Isothermal Enrichment of P-Concentrating Phase from CaO–SiO₂–FeO–MgO–P₂O₅ Melt with Super Gravity

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In this study, the investigation on the isothermal enrichment of P-concentrating phase from CaO–SiO₂–FeO–MgO–P₂O₅ melt with super gravity was carried out. The results show that there was an obvious stratification appearing in the sample after centrifugal enrichment. The upper part of the sample is loose and porous, while the lower part is smooth and compact. With the help of metallographic microscopy and X-ray diffraction, it is found that the P-concentrating phase gathered in the upper part, while it is hard to find any P-concentrating phase in the lower part of the sample. In addition, the volume fraction and equivalent diameter of P-concentrating phase present gradient distribution along the direction of super gravity. After centrifugal enrichment, the mass fraction of P₂O₅ in the concentrate is up to 4.92%, while that in the tailing is just 1.08%. The recovery ratio of P₂O₅ in the concentrate is up to 72.62% with the gravity coefficient G=600, time t=20 minutes and temperature T=1 623 K.

KEY WORDS: super gravity; P-concentrating phase; steelmaking slag; centrifugal separation.

1. Introduction

It is well known that steelmaking slag is one of the major byproducts in steelmaking process, whose productivity equals to 10–20% crude steel productivity. Due to its underutilization, large quantities of steelmaking slag are disposed as wastes, causing tremendous land resource waste and environment pollution. However, many useful components such as FeO, Fe₂O₃, MgO, MnO and CaO exist in steelmaking slag. If it can be reused in iron- and steelmaking process, the cyclic utilization of steelmaking slag in steelmaking process, the cyclic utilization of steelmaking slag and recovery of phosphorus from steelmaking slag has been recognized, no effective method has yet been developed with carbon by microwave irradiation, but this method has not been developed to the point where its practical application is assured. Considering the significant difference of magnetic properties between the P-concentrating phase and Fe-concentrating phase in slag, have separated P-concentrating phase from steelmaking slag with the aid of a strong magnetic field. However, due to its micron level size and embedded distribution in the matrix, the P-concentrating phase could not be separated effectively by magnetic field. Ono et al. took the advantage of the density difference between P-concentrating phase and the residual melt to remove P-concentrating phase by floating separation. But the rigorous production conditions, such as high temperature, extremely slow cooling speed and more than 30% FeO content, restricted the industrialization of this method. Morita et al. proposed a method in which phosphorus was extracted through the treatment of Na₂CO₃ flux from the Fe–P–C alloy obtained as a result of the reduction of the slag with carbon by microwave irradiation, but this method has not been developed to the point where its practical application is assured. Although the significance of the separation and recovery of phosphorus from steelmaking slag has been recognized, no effective method has yet been developed for this process. It is necessary, therefore, to find a novel method to separate phosphorus from steelmaking slag in order to increase slag recycling. Considering the fact that phosphorus in steelmaking slag is mostly enriched in dicalcium silicate precipitating early during solidification and its density is smaller than the residual melt, the P-concentrating phase can be separated by super gravity. In addition, super gravity technology has been successfully applied in the separation and recovery of various valuable elements from different kinds of slags, removing of impurities from alloy melt and preparation of functionally gradient

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Based on the characteristics above, it would be possible to separate P-concentrating phase precipitating early from steelmaking slag by super gravity. In this study, a novel method of super gravity was introduced to separate P-concentrating phase from CaO–SiO$_2$–FeO–MgO–P$_2$O$_5$ melt. The influence of the gravity coefficient, centrifugal time and temperature on the separation effects of P-concentrating phase and the recovery ratio of P$_2$O$_5$ were investigated.

2. Experimental

2.1. Slag Preparation

The CaO–SiO$_2$–FeO–MgO–P$_2$O$_5$ slag was prepared by mixing the reagent-grade CaO, SiO$_2$, MgO, 3CaO·P$_2$O$_5$ and synthesized FeO (obtained by reducing Fe$_3$O$_4$ with 5 L/min flow rate of mixed CO and CO$_2$ with 1:1 proportion at the temperature of 1 173 K). The chemical composition is listed in Table 1. The P$_2$O$_5$ and MgO content in the slag was 2.5% and 3% closed that in actual steelmaking slag. The slag basicity (CaO/SiO$_2$) was adjusted to 2.0 and the mass fraction of FeO was added up to 30% to reduce the melting point and viscosity of the slag melt at experimental temperature. The chemical powders were fully mixed and then placed into a magnesia crucible (60×120 mm) and heated in the muffle furnace to 1 773 K with a rate of 10 K/min in Ar atmosphere. After keeping for 30 minutes, the slag was cooled to 1 623 K with a rate of 3 K/min and kept at this temperature for 30 minutes to ensure the P-concentrating phase to fully precipitate, and then the sample was quenched in water. However, due to the corrosion of magnesia crucible by the slag, the chemical compositions of the prepared slag had a little difference from the initial slag, as shown in Table 1.

