Recycling Nickel Slag by Aluminum Dross: Iron-extraction and Secondary Slag Stabilization

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Nickel slag is a metallurgical solid waste from nickel refineries, which can be recycled as one of excellent secondary sources due to the amounts of iron contents. The iron content can reach up to 50 wt%.1,2 With the growing demand of nickel products nowadays, nickel slag is discharged continuously. It is estimated that 6–16 tons of nickel slag in flash smelting process are generated per ton of nickel produced.3,4 Taking Jinchuan Group Co. Ltd (Yinchuan, China) as an example, ~160 tons of granulated nickel slag are output annually and more than 33 million tons of the nickel slag have been accumulated to date. Generally, these waste streams are utilized in sectors like road construction, fertilizer and cement manufacture, while the rest are stockpiled in heaps or controlled in landfills.5 The negligent disposal raises serious environmental concerns and valuable resources waste.6,7 Therefore, effective technology for iron extraction from the nickel slag is deeply essential to the sustainable development of resource and environment.

Hydrometallurgy is regarded as an effective method to extract metals from waste slags including flotation, leaching, roasting followed by leaching and bioleaching.8,9 Nevertheless, its production efficiency is limited and the disposal of waste water produced is difficult.10 Compared with the hydrometallurgical processes, liquid waste would not be generated by pyrometallurgical methods. Moreover, considerable sensible heat contained in the molten nickel slag can be used adequately.11 Therefore, pyrometallurgical methods are used mainly for slag treatment in today’s recovery of base and precious metals, and in the past years, extensive researches have been carried out.12-16 Although most of the works are focused on the copper and other waste slags, the thermal reduction techniques they used can still provide lots of valuable ideas for recycling nickel slag.

On the one hand, reduction method was generally adopted in the recovery process.17-21 Coke and carbon powder were used as the reducing agent, and fluxes such as CaO, CaF2 and CaCO3 were added as the constituent regulators to modify the properties of the residual melt. Apart from the carbothermal reduction process, aluminothermic reduction method in waste copper slag was also investigated by Heo et al.18 Even though the clean secondary slag (by-product after the aluminothermic reduction) could be produced, high purity aluminum shots acting as the reducing agent were not economic. On the other hand, the oxidation method was also studied by Wang et al.22,23 Oxygen with a fixed flow rate was inlet into slag system to oxidize the iron-bearing phase from fayalite to magnetite, followed by the magnetic separation and reduction process.

Learning from the treatment techniques for cooper slag, aluminothermic reduction process is adopted for the iron recovery from nickel slag. However, aluminum dross is chosen as the substitution of pure aluminum shot. Aluminum dross is an inevitable metallurgical solid waste from aluminum industries, e.g. electrolytic or cast aluminum industries.24,25 Typically, the aluminum dross mainly consists of oxides Al2O3, SiO2, MgO, CaO and elementary substance Al, and the content of Al can reach up to 30–35 wt%, signifying an exciting new alternative source of aluminum. About 25–50 kg of aluminum dross is released per ton of aluminum produced, which is also piled up in the open-air resulting in serious environmental problems.26 Compared
with the processes aforementioned, the current innovative approach has the following characteristics:

(1) Short process can be acquired and considerable physical heat in molten nickel slag can be used sufficiently, which is out of reach to oxidation process.\(^{27,28}\)

(2) Carbothermal reduction process is an endothermic reaction, the main drawback of this technology is its high energy consumption.\(^{29}\) It was estimated that about 156 kJ heat would be consumed per kilogram of Fe generated. Alternatively, the aluminothermic reduction is considered as a self-sustaining method due to the generation of essential heat energy.\(^{30}\)

(3) Reduction by aluminum dross rather than by the high purity aluminum shot is economical as well as environmentally friendly.

In our previous works, we have discussed the viscosity evolution of slags during the reduction process, and a better kinetic condition could be obtained.\(^{31,32}\) The purpose of this paper is to investigate the feasibility of the innovative approach. As mentioned above, modifier (or flux) is of significance to the treatment process. It can not only separate the 'FeO' and increase its activity in nickel slag, but also facilitate the secondary stabilization. Therefore, the network modification process is firstly studied upon the thermodynamics analysis, and mechanism underlying the role of the modifier is investigated. Additionally, the reduction behavior is studied to take a knowledge of aluminothermic reduction by aluminum dross. What’s more, the reduced products and secondary slag stabilization are analyzed, respectively.

