Research Article

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Effect of 2CaO·SiO₂ particles addition on dephosphorization behavior

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Abstract: The effect of the addition of 2CaO·SiO₂ solid particles on dephosphorization behavior in carbon-saturated hot metal was investigated. The research results showed that the addition of 2CaO·SiO₂ particles have little influence on desilicication and demanganization, and the removal of [Si] and [Mn] occurred in the first 5 min with different conditions where the contents of 2CaO·SiO₂ particles addition for the conditions 1, 2, 3, 4, and 5 are 0, 2.2, 6.4, 8.6, and 13.0 g, respectively. The final dephosphorization ratios for the conditions 1, 2, 3, 4, and 5 are 61.2%, 66.9%, 79.6%, 63.0%, and 78.1%, respectively. The dephosphorization ratio decreases with the increase of 2CaO·SiO₂ particles in the first 3 min. The reason for this is that the dephosphorization process between hot metal and slag containing C₂S phase consisted of two stages: Stage 1. [P] transfers from hot metal to liquid slag and Stage 2, the dephosphorization production (3CaO·P₂O₅) in liquid slag reacts with 2CaO·SiO₂ to form C₆S–C₆P solid solution. The increase of 2CaO·SiO₂ particles increases the viscosity of slag and weakens the dephosphorization ability of the stage 1. The SEM and XRD analyses show that the phase of dephosphorization slag with the addition of diophosphorization slag with the addition of 2CaO·SiO₂ particles is composed of white RO phase, complex liquid silicate phase, and black solid phase (C₆S or C₆S–C₃P). Because the contents of C₂S–C₆P and 2CaO·SiO₂ in slag and the dephosphorization ability of the two stages are different, the dephosphorization ability with different conditions is different.

Keywords: 2CaO·SiO₂ solid particles, dephosphorization behavior, solid solution

1 Introduction

To satisfy the demand for low phosphorus steel and ultra-low phosphorus steel, the content of phosphorus should be controlled as low as possible. Many researchers [1–6] were involved to investigate the dephosphorization ability between the slags and molten steel. Winkler and Chipman [1] and Balajiva and Vajragupta [2] conducted the first comprehensive studies on dephosphorization during the 1940s. Wagner [3] proposed the defined phosphate capacity C₉O₅⁻ to describe the dephosphorization potential. Besides the defined phosphate capacity, the phosphorus partition Lᵣ between slags and hot metal was also used to represent the dephosphorization ability of slags. Basu et al. [4,5] studied the phosphorus partition between liquid steel and CaO–SiO₂–FeO–P₂O₅–MgO slags at 1,873 and 1,923 K, and the research results showed that the phosphorus partition was independent of FeO contents more than 19 wt pct. Yang et al. [6] studied prediction models for phosphorus partition between CaO-based slags and iron-based melts during dephosphorization processes and a Lᵣ model based on the ion and molecule coexistence theory, that is, the IMCT-Lᵣ model coupled with the proposed dephosphorization mechanism was developed.

In fact, in most cases, steelmaking slag is in a solid–liquid coexisting state containing dicalcium silicate solid phase. Dicalcium silicate can enrich phosphorus from the liquid slag and form 2CaO·SiO₂–3CaO·P₂O₅ (C₆S–C₆P) solid solution [7], which can improve the dephosphorization ability of slags. The formation mechanism of C₆S–C₆P solid solution was widely studied [8–13]. Inoue and Suito [8] studied the behavior of phosphorus transfer from CaO–SiO₂–FeO slags to 2CaO·SiO₂ particles and found that the maximum phosphorus distribution ratio between 2CaO·SiO₂ particle and slag was obtained at the nose composition of 2CaO·SiO₂ primary phase region in the CaO–SiO₂–FeO phase diagram. Kitamura et al. [9] studied the mass transfer of P₂O₅ between liquid slag and solid solution. Takeshita et al. [10] measured the free energies of 2CaO·SiO₂–3CaO·P₂O₅. Yang et al. [11–13] studied the phase relationships for the CaO–SiO₂–FeO–5mass%P₂O₅ system. However, the effect of
artificially prepared 2CaO·SiO₂ solid particles addition on dephosphorization behavior between slag and hot metal has not been conducted. In this study, the effect of the addition of 2CaO·SiO₂ particles on dephosphorization behavior was studied, and the dephosphorization mechanism by using 2CaO·SiO₂ addition was discussed.

