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Mineralogical Phase of Slag and Its Effect on Dephosphorization during Converter Steelmaking Using Slag-Remaining Technology

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Abstract: To achieve reuse of converter slag, the influence of the $P_2O_5$ content on the component activity of dephosphorization slag, the basicity of the slag, and the phosphorus content in liquid steel were studied and the influence of temperature on the end phosphorus content in liquid steel was analyzed. The results show that the slag remaining in the last heat still exhibited strong dephosphorization ability. Six heats of industrial basic oxygen furnace trials with a 100t-scale top-bottom combined blown converter were carried out using slag-remaining technology. The phases of dephosphorization slag were analyzed by scanning electron microscopy–energy dispersive X-ray spectroscopy and X-ray diffraction. The decomposition reaction of RO phase (a solid-solution composed of some divalent metal oxides, such as FeO, MgO, MnO, CaO, etc.) was relatively complete in slag with a high CaO/SiO$_2$ ratio but was not complete in the slag with a low CaO/SiO$_2$ ratio. Euhedral crystals of 2CaO·SiO$_2$ with certain amount of 3CaO·$P_2O_5$ were formed, resulting in a solid-solution phase of 2CaO·SiO$_2$–3CaO·$P_2O_5$. The liquid phase was present in greater amount and was mainly composed of calcium ferrite. The enrichment of SiO$_2$ and $P_2O_5$ was easier with increasing recycling times of the converter slag. To maintain a higher efficiency of dephosphorization, the $P_2O_5$ content in the slag should be maintained below 3% and the CaO/SiO$_2$ ratio of slag should be maintained above 3.

Keywords: Slag-remaining, Mineralogical phase, enrichment of slag composition, enrichment ratio

Introduction

Expanding the range of channels available for the harmless, high-value, and cyclic utilization of steelmaking slag is important for the development of a circular economy and for achieving sustainable development [1]. Reusing the slag in metallurgy is the most convenient approach to secondary use; it can not only reduce the cost of steelmaking, thereby resulting in direct economic benefits, but also protect the environment, providing a substantial social benefit. Returning steelmaking slag to blast-furnace sinter for use in steelmaking is one of the methods of reducing slag [2, 3].

Slag-remaining technology should be expanded to solve security problems related to the slag generated during converter steelmaking. Slag-remaining technology involves reusing slag in the next heat. High basicity, high temperatures, and certain quantities of T.Fe and MnO in the end slag tend to favor slag formation in the next heat at the early stage of blowing in converter steelmaking. Slag-remaining operation can effectively reduce the cost associated with the consumption of limestone and dolomite slagging material and increase the yield of metal.

Japanese steelmakers have spearheaded attempts to reduce slag volumes in the individual unit process and to achieve “slagless steelmaking” by recycling slags internally [4]. Steelmaking slag volumes have been reduced at the commercial scale from approximately 140 kg/t steel to 60 kg/t steel. Moreover, model predictions have shown that, in principle, all slag can be recycled and the phosphorous can be extracted to produce fertilizer [5]. This development is particularly important because the supply of low-phosphorous iron ores is diminishing at an ever-increasing rate [6].

Slag-remaining technology mainly includes single-slag technology, double-slag technology, and duplex melting technology in converter steelmaking. Determining a suitable quantity of remaining slag and a suitable heating time according to the actual conditions in the converter requires further research to extract the maximum value from converter slag. For small-converter steelmaking (< 150 t), single-slag technology has several advantages.
over double-slag technology and duplex melting technology with respect to production speed and cost.

\( \text{C}_2\text{S} \) forms a solid solution with tricalcium phosphate (C3P) at the treatment temperature over a wide composition range [7, 8], implying that the product of the dephosphorization reaction can be dissolved into the solid phase in slag. Measurements of the equilibrium distribution ratio of \( \text{P}_2\text{O}_5 \) between the solid solution and the liquid phase have revealed that \( \text{P}_2\text{O}_5 \) is concentrated in the solid solution with a high distribution ratio [9, 10].

