mg},\text{Al})(\text{Si},\text{Al})_2\text{O}_6$). With the help of SEM-EDS analysis shown in **Fig. 2** and **Table 2**, it was obvious that the titanium (Ti)

Fig. 1. XRD patterns of the titanium bearing slag.

Table 1. Chemical compositions (wt%) of the experimental materials.

Composition	CaO	SiO ₂	TiO ₂	Al ₂ O ₃	MgO	TFe	V ₂ O ₅	MnO ₂	P ₂ O ₅	S	Others	Basicity
Titanium bearing slag	28.64	25.44	23.35	11.09	7.06	2.82	0.20	0.75	0.02	0.12	0.50	1.13
Simulated slag	33.07	25.44	23.35	11.09	7.06	—	—	—	—	—	—	1.30

Table 2. Energy-dispersive spectrum data (wt%) of the mineral phases in the titanium bearing slag.

No.	Position	Ca	Si	Ti	V	Fe	Al	Mg	O
Pt. 1	Fig. 2(a)	32.80	—	39.24	—	—	—	—	27.96
Pt. 2	Fig. 2(b)	32.82	—	41.48	0.23	—	—	—	25.48
Pt. 3	Fig. 2(a)	5.40	23.33	4.21	—	—	10.71	13.02	43.33
Pt. 4	Fig. 2(b)	5.76	22.35	4.02	—	—	10.04	13.21	44.62
Pt. 5	Fig. 2(a)	23.76	12.35	13.02	—	—	10.04	6.21	34.62
Pt. 6	Fig. 2(b)	23.95	14.08	13.37	0.03	—	11.74	5.27	31.57
Pt. 7	Fig. 2(b)	—	—	—	—	100	—	—	—

Fig. 2. SEM photographs of the titanium bearing slag: (a) 500 $\times$, (b) 1 000 $\times$.

mainly existed in the form of perovskite and diopside, whereas the fine perovskite grains with dendritic shape uniformly distributed among the pyrope and diopside as well as some residual iron grains.

2.2. Apparatus

The super gravity field used in the separation experiments of perovskite from the titanium bearing slag melt was generated by the centrifugal apparatus as illustrated in Fig. 3. The heating furnace was heated by resistance wire with the isothermal zone of 80 mm long, and the temperature was controlled by a program controller with an R-type thermocouple, within the observed precision range of ± 3 K. Moreover, the heating furnace and a counterweight were fixed symmetrically onto the centrifugal rotor. Once the centrifugal rotor started running, they changed from vertical to horizontal and rotated therewith.

2.3. Experimental and Analytical Procedure

2.3.1. Precipitation Experiment of Molten Slag during Cooling Liquefaction

To investigate the precipitation behavior of different mineral phases in the molten slag, the simulated slag was first prepared by mixing chemical agents powders based on the main compositions of titanium bearing slag as shown in Table 1. Furthermore, according to the thermodynamic data of the reaction $\text{TiO}_2 + \text{CaO} = \text{TiCaO}_3(\text{s})$, raising the slag basicity (CaO/SiO_2) could increase the activity of CaO in the slag that promoted the forward reaction of perovskite precipitation. But increasing the slag basicity to over 1.30 would also facilitated the precipitation of calcium silicate according to the experiments of Li.¹¹⁾ Hence, the slag basicity was adjusted from 1.13 to 1.30 by adding CaO into the simulated slag, and the precipitation experiments by using of simulated slag were carried out. The prepared slags were put into some graphite crucibles lined with molybdenum sheet, and heated to 1 773 K in high purity argon in muffle furnace to avoid the possible reduction of titanium bearing

minerals. After heating at the constant temperature for 30 minutes to make the slag fully melted, the temperatures were rapidly decreased at a cooling rate of 20 K/min, and then the melted slags were slowly cooled at 1 743–1 713 K, 1 713–1 683 K, 1 683–1 653 K, 1 653–1 623 K, 1 623–1 593 K, 1 593–1 563 K, 1 563–1 533 K, 1 533–1 503 K, 1 503–1 473 K, 1 273–1 243 K, 1 243–1 213 K or 1 213–1 183 K with a cooling rate of 0.5 K/minute for 60 minutes, respectively. After that, the graphite crucibles were taken out and water-quenched. The samples obtained by cooling liquation at different temperature ranges were sectioned longitudinally along the center axis, and measured by metallographic microscope and X-ray diffraction (XRD) to gain the microstructures and mineral compositions of the precipitated phases.