2 Experimental

2.1 Sample preparation

2.1.1 2CaO·SiO₂

The mixture of reagent grade CaO and SiO₂ on molar ratio of 2:1 was pressed, and about 1% 3CaO·P₂O₅ was added to prevent powdering of 2CaO·SiO₂. Then, the mixture was loaded in an MgO crucible and heated to 1,550°C. After 3 h preservation, the sample was cooled and ground, pressed to the cylindrical shape and heated again at 1,550°C for 3 h, then the sample was ground, and analyzed by XRD. The XRD analysis showed that the phase of the mixture was 2CaO·SiO₂ as shown in Figure 1.

2.1.2 CaO–SiO₂–FeO–P₂O₅–MgO

CaO·P₂O₅, MgO, Fe₂O₃, SiO₂, and CaO of the reagent grade were mixed and melted at 1,600°C in an MgO crucible under Ar atmosphere for premelting. CaO was obtained by calcining limestone at 1,100°C for 2 h. After 60 min preservation, the slag sample was quenched by liquid nitrogen, and then, it was converted into powder. The slag chemical compositions are presented in Table 1.

2.2 Procedure

Pig iron of about 550 g loaded in an MgO crucible was melted at 1,673 ± 5 K (1,400 ± 5°C) in a resistance furnace with protection. The chemical compositions of pig iron used in the present experiment are presented in Table 2. After the pig iron was completely melted, hot metal was sampled as initial composition. Then, the premelted slag and Fe₂O₃ were added with 2CaO·SiO₂ particles. Because there was no oxygen blowing, Fe₂O₃ was used for dephosphorization. To investigate the effect of 2CaO·SiO₂ particles on dephosphorization behavior, there were five added conditions for 2CaO·SiO₂ as presented in Table 3. Hot metal and slag were sampled at 3, 5, 7, 10, and 15 min. The compositions of slag and hot metal samples were analyzed by XRF, and the phase of slag samples were analyzed by SEM/EDS and XRD.

3 Results and discussion

3.1 Changes in the contents of hot metal and slag

Figure 2 shows the changes of [Si] and (SiO₂) contents with reactions under different conditions. As shown in Figure 2, although the initial [Si] contents in hot metal with different

| Condition | Pre-melted slag/g | Fe₂O₃/g | 2CaO·SiO₂/g |
|-----------|-------------------|---------|-------------|
| 1         | 30                | 40      | 0           |
| 2         | 30                | 40      | 2.2         |
| 3         | 30                | 40      | 6.4         |
| 4         | 30                | 40      | 8.6         |
| 5         | 30                | 40      | 13.0        |

Table 1: Chemical compositions of slag (wt%)

| CaO/% | SiO₂/% | FeO/% | P₂O₅/% | MgO/% | R (CaO/SiO₂) |
|-------|--------|-------|--------|-------|--------------|
| 57    | 16     | 20    | 3      | 4     | 3.56         |

Table 2: Chemical compositions of pig iron (wt%)

| C       | Si  | Mn  | P    | S    |
|---------|-----|-----|------|------|
| 4.0–4.3 | 0.3–0.72 | 0.15–0.37 | 0.10–0.185 | 0.015–0.027 |
conditions are different, the variation tendency of [Si] and (SiO₂) are basically the same. [Si] content decreases rapidly, and (SiO₂) content increases seriously at the beginning of reactions and then they change slowly. This shows that the addition of 2CaO·SiO₂ particles has little influence on desilication, and the removal of [Si] is mainly completed in the first 5 min with different conditions.