2. Experimental

2.1. Materials

Nickel slag used in this experiment was the quenched flash smelting slag, which was supplied by a domestic nickel plant. The aluminum dross was collected from an aluminum enterprise. The compositions of these two solid wastes were listed in Table 1. The iron content containing in nickel slag reached up to 51.31 wt%, simultaneously, the content of metallic Al in aluminum dross was determined as 33.14 wt% by titration method. Due to the difference in activity, redox reactions can occur between different metal ions. As indicated in Eqs. (1)–(2), FeCl\(_3\) was adopted to react with aluminum dross, then KMnO\(_4\) was used to calibrate the amount of Fe\(^{3+}\) generated from Eq. (1). According to the consumption of KMnO\(_4\), the content of metallic Al was calculated. The repeated measurements were carried out at least three times, and the mean value was finally determined.

\[
\text{Al} + 3\text{Fe}^{3+} = \text{Al}^{3+} + 3\text{Fe}^{2+} \quad \text{(1)}
\]

\[
\text{MnO}^{4-} + 8\text{H}^+ + 5\text{Fe}^{2+} = \text{Mn}^{2+} + 5\text{Fe}^{3+} + 4\text{H}_2\text{O} \quad \text{(2)}
\]

Table 1. Chemical compositions of nickel slag and aluminum dross (wt%).

<table>
<thead>
<tr>
<th>Compositions</th>
<th>'FeO'</th>
<th>CaO</th>
<th>MgO</th>
<th>SiO(_2)</th>
<th>Al(_2)O(_3)</th>
<th>Al</th>
<th>AlN</th>
<th>Na(_2)O</th>
<th>Ni</th>
<th>Co</th>
<th>Cu</th>
<th>S</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nickel slag</td>
<td>51.31</td>
<td>3.58</td>
<td>8.41</td>
<td>31.60</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>0.16</td>
<td>0.08</td>
<td>0.22</td>
<td>1.20</td>
<td></td>
</tr>
<tr>
<td>Aluminum dross</td>
<td>–</td>
<td>2.04</td>
<td>6.97</td>
<td>10.12</td>
<td>40.83</td>
<td>33.14</td>
<td>4.85</td>
<td>2.05</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>–</td>
</tr>
</tbody>
</table>

The detection of Na\(_2\)O was carried out by X-Ray Fluorescence (XRF, ZSX100e, Rigaku, Japan). A ZSX100e spectrometer was applied to determine the chemical compositions of the slag system, and the measurement range of which was from 5 ppm to 100%. Each element has a specific wavelength of the fluorescent ray, and the intensity of the fluorescent ray is proportional to its concentration. After the intensity detection of elemental Na and the corrections of matrix effect, spectral overlap and peak-position background, the content of Na was obtained. Thereafter, the content of Na\(_2\)O was calculated. As for the AlN content, pulsed infrared and thermal-conductive method was applied in analyzing the content of elemental N in dross. Oxygen, nitrogen and hydrogen combined analyzer (ONH 836, LECO, USA) was adopted, the N content range of which covers from 0.05% to 3.0%. The analysis power was 5.0 kW, and the minimum integration time was 45 s. The content of AlN in dross was calculated based on the content of elemental N. Additionally, the process conditions without soluble-salt addition induced the lower contents of F, K and Cl (~0.14%) in aluminum dross, which had been ignored in this work.

Figure 1 showed the characterization of nickel slag and aluminum dross. In order to take an understanding of the crystal phases in materials, X-ray diffraction (XRD, D8 Advance, Bruker AXS GmbH, Germany) pattern was conducted and the results were shown in Figs. 1(a) and 1(c). XRD pattern was operated at 40 kV and 40 mA with Cu K\(\alpha\) radiation, and the pattern data were collected in the 2-theta range between 10° and 90°. It could be observed that minerals in nickel slag were fayalite (2FeO\(\cdot\)SiO\(_2\)) and hortonolite (2(MgO, FeO)\(\cdot\)SiO\(_2\)), and 2FeO\(\cdot\)SiO\(_2\) were the dominant phase. Figure 1(b) presented the scanning electron microscope (SEM, Zeiss ULTRA 55) image of nickel slag. Element distributions of points A, B and C in the image were analyzed by energy dispersive spectrometer (EDS), and the results were listed in Table 2. From the atomic ratio of elemental Fe, Mg and O, points A and B represented the hortonolite phases with clumpy and layered structure, respectively. The difference in the structure lied in the different contents of elemental Fe and Mg. While, the result of point C indicated that the matrix of nickel slag was fayalite phase. As shown in Figs. 1(c) and 1(d), aluminum dross was characterized by the metallic Al and other oxides, keeping a good accordance with the chemical analysis. The particle size distribution of dross was analyzed by Master sizer 2000 (Malvern, England), and the mean size was 839.365 \(\mu\)m.