The task of a converter slag-making system is to convert the monticellite into dicalcium silicate and tricalcium silicate slag with a high-concentration of reaction of RO phase as quickly as possible. The conversion of monticellite to dicalcium silicate and tricalcium silicate is completed by replacement of other oxides of metallic elements. The chemical affinity of monticellite toward \( \text{SiO}_2 \) is lower than that toward \( \text{CaO} \). The phase of RO and \( 2\text{CaO}·\text{SiO}_2 \) can form eutectics. A large amount of dicalcium silicate phase enters the RO phase after instant formation of the RO phase. Dicalcium silicate cores lump together and grow during the final stage of blowing, whereas the RO phase fully gathers during crystallization of the dicalcium silicate. The amount of RO phase increases sharply at the blowing endpoint [11–13].

On the basis of the aforementioned analysis, in the present work, a slag-remaining experiment was carried out via single-slag technology combined with production process conditions in a 100t converter under slag-remaining operating conditions. The single-slag method of the slag-remaining process based on single-slag technology was investigated through theoretical calculations and an analysis of the mineralogical phases. The influences of slag composition on the mineralogical and final element contents of liquid steel were studied through phase analysis of the recycled converter slag.

### Theoretical calculations of slag-remaining process

#### Effect of \( \text{P}_2\text{O}_5 \) content in final slag on the activities of components in slag

When the blowing process in converter steelmaking is near the endpoint, phosphorus in the slag is more easily transferred into the liquid steel because of the high temperatures, where it adversely affects dephosphorization. To study the influence of the \( \text{P}_2\text{O}_5 \) content in slag on the dephosphorization ability of the slag, slag systems were prepared as described in Table 1, where the \( \text{P}_2\text{O}_5 \) content of the slags was varied between 1% and 10% with constant basicity. The activities of the slag components at 1660°C were calculated through the FactSage 7.0 software.

#### Calculation of dephosphorization using recycled converter steelmaking slag

1. **Calculation of dephosphorization using recycled converter steelmaking slag**

   The chemical thermal equilibrium of dephosphorization at the blowing endpoint was calculated with the FactSage7.0 software. For calculation of the activity of each component in the slag, the pure substance was taken as the standard state.

   The influences of \( \text{P}_2\text{O}_5 \) content of slag on the activities of the slag components were shown in Figure 1. It can be seen the \( \text{P}_2\text{O}_5 \) content of the final slag, which was less than 3%, had little effect on the activities of \( \text{CaO}, \text{SiO}_2, \text{FeO}, \) and \( \text{P}_2\text{O}_5 \), whereas the activities of the components of the final slag clearly changed when the \( \text{P}_2\text{O}_5 \) content in the final slag was greater than 3%. The value of \( a_{\text{CaO}} \) decreased sharply, whereas the values of \( a_{\text{SiO}_2}, a_{\text{FeO}}, \) and \( a_{\text{P}_2\text{O}_5} \) increased sharply with increasing \( \text{P}_2\text{O}_5 \) content. A change in the trend of the other components’ activities was very obvious when the \( \text{P}_2\text{O}_5 \) content of the final slag was greater than 3%. Therefore, the \( \text{P}_2\text{O}_5 \) content of the final slag should be controlled to less than 3% to reduce the influence of the activity changes of other components on the composition of the liquid steel.

### Table 1: Chemical compositions of the studied pre-set slag system.

| Number | Mass fraction of components in slag/% | \( \text{CaO}/\text{SiO}_2 \) |
|--------|--------------------------------------|-----------------------------|
| 1      | 46.75 8 5 20 15.25 1 3               |
| 2      | 45 8 5 20 15 2 3                     |
| 3      | 44.25 8 5 20 14.75 3 3               |
| 4      | 43.5 8 5 20 14.5 4 3                 |
| 5      | 42.75 8 5 20 14.25 5 3               |
| 6      | 42 8 5 20 14 6 3                     |
| 7      | 41.25 8 5 20 13.75 7 3               |
| 8      | 40.5 8 5 20 13.5 8 3                 |
| 9      | 39.75 8 5 20 13.25 9 3               |
| 10     | 39 8 5 20 13 10 3                    |