Figure 3 shows the variations of [Mn] content with reactions under different conditions. It could be observed that the changes of [Mn] content with different conditions are also basically the same. [Mn] content in hot metal decreases quickly in the first 5 min for the conditions 2, 3, and 4 and in the first 7 min for the conditions 1 and 5. The reason for the difference of demanganization behavior is that the initial [Mn] content in hot metal for the condition 1 is higher than others and the initial [FeO] content in slag for the condition 5 is lower than others. This shows that the addition of 2CaO·SiO₂ particles also have little influence on demanganization.

Figure 4 shows the changes of [P] content and (P₂O₅) content with reactions under different conditions. As shown in Figure 4, [P] content decreases seriously in the first 7 min for the conditions 1, 2, and 4 and in the first 10 min for the conditions 3 and 5 and then they change little. (P₂O₅) contents for the conditions 1, 2, and 4 increase quickly in the first 7 min and then change little. By contrast, (P₂O₅) content in slag increases gradually with reactions for the conditions 3 and 5. The final [P] contents for the conditions 3 and 5 are lower than those of the conditions 1, 2, and 4.

Figure 5 shows the changes of the demosphorization ratio at different times with different conditions. Figure 5 shows that the demosphorization ratio increases with reactions under different conditions, and rephosphorization is not observed. The final demosphorization ratios (calculated by (mass% P at 0 min – mass% P at 15 min)/mass% P
at 0 min) for the conditions 1, 2, 3, 4, and 5 are 61.2%, 66.9%, 79.6%, 63.0%, and 78.1%, respectively. The final dephosphorization ratios for the conditions 3 and 5 are higher than those of the conditions 1, 2, and 4. However, the dephosphorization ratios for the conditions 3 and 5 in the first 3 min are lower than those of the conditions 1, 2, and 4. Moreover, the dephosphorization ratios decrease with the increase of $2\text{CaO} \cdot \text{SiO}_2$ particles in the first 3 min. The reason for this is that the increase of $2\text{CaO} \cdot \text{SiO}_2$ particles increase the viscosity of slag, which weakens the dephosphorization ability of the stage 1 as shown in Figure 9.

Figure 6 shows the changes of basicity ($R$) and FeO contents with reactions. Figure 6 shows the changes of basicity ($R$ (defined as $\text{CaO}/\text{SiO}_2$)) and FeO contents with reactions under different conditions, and it is clear that $R$ decreases quickly in the first 3 min for different conditions and then changes little. The final $R$ for the conditions 1, 2, 3, 4, and 5 are 1.50, 1.71, 1.90, 1.70, and 1.85, respectively. FeO content decreases gradually with reactions under different conditions, the final FeO contents for the conditions 3 and 5 are lower than those of the conditions 1, 2, and 4. Because there is no oxygen blowing in the present experiment, Fe$_2$O$_3$ is used as an oxygen source, and the oxygen in Fe$_2$O$_3$ is consumed by the oxidation reaction of hot metal components such as [Si], [Mn], [P], and [C].

### 3.2 Phase of dephosphorization slag

To determine the dephosphorization behavior with $\text{CaS}$ addition clearly, the mineralogy of slag with reactions was analyzed by using SEM/EDS and XRD.

Figure 7 shows the mineralogical structure of dephosphorization slag at 3 and 5 min with different conditions. The compositions of different phase in Figure 7 are analyzed by EDS and presented in Table 4. As shown in Figure 7, the slag phase of different conditions is mainly composed of black, gray, and white phases; the proportion of white phase decreases gradually and the proportion of black phase increases gradually with reactions. According to the EDS analysis of positions 1–3 as presented in Table 4, the white phase is proved to be FeO, the gray phase corresponds to the liquid phase, and the black phase is proved to be the solid phase. Phosphorus is mainly distributed in the black phase. It is clear that the black phase can enrich phosphorus from liquid slag and will affect the dephosphorization process.