2.2. Thermodynamic Analysis

In conjunction with the results of XRD and SEM, ‘FeO’ in nickel slag was always presenting as the form of silicate phases. Since SiO\(_2\) would act as the network former in slag structure, either the fayalite or hortonolite had a highly polymerized structure.\(^{33-35}\) Therefore, modifier was essential to adjust nickel slag to separate ‘FeO’ and to obtain a higher activity.\(^{36,37}\) As studied by Li et al., network modifi-

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cation of slag had been widely applied into improving slag properties during processes of waste treatment, such as disintegration, cementitious reactivity, heavy metal leaching and iron component recovery.\textsuperscript{38,39} Appropriate modification method would benefit the extraction of valuable metals from molten slag and the transition of secondary slag into value-added materials.

Thermodynamic analysis for slag made it possible to predict the proper modifier. The standard Gibbs free energy ($\Delta G^\theta$) of related reactions was presented as Eqs. (3)–(8), which was calculated by the thermodynamic software FactSage 7.0 (CRCT ThermFact, Inc., Montreal, Canada).\textsuperscript{40} During the calculation process, phase (solid or liquid) of each compound was determined as the stable one under the current temperature. Figure 2 showed the relationship between $\Delta G^\theta$ and temperature. With temperature increasing from 273 to 1 873 K, the positivity for Eq. (3) signified the reaction would not take place under the standard state. On the contrary, the values of $\Delta G^\theta$ for Eqs. (6)–(8) were all negative over the temperature range, indicating the thermodynamic possibilities of these four reactions. The lower values for Eqs. (7) and (8) suggested the stronger binding ability between CaO and SiO$_2$, and CaO should be the optimum modifier in comparison with MgO and Al$_2$O$_3$.

Al$_2$O$_3$($s$) + 2FeO・SiO$_2$($s/1$) = Al$_2$O$_3$・SiO$_2$($s$) + 2FeO($s/1$)  ...................................................... (3)
2.3. Target Basicity of Modified Slag

Compositions of the modified slags (mixture of nickel slag and CaO) with different slag basicities (mass ratio of CaO/SiO$_2$ in slag system) were plotted in Fig. 3.\(^4\) As shown in Figs. 3(a) and 3(b), CaO–FeO–SiO$_2$–MgO and CaO–Al$_2$O$_3$–SiO$_2$–MgO were the corresponding slag systems before and after the reduction process, respectively. Point A represented the composition of nickel slag, while points B, C, D, E and F represented the compositions of the modified slag with basicities of 0.5, 0.75, 1.0, 1.25 and 1.5, respectively. After the reduction reaction, these points evolved to be points A’ to F’ correspondingly. According to the isothermals, the influences of CaO addition on the evolution of the melting points of slags were different.

Effects of slag basicity on the melting points of slags and the activity of ‘FeO’ were shown in Fig. 4, where Fig. 4(a) showed the melting points of initial and secondary slags under different slag basicities.\(^4\) Here, the initial slag was defined as the slag before aluminothermic reduction. For the initial slags, the melting points changed non-monotonically with addition of CaO and exhibited a minimum value of 1 386 K at basicity of 0.5. As studied by Lee et al., CaO addition changed the equilibrated primary phase of the slag, and the increment of melting points of slags was attributed to the enhanced crystallization effect by stoichiometric compound.\(^35,41\) In practice, the discharging temperature of nickel slag varied from 1 573 to 1 633 K. If basicity of the modified slag was higher than 1.25, the melting point of initial slag was higher than the discharging temperature. Therefore, the basicity of modified slag should be controlled lower than 1.25 to guarantee a liquid state. Alternatively, when the basicity of modified slag was lower than 0.75, the melting point of secondary slag was higher than 1 820 K.

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\[ \frac{3}{2} \text{Al}_2\text{O}_3(s) + 2\text{FeO} \cdot \text{SiO}_2(s/l) = \frac{3}{2} \text{Al}_2\text{O}_3 \cdot \text{SiO}_2(s) + 2\text{FeO}(s/l) \]  
\[ \text{MgO}(s) + 2\text{FeO} \cdot \text{SiO}_2(s/l) = \text{MgO} \cdot \text{SiO}_2(s/l) + 2\text{FeO}(s/l) \]  
\[ 2\text{MgO}(s) + 2\text{FeO} \cdot \text{SiO}_2(s/l) = 2\text{MgO} \cdot \text{SiO}_2(s) + 2\text{FeO}(s/l) \]  
\[ \text{CaO}(s) + 2\text{FeO} \cdot \text{SiO}_2(s/l) = \text{CaO} \cdot \text{SiO}_2(s/l) + 2\text{FeO}(s/l) \]  
\[ 2\text{CaO}(s) + 2\text{FeO} \cdot \text{SiO}_2(s/l) = 2\text{CaO} \cdot \text{SiO}_2(s) + 2\text{FeO}(s/l) \]
K, suggesting a higher reduction temperature. In terms of energy conservation, the basicity of modified slag lower than 0.75 was not advisable.