The mass balance of phosphorus was estimated by molecular theory. Thus, the thermodynamic relations of
the dephosphorization reaction between liquid steel and the molten slag are shown as follows [14]:

\[
2[P] + 5(\text{FeO}) + 4(\text{CaO}) = (4\text{CaO} \cdot \text{P}_2\text{O}_5) + 5[\text{Fe}]
\]

(1)

\[
\lg K = \lg \frac{a_{4\text{CaO} \cdot \text{P}_2\text{O}_5}}{[\%P]^2 \cdot a_{\text{FeO}} \cdot a_{\text{CaO}}} = \frac{40067}{T} - 15.06
\]

(2)

\[
\lg [\%P]_{\text{equil}} = \frac{1}{2} \left( \lg a_{4\text{CaO} \cdot \text{P}_2\text{O}_5} - 5\lg a_{\text{FeO}} - 4\lg a_{\text{CaO}} - \frac{40067}{T} + 15.06 \right)
\]

(3)

where [\%P]_{\text{equil}} is the phosphorus content at thermodynamic equilibrium of dephosphorization under the conditions of the blowing endpoint; it can be obtained by substituting the activities of the corresponding components in Table 1 calculated using molecular theory in the equations below. The moles of each component were calculated with reference to 100 g of slag.

Assuming that complex oxides such as 2RO·SiO$_2$ and 4RO·P$_2$O$_5$ can be formed, the calculation is carried out as

\[
n_{\text{RO} \cdot \text{SiO}_2}^0 = n_{\text{SiO}_2}^0 n_{\text{RO} \cdot \text{P}_2\text{O}_5}^0 = n_{\text{P}_2\text{O}_5}^0
\]

\[
n_{\text{RO}} = n_{\text{CaO}}^0 + n_{\text{MnO}}^0 + n_{\text{MgO}}^0 - 2n_{\text{RO} \cdot \text{SiO}_2}^0 - 4n_{\text{RO} \cdot \text{P}_2\text{O}_5}^0
\]

\[
n_{\text{FeO}} = n_{\text{FeO}}^0
\]
\[ \sum n_i = n_{RO} + n_{FeO} + n_{2RO \cdot SiO_2} + n_{4RO \cdot P_2O_5} \]

The activities of the slag components are expressed as

\[ a_{CaO} = X_{RO} = \frac{n_{RO}}{\sum n_i} \tag{4} \]

\[ a_{FeO} = X_{FeO} = \frac{n_{FeO}}{\sum n_i} \tag{5} \]

\[ a_{4CaO \cdot P_2O_5} = X_{4RO \cdot P_2O_5} = \frac{n_{4RO \cdot P_2O_5}}{\sum n_i} \tag{6} \]

(2) Calculation results

The phosphorus content was calculated under the assumption that equilibrium was reached at 1660°C. The dephosphorization capacity of the slag at the terminal state was analyzed. As shown in Figure 2, the equilibrium phosphorus content decreases with increasing basicity under the premise of a constant P\(_2\)O\(_5\) content at 1660°C. The equilibrium phosphorus content increases with increasing P\(_2\)O\(_5\) content at constant basicity. The phosphorus content of liquid steel will increase when P\(_2\)O\(_5\) is enriched in the slag during the continuous remaining slag process. Thus, the CaO/SiO\(_2\) ratio of the slag should be increased to ensure a low phosphorus content of the steel.

As shown in Figure 3, the remaining slag still demonstrates strong dephosphorization ability because of the lower temperature. However, the slag-remaining operation may lead to the enrichment of P\(_2\)O\(_5\) in the slag.

Parameters of slag-remaining tests

The slag-remaining tests were carried out in a 100t-scale top–bottom combined blown converter. The converter was equipped with a dynamic control system but did not have a pre-dephosphorization program and was not equipped with a double converter. Briefly, the experiments were numbered from L0 to L6, where L0 was the heat without slag-remaining and L1–L6 were heats conducted under slag-remaining conditions. The compositions of hot metal, the amounts of auxiliary materials, the temperature, compositions of end-liquid steel and the end-slag compositions are shown in Tables 2–5, respectively. The weight of remaining slag was approximately 3 t for each heat. The detailed slag-remaining operation was 3 t of final slag from heat L0 remained for the next heat L1 when deslagging and so on until heat L6.