Figure 8 shows the slag phase at 3, 5, and 15 min of different conditions analyzed by XRD. Figure 8 shows that the main phase of dephosphorization slag for different conditions is composed of FeO, $\text{Ca}_3\text{MgSiO}_4$, $2\text{Ca}_3\text{Fe}_2\text{O}_5$, $\text{C}_2\text{S}−\text{C}_3\text{P}$ ($2\text{CaO} \cdot \text{SiO}_2−3\text{CaO} \cdot \text{P}_2\text{O}_5$), and $2\text{CaO} \cdot \text{SiO}_2$. The results of the EDS analysis is presented in Table 4, and it is evident that the black phase is $2\text{CaO} \cdot \text{SiO}_2$ or $\text{C}_2\text{S}−\text{C}_3\text{P}$. Due to the complexity of slag composition, together with many kinds of phase in slag, it is difficult to know the content of each phase exactly. But the content of the main phase in slag such as FeO, $\text{C}_2\text{S}−\text{C}_3\text{P}$, and $2\text{CaO} \cdot \text{SiO}_2$ can be approximated by the area and the intensity of peak. The intensity of $\text{C}_2\text{S}−\text{C}_3\text{P}$ peaks for the conditions 3 and 5 is stronger than those of the conditions 2 and 4, and the intensity of $2\text{CaO} \cdot \text{SiO}_2$ peaks for the conditions 3 and 5 is weaker than those of the conditions 2 and 4. This shows
Figure 7: Mineralogy of dephosphorization slag with different conditions. (a) Condition 1–3 min, (b) condition 1–5 min, (c) condition 2–5 min, (d) condition 2–5 min, (e) condition 3–3 min, (f) condition 3–5 min, (g) condition 4–3 min, (h) condition 4–5 min, (i) condition 5–3 min, (j) condition 5–5 min.
that the slag of the conditions 3 and 5 contains more CaS–C2P phase and less 2CaO·SiO₂ phase.

### 3.3 Dephosphorization mechanism by using C₂S addition

It is known that phosphorus is removed by oxidation reaction, and the dephosphorization reaction is usually divided into the following two steps. First, phosphorus in hot metal is oxidized by (Fe₅O₄) in slag to form P₂O₅ as shown in equation (1).

\[
2\text{P} + 5(\text{Fe}_5\text{O}_4) = \text{P}_2\text{O}_5 + 5\text{Fe}\quad \log K = \log \frac{a_{\text{P}_2\text{O}_5}}{a_{\text{P}}a_{\text{Fe}_5\text{O}_4}} \tag{1}
\]

Second, the dephosphorization production of P₂O₅ in liquid slag is stabilized by CaO to form tricalcium phosphate (3CaO·P₂O₅).

\[
P₂O₅ + 3(\text{CaO}) = (3\text{CaO}·\text{P}_2\text{O}_5)\quad \log K = \log \frac{a_{3\text{CaO}·\text{P}_2\text{O}_5}}{a_{P₂O₅}a_{3\text{CaO}}} \tag{2}
\]

However, the research results of the study by Fix et al. [7] suggested that (2CaO·SiO₂) and (3CaO·P₂O₅) could form a solid solution during the steelmaking in temperature in a wide composition range, and the steelmaking slag was mostly saturated with the (2CaO·SiO₂) phase according to the phase diagram of CaO–SiO₂–FeO system [16]. So the dephosphorization reaction should contain the third step, which can be expressed as follows:

\[
\text{x}(3\text{CaO}·\text{P}_2\text{O}_5) + \text{y}(2\text{CaO}·\text{SiO}_2) = \text{x}(3\text{CaO}·\text{P}_2\text{O}_5·y(2\text{CaO}·\text{SiO}_2)
\]

\[
\log K = \log \frac{a_{3\text{CaO}·\text{P}_2\text{O}_5·y(2\text{CaO}·\text{SiO}_2)}}{a_{3\text{CaO}·\text{P}_2\text{O}_5·y(2\text{CaO}·\text{SiO}_2)}} \tag{3}
\]