2.3.2. Separation Experiment of Perovskite Form Slag Melt by Super Gravity

On the basis of the earlier precipitation experiments, titanium bearing slag with a basicity of 1.30 was melted at 1 773 K and then cooled slowly at 1 593–1 563 K for 60 minutes to promote the further precipitation of perovskite. After that, the sample was water-quenched and used for the separation experiments by super gravity. 20 g of the sample was put onto a graphite fiber felt of 10 mm height with the pore size less than 0.01 mm, and held on a graphite filter (I.D. 19 mm) with the pore size of $d = 0.5$ mm, and then put into a graphite crucible, which was reheated to 1 578 K in the heating furnace of centrifugal apparatus to make the other minerals change to molten slag while the perovskite remain in solid state. And then, the centrifugal apparatus was started and adjusted to the specified angular velocity of 1 638 RPM, namely $G = 660$ to $G = 840$ in the upper part and bottom part of the sample at the constant temperature for 10 minutes. After that, the centrifugal apparatus was shut off, and the graphite crucible was taken out and water-quenched. Simultaneously, the parallel experiment was carried out at 1 578 K for 10 minutes without super gravity

Fig. 3. Sketch of the centrifugal apparatus: (1) counterweight, (2) centrifugal axis, (3) conductive slipping, (4) thermocouple, (5) insulating layer, (6) temperature controller, (7) graphite fiber felt and filter, (8) graphite crucible, (9) perovskite grains, (10) perovskite phase, (11) slag phase.

treatment. The gravity coefficient was calculated as the ratio of super-gravitational acceleration to normal-gravitational acceleration *via* Eq. (1).

$$G = \frac{\sqrt{g^2 + (\omega^2 R)^2}}{g} = \frac{\sqrt{g^2 + \left(\frac{N^2 \pi^2 R}{900}\right)^2}}{g} \dots\dots\dots (1)$$

where G is the gravity coefficient, g is the normal-gravitational acceleration ($g = 9.80 \text{ m/s}^2$), ω is the angular velocity ($\text{rad} \times \text{s}^{-1}$), N is the rotating speed of the centrifugal (RPM), and R is the distance from the centrifugal axis to the upper part and bottom part of the sample ($R = 0.22 \text{ m}$ to 0.28 m).

After the separation experiments by super gravity, the samples that held on and went through the filter were sectioned longitudinally into two parts along the direction of super gravity. One part was measured by scanning electron micrograph and energy-dispersive spectrum (SEM-EDS) for analyzing the microstructures of the separated samples. The other part was crossly divided along the filter and characterized by XRD and XRF methods to determine the mineral compositions and chemical components in the separated samples. After that, the recovery ratio of Ti was calculated *via* Eq. (2).

$$\varepsilon_{\text{Ti}} = \frac{m_P \times \omega_{\text{Ti-P}}}{m_P \times \omega_{\text{Ti-P}} + m_S \times \omega_{\text{Ti-S}}} \times 100\% \dots\dots\dots (2)$$

where ε_{Ti} is the recovery ratio of Ti in the separated perovskite phase, m_P and m_S are the mass fractions of the separated perovskite phase and slag phase, and $\omega_{\text{Ti-P}}$ and $\omega_{\text{Ti-S}}$ are the mass fractions of Ti in the separated perovskite phase and slag phase.

3. Results and Discussion

3.1. Precipitation Behavior of Molten Simulated Slag during Cooling Liquefaction

The mineral compositions of the molten simulated slag with a basicity of 1.30 after cooling at different temperature ranges with a cooling rate of 0.5 K/min are shown in **Fig. 4**. Obviously, perovskite was the first precipitated phase from

Fig. 4. XRD patterns of the molten slag after cooling at different temperature ranges.

the molten slag, whose diffraction peak intensity increased with temperature decreased from 1743 K to 1563 K , and the peak value appeared significantly in 1593 – 1563 K . When temperature decreased to 1563 – 1553 K , the second precipitated phase (pyrope) started to appear and the diffraction peak intensity increased with decreasing temperature. When temperature further decreased to 1473 – 1443 K , the third precipitated phase (diopside) also appeared. However, with the successive precipitation of pyrope and diopside, the viscosity of molten slag increased, and the migration rate of Ca^{2+} and TiO_3^{2-} in the melt decreased, and so the nucleation and growth of perovskite crystals was blocked, so the diffraction peak intensity of perovskite decreased with the temperature further decreased from 1563 K to 1383 K .