The main factors that influence the slag quantity are the silicon content and the phosphorus content in the molten iron. A higher silicon content requires a greater amount of CaO at a constant slag basicity. The amount of slag and auxiliary materials increases with increasing phosphorus content in the molten iron if the composition of the final slag is assumed to be essentially identical.

As shown in Table 2, the conditions of test heats were relatively stable. The silicon content and the phosphorus content changed slightly. The charge weight of hot metal ranged from 98 to 102 t, and the scrap weight was approximately 12 t for each heat.
The amount of CaO in each heat ranged from 3198 to 3755 kg. The average amount was 3413 kg.

Mineralogical structure and composition of remaining slag

These experiments were carried out in a muffle furnace with a temperature accuracy of ± 1°C. A magnesia crucible was used for re-treatment of the slag and was protected by a graphite crucible. The inner diameter, outer diameter, height, and thickness of the magnesia crucible were 40, 45, 110, and 3 mm, respectively. The inner diameter, thickness, and bottom thickness of the graphite crucible were 100, 5, and 10 mm, respectively. The temperature program used in the experiments is shown in Figure 4.

Slag phase in the experiments with no slag-remaining operation

The end phosphorus content of the slag in experiment L0 was 0.024%. The final P₂O₅ content was 3.48%, and the end temperature was 1685°C.

The scanning electron micrograph of slag L0 (Table 6) is shown as Figure 5, and the results of the phase composition analysis by energy dispersive X-ray spectrometry is shown in Table 6. Figure 6 shows the X-ray diffraction (XRD)
Table 6: The EDS of slag of experiment L0.

| Number | O  | Ca  | Fe  | Si  | Mg  | Mn  | P  |
|--------|----|-----|-----|-----|-----|-----|----|
| L0-1   | wt%| 26.60 | 54.80 | 1.92 | 11.74 | 0.30 | 0.10 | 4.53 |
|        | at%| 45.64 | 37.54 | 0.95 | 11.74 | 0.34 | 0.05 | 4.02 |
| L0-2   | wt%| 15.70 | 2.93  | 48.24 | 0.33 | 24.56 | 6.83 | 1.40 |
|        | at%| 31.56 | 2.35  | 27.77 | 0.38 | 32.49 | 4.00 | 1.45 |
| L0-3   | wt%| 21.62 | 59.99 | 11.27 | 0.27 | 0.96 | 4.85 | 1.04 |
|        | at%| 41.95 | 46.47 | 6.27  | 0.30 | 1.23 | 2.74 | 1.04 |

The pattern of the slag from experiment L0. Figures 5 and 6 and Table 6 reveal that L0-1 is a phosphorus-rich phase composed mainly of the solid solution 2CaO-SiO$_2$-3CaO-P$_2$O$_5$. L0-2 is the matrix phase, which is mainly composed of...
CaO·FeO. Meanwhile, a small amount of phosphorus is contained in the liquid phase. L0-3 is an iron-rich phase RO, existing mainly in the form of iron oxide or magnesium iron manganese oxide. Most of the iron exists in this phase. These results are similar to the results of the FactSage 7.0 calculations.

Three typical areas of slag were analyzed by scanning electron microscopy (SEM), as shown in Figure 7. The corresponding EDS result is shown in Table 7. The area of L2-3 is mainly composed of Ca, O, Fe, and Mn. The concentrations of other elements are negligible. The area between the two dotted lines in Figure 7 is considered the phosphorus-enrichment area, which is composed of Ca, O, Si, and P; other elements can be ignored. L2-2 is composed of Fe and Mg; therefore, the area is mainly magnesium iron phase. Thus, the phosphorus in slag is mainly enriched in the L2-1 area, which is composed of a solid solution 2CaO·SiO₂–3CaO·P₂O₅.

Mineralogical composition of slag in experiment with the lowest final phosphorus content

The final phosphorus content of the L2 slag was equal to 0.0156%, which is the lowest among the investigated slags. The final P₂O₅ content was 3.22%, and the end temperature was 1673°C.