2.2. Sample Preparation and Analytical Procedure

The super gravity field was generated by the centrifugal apparatus whose schematic diagram was shown in Fig. 1. The heating furnace balanced with a counterweight across the rotation axis was fixed into a centrifugal rotor. The gravity coefficient, a ratio of super-gravitational acceleration to gravitational acceleration, was calculated by Eq. (1).

$$G = \frac{\sqrt{g^2 + \left(\frac{\omega^2 r}{900}\right)^2}}{g}$$

where $N$ is the rotating speed of the centrifugal, r/min; $\omega$ is the angular velocity, rad/s; $r$ is the distance from the centrifugal axis to the sample, 0.25 m; $g$ is normal gravitational acceleration, 9.8 m/s$^2$. When $N=0$, $G$ is equal to 1.

Thirty grams of prepared slag was put into a magnesia crucible (I.D.: 18 mm and H.: 60 mm) and heated to specified temperature. The temperature of the furnace was kept for 10 minutes at this temperature to ensure the slag being in the state of solid-liquid mixture and then the centrifugal apparatus was started and adjusted to the specified angular velocity. The centrifugal apparatus was not turned off until the target time and then the sample was taken out to cool in air. The sample obtained by super gravity was divided into halves along the center axis. One part was polished and then taken optical micrographs randomly along the specific location (average 20 fields) by metallographic microscope (9XB-PC type). The image analyzer software (Image-Pro plus 6.0) was used to gain the volume fraction of P-concentrating phase by calculating the average value of areal fraction of 20 fields. At the same time, the equivalent diameter, namely the diameter of a circle which has the equivalent area of P-concentrating phase, was measured by the line intercept method. The other one was cut along the sample stratified interface into two parts, and both of them

| Table 1. Chemical composition of the slag (mass fraction, %). |
|-------------------------------|-----------------|----------------|--------------|-----------------|----------------|
| Slag | CaO | SiO$_2$ | FeO | MgO | P$_2$O$_5$ | Sum | Basicity |
| Initial | 43 | 21.5 | 30 | 3 | 2.5 | 100 | 2.0 |
| Prepared | 42.7 | 21.4 | 29.8 | 3.5 | 2.5 | 100 | 1.9 |

Fig. 1. Schematic diagram of centrifugal separation apparatus; 1. Counterweight; 2. Centrifugal axis; 3. Base; 4. Magnesia crucible; 5. Slag melt; 6. Resistance coil; 7. P-concentrating phase before centrifugal enrichment; 8. P-concentrating phase after centrifugal enrichment; 9. Thermocouple; 10. Horizontal rotor; 11. Conductive slipping; 12. Temperature controller.
were characterized by X-ray fluorescence (XRF-1800X from Shimadzu Corporation) and X-ray diffraction (TTRIII from Rigaku Corporation) to determine the chemical composition and the mineral phases, respectively. At the same time, the parallel sample was prepared at 1 623 K for 20 minutes without centrifugal treatment.

3. Results and Discussion

3.1. Macro and Micro Structure of the Samples

Figure 2 shows the macro structure of the sample obtained by centrifugal enrichment with the gravity coefficient \( G = 600 \), temperature \( T = 1 623 \text{ K} \) and time \( t = 20 \) minutes, compared with the parallel sample under the conditions of gravity coefficient \( G = 1 \), temperature \( T = 1 623 \text{ K} \) and time \( t = 20 \) minutes. As shown in Fig. 2, the sample after centrifugal enrichment appears obvious stratification, while the uniform structure presents in the parallel sample. The surface comparison of two stratified areas of the sample obtained by super gravity was shown in Fig. 3, the upper part of the sample after centrifugal enrichment is loose and porous, while the lower part is smooth and compact. In order to further investigate the microstructure of the sample after centrifugal enrichment, the positions of six areas in equal interval (5 mm) in sequence from the upper part characterized by the metallographic microscopy was shown in Fig. 2(b), and the corresponding microstructures was shown in Fig. 4. The results show that the upper part from area (a) to (d) is mainly composed of P-concentrating phase which appeared to be dark gray and oval particles in the micrograph. The size increased with the area approaching to the top of the sample, namely the gradient size distribution along the direction of super gravity after centrifugal enrichment as well as the volume fraction. However, it was scarcely possible to find any P-concentrating phase with the similar size like that in the upper part from the area (e) to (f). The stratified region locates in the area (d).