Furthermore, the microstructures of above samples are shown in **Fig. 5**. As shown in Figs. 5(a) and 5(b), white perovskite grains changed from fine floc or needle shaped crystals to larger equiaxed crystals with the temperature decreased from 1743 K to 1563 K , and the larger size of perovskite grains appeared in 1593 – 1563 K . When it came to the temperature below 1563 K as shown in Figs. 5(c) and 5(d), the dark grey pyrope and light grey diopside grains appeared successively at 1563 – 1533 K and 1473 – 1443 K , which uniformly included among the first precipitated perovskite grains. According to the earlier XRD analysis and by comparing the microstructures of perovskite grains at different temperature ranges, it was indicated that the 1593 – 1563 K was the advantageous precipitation temperature of perovskite from the molten slag with the compositions of $33.07 \text{ wt\%}$ of CaO , $25.44 \text{ wt\%}$ of SiO_2 , $23.35 \text{ wt\%}$ of TiO_2 , $11.09 \text{ wt\%}$ of Al_2O_3 and $7.06 \text{ wt\%}$ of MgO .

3.2. Separation of Perovskite Phase from Titanium Bearing Slag Melt by Super Gravity

3.2.1. Macrostructures of the Samples Obtained by Super Gravity Separation

The vertical sections of the sample obtained by super gravity separation with gravity coefficient of $G = 660$ to $G = 840$ compared with the parallel sample with gravity coefficient of $G = 1$ are illustrated in **Fig. 6**. It was obvious

Fig. 5. Microstructures of the molten slag after cooling liquefaction at different temperature ranges: (a) 1743 – 1713 K , (b) 1593 – 1563 K , (c) 1563 – 1533 K , (d) 1473 – 1443 K .

that the layered structures appeared significantly in the sample obtained by super gravity separation. In a macroscopic view, the sample that held on the filter appeared as a black, porous structure, whereas the sample that went through the filter presented in a grey, glassy state. In contrast, the whole sample that was held on the filter remained in normal gravity, and a uniform structure presented in it.

3.2.2. Mineral Compositions of the Separated Samples

With the help of XRD analysis, the mineral compositions of samples that held on and went through the filter obtained by super gravity separation with gravity coefficient

Fig. 6. Vertical sections of the sample obtained by super gravity separation compared with the parallel sample in normal gravity: (a) $G = 1$, (b) $G = 660$ to $G = 840$.

Fig. 7. XRD patterns of the samples that held on and went through the filter obtained by super gravity separation compared with the parallel sample in normal gravity.

of $G = 660$ to $G = 840$ compared with the parallel sample are shown in Fig. 7. Obviously, compared with the parallel sample comprised of perovskite, pyrope and diopside, after super gravity separation, only the diffraction peak of perovskite appeared in the sample held on the filter, but the diffraction peaks of pyrope and diopside appeared in the sample went through the filter. It gave further evidence that the perovskite transformed into solid particle while the other minerals formed into the molten slag at the separating temperature, and the two phases showed different physical states. As a driving force, the super gravity forced the molten slag moved along the super gravity direction and went through the filter, and then concentrated as slag phase in the bottom crucible; whereas the perovskite grains with the size of 0.05–0.12 mm were intercepted by the filter with the pore size less than 0.01 mm, and concentrated as perovskite phase on the filter.

3.2.3. Microstructures of the Separated Samples

In order to further investigate the microstructures of the separated samples obtained by super gravity, the SEM-EDS analysis of the samples that held on and went through the filter were carried out. According to the energy-dispersive spectrum data given in Table 3, the perovskite phase was comprised of calcium (26.69–27.84 wt%), titanium (32.61–33.82 wt%) and oxygen (38.31–40.15 wt%), whereas the slag phase mainly consisted of pyrope and diopside. As shown in Fig. 8, the separated perovskite phase appeared as the typical dendrite structure from a microscopic view, and the secondary dendrite were very developed with some fine iron grains distributed on it. In contrast, the separated slag phase presented in a compact structure, in which it was practically impossible to find any perovskite grains. It gave further evidence that all the perovskite grains were effectively separated from the molten slag phase by using a driving force of super gravity.