Thus, the schematic of the dephosphorization process between hot metal and slag containing the C₂S phase is shown in Figure 9. Figure 9 shows that the dephosphorization reaction consists of two stages. First, [P] transfers from hot metal to liquid slag by the oxidized reaction. Second, the dephosphorization production (3CaO·P₂O₅) in liquid slag reacts with (2CaO·SiO₂) to form C₃S–C₂P solid solution. Dephosphorization is determined by the two stages. The phosphorous distribution between slag and hot metal, \(L_P = (\% \text{P}_2\text{O}_5)_{\text{in slag}}/(\% \text{P}_2\text{O}_5)_{\text{in hot metal}}\), is used to represent the dephosphorization ability of the stage 1. The phosphorous distribution between C₃S–C₂P solid solution and slag, \(L'_P = ((\% \text{P}_2\text{O}_5)_{\text{in C}_3\text{S}-\text{C}_2\text{P}})/(\% \text{P}_2\text{O}_5)_{\text{in slag}}\), is used to represent the dephosphorization ability of the stage 2.

Figure 10 shows the changes of \(L_P\) and \(L'_P\) with reactions. As shown in Figure 10, \(L'_P\) changes little with reactions under different conditions, and it varies from 2 to 5. The reason for this is that \(L'_P\) or \(P_2O_5\) content in C₃S–C₂P is mainly influenced by the slag compositions [15], and Figure 6 shows that the differences of \(R\) and FeO content between different conditions are small. The changes of \(L'_P\) indicate that the addition of 2CaO·SiO₂ solid particles have little influence on the dephosphorization process of the stage 2. Compared with \(L'_P\), \(L_P\) is significantly bigger than \(L'_P\) for different conditions. Although the variation tendency of different conditions is basically the same, \(L_P\) at 10–15 min for the conditions 3 and 5 is significantly bigger than those for the conditions 1, 2, and 4. This shows that the dephosphorization ability of stage 1 for the conditions 3 and 5 is stronger than that of the conditions 1, 2, and 4. The reason for that is the [Si] content for the conditions 3 and 5 is lower than that for the conditions 1, 2, and 4, and iron oxide consumed by [Si] oxidation.

### Table 4: Results of EDS analysis for Figure 7 (wt%)

|        | Ca  | Si  | Fe  | P   | O   | Mg  |
|--------|-----|-----|-----|-----|-----|-----|
| 1-3 min⁻¹ | 37.8 | 13.6 | 5.2 | 3.7 | 34.1 | —   |
| 1-3 min⁻² | 3.7  | 2.3  | 55.7 | —   | 22.3 | 2.3  |
| 1-3 min⁻³ | —    | —    | 64.1 | —   | 23.7 | 1.5  |
| 1-5 min⁻¹ | 36.1 | 13.6 | 6.8  | 3.9 | 33.5 | —   |
| 1-5 min⁻² | 5.0  | 1.6  | 36.0 | 3.4 | 30.0 | 6.5  |
| 1-5 min⁻³ | 0.9  | —    | 57.9 | —   | 20.2 | 2.8  |
| 2-3 min⁻¹ | 33.4 | 13.5 | 10.8 | 2.4 | 36.5 | —   |
| 2-3 min⁻² | 35.4 | 2.2  | 35.3 | 0.2 | 18.6 | —   |
| 2-3 min⁻³ | 4.2  | 2.5  | 60.6 | —   | 24.9 | 3.2  |
| 2-5 min⁻¹ | 44.2 | 13.1 | 2.8  | 3.1 | 37.0 | —   |
| 2-5 min⁻² | 31.5 | 1.4  | 28.1 | —   | 32.8 | —   |
| 2-5 min⁻³ | 0.9  | —    | 67.0 | —   | 24.6 | 4.3  |
| 3-3 min⁻¹ | 44.8 | 13.1 | 1.4  | 3.6 | 37.3 | —   |
| 3-3 min⁻² | 30.4 | 1.9  | 36.3 | —   | 28.1 | —   |
| 3-3 min⁻³ | 1.6  | —    | 63.6 | —   | 22.8 | 2.9  |
| 3-5 min⁻¹ | 41.9 | 13.5 | —    | 3.9 | 42.1 | —   |
| 3-5 min⁻² | 29.1 | 1.4  | 41.3 | —   | 27.8 | —   |
| 3-5 min⁻³ | 1.0  | —    | 61.6 | —   | 25.3 | 2.0  |
| 4-3 min⁻¹ | 36.5 | 12.2 | 1.6  | 2.1 | 42.1 | —   |
| 4-3 min⁻² | 29.5 | 0.8  | 38.7 | —   | 31.0 | —   |
| 4-3 min⁻³ | 3.2  | —    | 53.9 | —   | 37.3 | 5.6  |
| 4-5 min⁻¹ | 32.5 | 10.6 | 4.2  | 2.9 | 37.4 | —   |
| 4-5 min⁻² | 27.2 | 9.2  | 15.2 | —   | 41.0 | 1.0  |
| 4-5 min⁻³ | 4.3  | 1.8  | 62.3 | —   | 26.7 | 2.0  |
| 5-3 min⁻¹ | 41.9 | 13.8 | 3.2  | 2.2 | 38.9 | —   |
| 5-3 min⁻² | 19.8 | 6.2  | 36.5 | —   | 34.9 | —   |
| 5-3 min⁻³ | 3.1  | —    | 67.0 | —   | 26.2 | 3.3  |
| 5-5 min⁻¹ | 42.5 | 13.4 | 2.1  | 2.6 | 39.5 | —   |
| 5-5 min⁻² | 13.9 | 3.3  | 40.8 | —   | 24.9 | 1.1  |
| 5-5 min⁻³ | 1.3  | —    | 69.6 | —   | 23.3 | 2.7  |
is less and that by dephosphorization is more. So, more [P] was transferred to the slag to react with the C₃S and form C₃S–C₅P, which reduced [P] reversion rates.