As mentioned above, the dicalcium silicate will early precipitate with tricalcium phosphate from steelmaking slag to form P-concentrating phase during solidification process.\textsuperscript{15) When the temperature was 1 623 K, the slag for the composition shown in Table 1 located in the solid-liquid co-existence zone of ternary phase diagram of CaO–SiO\textsubscript{2}–FeO, and the dicalcium silicate companions with the most of tricalcium silicate precipitated from the slag to form solid solution, namely P-concentrating phase. At normal gravity (\( G = 1 \)), although the density difference between P-concentrating phase and residual melt exists practically, the P-concentrating phase could not be separated from melt because of the large viscosity, therefore, the uniform structure presents in Fig. 2(a). However, super gravity as an intense separation way could control the distribution of different phases, and the density of P-concentrating phase is lighter than that of residual melt, so they could be driven by super gravity to the top of the sample, as shown in the Fig. 4. When the sample was cooled to room temperature, there was no efficient liquid between P-concentrating solid particles to supply shrink, which led to loose and porous structure in the upper part. While residual melt filled the lower part to form smooth and compact surface. Therefore, there was an obvious stratification in Fig. 2(b). The larger pore in the middle of the sample was caused by solidification shrinkage of the residual melt.

3.2. Volume Fraction and Equivalent Diameter of P-Concentrating Phase

Tables 2, 3 and 4 present the variations of volume fraction of P-concentrating phase in different areas of the samples with different gravity coefficients at \( t = 20 \) minutes, \( T = 1 623 \text{ K} \), different centrifugal time at \( G = 600 \), \( T = 1 623 \text{ K} \) and different temperature at \( G = 600 \), \( t = 20 \) minutes, respectively. The corresponding equivalent diameter of P-concentrating phase in different areas of the samples is shown in Figs. 5, 6 and 7. From these tables, the volume fraction of P-concentrating phase after centrifugal enrichment gradually increases from...
area (f) to area (a) along the direction of super gravity, and the peak value appears in area (a). The bigger, longer and higher the gravity coefficient, centrifugal time and temperature are, the more obvious the gradient distribution of volume fraction is, as shown in Tables 2, 3 and 4. It is also observed from Figs. 5, 6 and 7 that the equivalent diameter of the P-concentrating phase increases gradually from area (d) to (a) after centrifugal enrichment.

As shown in Fig. 4, P-concentrating phase in the sample precipitated in the form of equiaxed crystal and presented as nearly circular solid particles. The motion of solid particles in viscous liquid under centrifugal force can be explained by Stokes’ law. Therefore, the motion velocity of P-concentrating phase in slag melt obeys the following equation:

$$\frac{dx}{dt} = \frac{(\rho_p - \rho_m) G g D_p^2}{18 \eta} \quad \text{(2)}$$

Fig. 4. Micrographs of six areas in equal interval (5 mm) obtained by centrifugal enrichment at \(G = 600, t = 20\) minutes, \(T = 1\,623\) K.

Fig. 5. Effects of different gravity coefficients on equivalent diameters of P-concentrating phase in different areas of the samples at \(t = 20\) minutes, \(T = 1\,623\) K (volume fraction, %).

Fig. 6. Effects of different time on equivalent diameters of P-concentrating phase in different areas of the samples at \(G = 600, T = 1\,623\) K (volume fraction, %).
where \( \frac{dx}{dt} \), \( \rho \), \( G \), \( g \), \( D \) and \( \eta \) are velocity, density, gravity coefficient, normal gravitational acceleration, particle diameter and viscosity of the slag melt, respectively. The subscripts ' \( p \) ' and ' \( m \)' denote particle and matrix, respectively.