Fig. 8. SEM photographs of the separated samples: (a) the sample that held on the filter, (b) the sample that went through the filter.

Table 3. Energy-dispersive spectrum data (wt%) of the mineral phases in the separated samples.

No.	Position	Ca	Si	Ti	V	Fe	Al	Mg	O
Pt. 1	Fig. 8(a)	26.69	—	32.61	0.55	—	—	—	40.15
Pt. 2	Fig. 8(a)	27.48	—	33.82	0.39	—	—	—	38.31
Pt. 3	Fig. 8(a)	4.95	—	2.24	—	90.29	—	—	2.52
Pt. 4	Fig. 8(b)	18.63	15.64	3.32	—	—	15.37	7.45	39.59
Pt. 5	Fig. 8(b)	4.44	20.54	4.83	—	—	11.47	13.44	45.28

Table 4. XRF results of the separated samples obtained by super gravity separation compared with the parallel sample (wt%).

Sample	TiO ₂	CaO	SiO ₂	Al ₂ O ₃	MgO	Fe ₂ O ₃
Perovskite phase	46.36	36.13	7.13	4.22	3.28	2.88
Slag phase	8.77	28.85	35.9	14.86	10.67	0.95
Parallel sample	23.38	31.05	25.56	11.21	7.13	1.67

Table 5. Recovery ratio of Ti in the separated samples obtained by super gravity separation.

Sample	Mass fraction (%)	Chemical composition of TiO ₂ (wt%)	Recovery ratio of Ti (%)
Perovskite phase	40.4	46.36	78.17
Slag phase	59.6	8.77	21.83

3.2.4. Components and Recovery Ratio of Ti in the Separated Samples

Measured by XRF method, the chemical compositions of the separated samples obtained by super gravity compared with the parallel sample are shown in **Table 4**. After dealing with the titanium bearing slag melt with a basicity of 1.30 by super gravity with the gravity coefficient of $G = 660$ to $G = 840$ at 1 578 K for 10 minutes, the mass fraction of TiO₂ in the perovskite phase was up to 46.36 wt%, whereas that of the slag phase was only 8.77 wt%. Simultaneously, the mass fractions of other minerals (SiO₂, Al₂O₃ and MgO) in the slag phase were up to 35.9 wt%, 14.86 wt% and 10.67 wt%, whereas that of the perovskite phase were only 7.13 wt%, 4.22 wt% and 3.28 wt%, respectively. This indicated that super gravity field effectively increased the diffusion coefficient of the molten slag, and forced it to move through the filter and separate from the perovskite grains. Thus, it was definitely beneficial for the separation of perovskite phase and slag phase. Consequently, in the case of $G = 660$ to $G = 840$, $T = 1\,578\text{ K}$ and $t = 10\text{ min}$, the recovery ratio of Ti in the perovskite phase was up to 78.17% as listed in **Table 5**.

4. Conclusions

Based on the precipitation experiment results of molten slag during cooling liquation at a cooling rate of 0.5 K/min, perovskite was the first precipitated phase, and pyrope and diopside were the second and third precipitated phases with temperature decreased to 1 563–1 553 K or 1 473–1 443 K, respectively. Combined with the variations of diffraction peak intensity and microstructure of perovskite at different temperature ranges, 1 593–1 563 K was the advantageous precipitation temperature of perovskite from the molten

simulated slag with a basicity of 1.30.

The experiment results confirmed that separating the perovskite from titanium bearing slag melt by super gravity at 1 578 K was an effective method. The molten slag moved along the super gravity direction and went through the filter and then concentrated as slag phase in the bottom crucible, in which it was practically impossible to find any perovskite grains. In contrast, all the perovskite grains were intercepted by the filter and concentrated as perovskite phase on the filter, which appeared typical dendrite structure.

The super gravity field was definitely beneficial for the solid-liquid separation between perovskite and slag phases from the slag melt with a basicity of 1.30. In the case of $G = 660$ to $G = 840$, $T = 1\,578\text{ K}$ and $t = 10\text{ min}$, the mass fraction of TiO₂ in the perovskite phase was up to 46.36 wt%, whereas that of the slag phase was only 8.77 wt%. In this case, the recovery ratio of Ti in the perovskite phase was up to 78.17%.

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