The \( L_p \) obtained in this article is also compared with the calculation results proposed by Ogawa et al. [16] as shown in equation (4). The achievement degree of the \( L_p \) observed in the present experiment and calculated by equation (4) is shown in Figure 11. It can be observed that the \( L_p \) at 10 min after reaction for the condition 1 is consistent with calculation. However, the observed \( L_p \) for the conditions with the addition of 2CaO·SiO₂ is different from calculations, and the deviation level increases with the increase of 2CaO·SiO₂ particles.

\[
\log \frac{[\%P]}{[\%P]} = 2.5 \log([\%T-Fe] + 0.0715([\%CaO] \\
+ 0.25([\%MgO]) + \frac{7710.2}{T} \\
- 8.55 + (105.1/T + 0.0723)[\%C])
\]

Figure 8: The slag phase at 3, 5, and 15 min analyzed by XRD. (a) 3 min, (b) 5 min, (c) 15 min.
4 Conclusions

The effect of the addition of $2\text{CaO} \cdot \text{SiO}_2$ particles on dephosphorization behavior was studied. The conclusions obtained in the present study are summarized as follows:

1. The addition of $2\text{CaO} \cdot \text{SiO}_2$ particles have little influence on desilication and demanganization, and the removal of $[\text{Si}]$ and $[\text{Mn}]$ mainly takes place in the first 5 min with different conditions.

2. The final dephosphorization ratios for the conditions 3 and 5 are higher than those of the conditions 1, 2, and 4, but the dephosphorization ratio decreases with the increase of $2\text{CaO} \cdot \text{SiO}_2$ particles in the first 3 min.

3. The XRD intensity of $\text{C}_2\text{S} – \text{C}_3\text{P}$ for the conditions 3 and 5 is stronger than those of the conditions 2 and 4, and the XRD intensity of $2\text{CaO} \cdot \text{SiO}_2$ for the conditions 3 and 5 is weaker than those of the conditions 2 and 4. The slag of the conditions 3 and 5 contains more $\text{C}_2\text{S} – \text{C}_3\text{P}$ phase and less $2\text{CaO} \cdot \text{SiO}_2$ phase.

4. Dephosphorization is determined by the two stages. The addition of $2\text{CaO} \cdot \text{SiO}_2$ solid particles has little influence on the dephosphorization process of the stage 2.

Figure 10: Changes of $L_P$ and $L'_P$ with reactions.

Figure 11: Changes of apparent equilibrium achievement degree with different conditions.
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