Figure 8 shows the XRD pattern of the slag from experiment L2. According to the EDS and XRD results, phase L2-1 is mainly composed of Ca, Si, O, and P, where the Ca/Si/P ratio is approximately 40/12/4.5. The slag phases are mainly 2CaO·SiO₂ and 3CaO·P₂O₅, and these phases may include a small amount of iron and magnesium in terms of the atomic

| Number | O   | Ca  | Fe  | Si  | Mg  | Mn  | P   |
|--------|-----|-----|-----|-----|-----|-----|-----|
| L2-1   |     |     |     |     |     |     |     |
| wt%    | 23.85 | 57.23 | 1.86 | 11.97 | 0.04 | 0.04 | 5.01 |
| at%    | 42.08 | 40.31 | 0.94 | 12.04 | 0.94 | 0.04 | 4.57 |
| L2-2   |     |     |     |     |     |     |     |
| wt%    | 13.12 | 2.80 | 51.95 | 0.27 | 24.55 | 5.97 | 1.34 |
| at%    | 27.42 | 2.33 | 31.09 | 0.32 | 33.76 | 3.63 | 1.44 |
| L2-3   |     |     |     |     |     |     |     |
| wt%    | 19.20 | 61.70 | 12.06 | 0.27 | 1.05 | 4.72 | 1.00 |
| at%    | 38.38 | 49.24 | 6.91 | 0.31 | 2.75 | 1.38 | 1.03 |

Figure 7: SEM spot scanning of the slag in experiment L2.

Figure 8: XRD pattern of the slag of experiment L2.
ratio. The \(3\text{CaO} \cdot \text{P}_2\text{O}_5\) content was not measured because of its low concentration. Therefore, we concluded that the L2-1 phase is mainly a solid solution of \(2\text{CaO} \cdot \text{SiO}_2\)–\(3\text{CaO} \cdot \text{P}_2\text{O}_5\), the L2-2 phase mainly consists of Fe–Mg–Mn with a small amount of Ca and P, and the L2-3 phase is mainly a calcium ferrite phase, where the concentrations of Fe, Mg, and P are negligible.

**Analysis of the phase of the remaining slag**

The mineralogical phases of the final slag are shown in Figure 9. \(L_x\)-1 (\(x = 1–6\)) are phosphorus-rich phases. The area of the phosphorus-rich phase gradually expands beginning with slag L3 (Figure 9), indicating that the phosphorus-enrichment capability of the slag increases. The amount of \(2\text{CaO} \cdot \text{SiO}_2\) precipitation increases with decreasing slag basicity, and the area of the precipitated phase increases with decreasing slag basicity. The basicity of slag decreases because the CaO content of slag changes slightly but the SiO\(_2\) content increases. Larger amounts of SiO\(_2\) combine with CaO to form \(2\text{CaO} \cdot \text{SiO}_2\); therefore, less CaO is available to combined with \(\text{P}_2\text{O}_5\) to form the \(3\text{CaO} \cdot \text{P}_2\text{O}_5\) phase.

Figure 9 shows that the mineral of the final slag L2 is a \(2\text{CaO} \cdot \text{SiO}_2\) euhedral crystal. Meanwhile, the amount of liquid phase, which is mainly calcium ferrite, increases. The RO is not fully decomposed, forming an irregular \(2\text{CaO} \cdot \text{SiO}_2\) phase. The \(\text{P}_2\text{O}_5\) content of the slag and that of the liquid iron at the endpoint of steelmaking gradually increase after experiment L2, mainly because the steel–slag reaction \(2\text{CaO} + \text{SiO}_2 \rightarrow 2\text{CaO} \cdot \text{SiO}_2\) occurs, followed by the decomposition reaction of RO: CaO + RO \(\rightarrow\) CF + MgO. Only separated FeO is saturated by CaO completely, which generates \(2\text{CaO} \cdot \text{FeO}, \text{C}_2(\text{A,F}), \text{or C}_4\text{AF}\), possibly leading to the reaction \(3\text{CaO} + \text{P}_2\text{O}_5 \rightarrow 3\text{CaO} \cdot \text{P}_2\text{O}_5\).