It is obvious from Eq. (2) that the motion velocity of P-concentrating phase in the slag melt is proportional to the gravity coefficient, the square of particle diameter and the reciprocal of viscosity. The P-concentrating particles under high gravity coefficient have a larger velocity than that under low gravity coefficient. On the other hand, the P-concentrating phase of large size moves faster than the small ones in case of same gravity coefficient. Increasing temperature is equivalent to reducing the melt viscosity, which can reduce the movement resistance of P-concentrating phase and make it move faster in higher temperature. Therefore, the large P-concentrating phase particles accumulated in the upper part of the sample, while small ones in the middle part after centrifugal enrichment. The volume fraction of the P-concentrating phase in the upper part of the sample will increase with the increase of gravity coefficient, centrifugal time and temperature.

### 3.3. Components of Stratified Sample and Removal Ratio of \( \text{P}_2\text{O}_5 \) after Centrifugal Enrichment

As shown in Fig. 8, there are mainly dicalcium silicate and magnesium iron oxide phases in the parallel sample. After centrifugal enrichment with the parameter of \( G = 600 \), \( t = 20 \) minutes, \( T = 1623 \) K, a large quantity of dicalcium silicate phase precipitating with tricalcium phosphate at this temperature moved up to the upper part of the sample as concentrate, while the residual melt forming the magnesium iron oxide and dicalcium silicate at room temperature accumulated at the lower part of the sample as tailing. Table 5 shows the X-ray fluorescence result of stratified samples under the conditions of gravity coefficient \( G = 600 \), \( t = 20 \) minutes, \( T = 1623 \) K. The mass fraction of \( \text{P}_2\text{O}_5 \) in the concentrate is up to 4.92%, while that in the tailing is just 1.08%. As shown in Table 6, the recovery ratio of \( \text{P}_2\text{O}_5 \) in the concentrate is up to 72.62%, which can be calculated via Eq. (3).

\[
R_c = \frac{m_c \times \omega_c}{m_t \times \omega_t + m_c \times \omega_c} \times 100\% \quad \text{(3)}
\]

![Fig. 7. Effects of different temperatures on equivalent diameters of P-concentrating phase in different areas of the samples at \( G = 600 \), \( t = 20 \) minutes (volume fraction, %).](image)

![Fig. 8. X-ray diffraction of the sample obtained by centrifugal enrichment and the parallel sample.](image)
where $R_c$ is recovery ratio of $P_2O_5$ in the concentrate, $m_c$ and $m_t$ are mass of the concentrate and tailing, $\alpha_c$ and $\alpha_t$ are mass fraction of $P_2O_5$ in the concentrate and tailing, respectively.

### 4. Conclusions

It was experimentally confirmed that the super gravity was a novel effective method to isothermally enrich P-concentrating phase from CaO–SiO$_2$–FeO–MgO–P$_2$O$_5$ melt. The P-concentrating phase driven by super gravity moved to the upper part of the sample, while the residual melt gathered in the lower part, and there was an obvious stratification appearing in the sample.

The volume fraction and equivalent diameter along the direction of super gravity present gradient distributions, with large volume fraction and equivalent diameter of P-concentrating phase appearing in the upper part of the sample, while small ones in the middle part after centrifugal enrichment at $G=600$, $t=20$ minutes, $T=1,623$ K.

The concentrate obtained by the gravity coefficient $G=600$, time $t=20$ minutes and temperature $T=1,623$ K was mainly composed of P-concentrating phase, in which the mass fraction of $P_2O_5$ is up to 4.92%, while that in the tailing is just 1.08%. Through calculation, the recovery ratio of $P_2O_5$ in the concentrate is up to 72.62%.

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### Table 5

| Products     | CaO  | SiO$_2$ | FeO  | MgO  | P$_2$O$_5$ |
|--------------|------|---------|------|------|------------|
| Concentrate  | 53.71| 23.65   | 13.65| 4.06 | 4.92       |
| Tailing      | 33.89| 18.52   | 42.52| 3.91 | 1.08       |
| Parallel     | 44.42| 20.92   | 29.04| 3.07 | 2.52       |

### Table 6

| Products     | Mass/g | Chemical composition | Recovery ratio of P$_2$O$_5$% |
|--------------|--------|----------------------|-----------------------------|
| Concentrate  | 11.04  | 4.92                 | 72.62                       |
| Tailing      | 18.96  | 1.08                 | 27.38                       |
| Parallel     | 30     | 2.52                 | –                           |

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