The amount of \(2\text{CaO} \cdot \text{SiO}_2\) in slag increases because a slight change in the CaO content of slag leads to the formation of small amounts of the \(3\text{CaO} \cdot \text{P}_2\text{O}_5\) phase. The amount of phosphorus transferred into the slag decreases, resulting in an increase in the P content in liquid steel. The decrease in amount of calcium phase results from the increase of \(2\text{CaO} \cdot \text{SiO}_2\), reducing the T.Fe content of the slag.

**Application of the results of slag-remaining tests**

As shown in Figure 10, the \(\text{P}_2\text{O}_5\) content in the final slag of experiment L2 rapidly decreased to the lowest level (3.22%), rising to 3.52% in experiment L3 and then rising to the highest level (3.65%) in experiment L6. Because the phosphorus content of hot metal in each experiment is similar and because the total amount of accessories changes little, we concluded that the enrichment of \(\text{P}_2\text{O}_5\) began in experiment L3.

![Figure 9: SEM micrographs of certain mineralogical phases of the slag samples.](image-url)
The trend of the endpoint phosphorus content of the liquid steel is similar to the trend of the $P_2O_5$ content in the slag. The higher the $P_2O_5$ content in the slag, the higher the endpoint phosphorus content of the liquid steel. The change trend is consistent with that predicted by theoretical calculation (Figure 2).

The enrichment of $P_2O_5$ should be considered during continuous remaining slag processing. The amount of CaO should be increased to enhance the CaO/SiO$_2$ ratio if $P_2O_5$ enrichment occurs. Given the calculation results showing the influence of $P_2O_5$ content of slag on the activities of components in slag, slag should be poured out entirely to make new slag again if the enrichment ratio of $P_2O_5$ is greater than 3%.

As evident in Figure 11, the T.Fe content in the slag of the first remaining heat was the highest (18.42%); the T.Fe content then gradually decreased with the number of times the remaining slag was reused. The T.Fe content in slag of the last experiment was equal to 11.93%. The SiO$_2$ content in the slag decreased to 12.47% in experiment L2, then, gradually increased with increasing number of times the remaining slag was reused. The SiO$_2$ content in the last experiment increased to a maximum of 16.45%. The basicity of the final slag decreased with increasing SiO$_2$ content because of the narrow range of Si content and the amount of molten iron; by contrast, the phosphorus content of the final liquid steel substantially increased. The changing trend of the T.Fe content in the final slag was opposite that of the SiO$_2$ content.

From the perspective of mineralogical phases, the increase in the amount of dicalcium silicate and tricalcium silicate phase in the slag results from the increase in the SiO$_2$ content of the slag, which is attributable to the amount of CaO and calcium ferrite phase in slag decreasing slightly and Fe being restored to the liquid steel.

The enrichment of SiO$_2$ should be considered during slag-remaining operation. The amount of CaO should be increased to increase the CaO/SiO$_2$ ratio if the enrichment
ratio of SiO$_2$ is high. Slag should be poured out entirely and new slag should be formed if the enrichment ratio of SiO$_2$ is too high.

Conclusions

(1) The enrichment of SiO$_2$ occurs easily if the silicon content of hot metal exhibits little fluctuation during continuously remaining slag processing. The amount of CaO should be increased to increase the CaO/SiO$_2$ ratio. Slag should be poured out entirely and new slag should be formed if the enrichment ratio of SiO$_2$ is too high and the basicity is greater than 3.

(2) When the P$_2$O$_5$ content of the final slag is greater than 3%, the activities of the components in the slag change substantially. Meanwhile, the endpoint P content of the liquid steel increases dramatically. The lower the CaO/SiO$_2$ ratio, the faster the increase in endpoint P content. The P$_2$O$_5$ content of the final slag should be controlled at less than 3%.

(3) Elliptical euhedral crystals of 2CaO-SiO$_2$–3CaO-P$_2$O$_5$ form in the final slag with a high ratio of CaO/SiO$_2$ during the continuously remaining slag process. Meanwhile, the liquid phase becomes predominant and is composed mainly of calcium ferrite; the amounts and area of the RO phase are small. The CaO/SiO$_2$ ratio is low, the decomposition of the RO phase is incomplete, and the quantity of liquid phase is small, resulting in the formation of an irregular 2CaO-SiO$_2$–3CaO-P$_2$O$_5$ phase.

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