Figure 4(b) represented the relationship between activity of ‘FeO’ (α(‘FeO’)) and slag basicity. α(‘FeO’) increased first then decreased, and the maximum value of 0.729 was obtained at the basicity of 1.0. Combining with the evolution of melting points of slags, the proper basicity of modified slag should be 1.0.

2.4. Experimental Procedure and Equipment

Modification experiment was carried out firstly. Nickel slag and the CaO powder were mixed, where CaO powder was prepared by calcining the reagent grade CaCO3 powder (purity ≥ 99.9%) at 1 173 K. The appropriate amount of CaO was added to guarantee basicity of the modified slag of 1.0. The experiment was performed using an electric tubular furnace, as shown in Fig. 5.

It was equipped with six U-shape MoSi2 heaters. After the intensive mixing, 20 grams of samples were charged into a magnesia crucible (outer diameter: 30 mm, inner diameter: 24 mm, height: 45 mm), which was then placed into the furnace and kept in the high temperature zone. The slag temperature was precisely controlled by a PtRh30-PtRh6 thermocouple, the deviation of which was within ±2 K. The experimental temperature was determined as 1 773 K in considerations of the melting point of the secondary slag and the exothermic characteristic of aluminothermic reaction. After heated to the required temperature, the mixture was held at 1 773 K for 30 min to ensure the formation of homogeneous molten slag. In the whole procedure, high purity argon with the flow rate of 0.5 L∙min⁻¹ was guaranteed to avoid the oxidation of slag components. Then, the crucible with molten slag was taken out rapidly and quenched in liquid nitrogen, waiting for the examination by SEM equipped with EDS.

For the aluminothermic reduction experiment, the technological procedure was similar. After the modified slag was homogeneous at 1 773 K, a certain proportion of aluminum dross was added into the molten slag by a quartz tube. In the present experiment, Al/‘FeO’ ratio of 0.67 was determined upon the reduction reaction Eq. (9).42)

\[
\text{Al(l) + 1.5FeO(l) = 1.5Fe(l) + 0.5Al}_2\text{O}_3(s), \quad \Delta G^0 = -444 428.5 + 89.55T \text{ J mol}^{-1}
\]

After different reaction time (5, 10, 30, 60 and 120 min, respectively), the crucible was taken out quickly and quenched in liquid nitrogen. Based on our pre-experiment (MgO=6.84–9.48 wt% in preliminary slag composition) and other related researches,14,41) the magnesia crucible could keep a perfect condition after reaction time of 60 min, and the reaction time of 120 min was designed to investigate the variation of recovery degree of reduced product. The sample microstructure was investigated by SEM and the compositions of the reduced product were analyzed by XRF.

3. Results and Discussion

3.1. Element Mapping Analysis of the Modified Slag

Figure 6 presents the element mapping of the modified slag. It can be found that iron element has been separated from the silicate structure totally and exists as the free ‘FeO’ in slag. Simultaneously, CaO is mostly associated with the SiO2, implying the sufficient reaction between CaO and fayalite.

Table 3 lists the EDS results of phases in the modified slag. The atomic ratios of points D and E illustrate that the oxides ‘FeO’ and MgO are surrounded by the calcium silicate (CaO·SiO2) and dicalcium silicate (2CaO·SiO2). Based on Eqs. (5)–(8) and the distribution of elemental Mg, Fe and Si, it could concluded that the stabilities of fayalite and hornblende are weaker than those of CaO·SiO2 and 2CaO·SiO2. Therefore, modification by CaO is of significance to adjust the nickel slag to obtain a higher α(‘FeO’).

![Fig. 5. Schematic diagram of experimental setup. (Online version in color.)](image1)

![Fig. 6. Element mapping of the modified slag. (Online version in color.)](image2)
3.2. Aluminothermic Reduction Results

3.2.1. Iron Recovery

Figure 7 shows the images of the reduced product and secondary slag after reaction for 120 min. Iron ingot with the dimension of 10 mm has been coalesced at the bottom of crucible. Moreover, many visible iron droplets have been generated and dispersed in the porous slag or adhered to the inner surface of the crucible.

3.2.2. Slag Microstructure after Reduction Reaction

Figure 8 represents the slag microstructure after reduction reaction for 120 min. The produced iron particles with various shapes randomly distribute in the slag, some of which tend to aggregate together and grow up. The average size of the particles is about 10 μm. The distribution of elemental Fe and Cu in element mapping implies that the copper has been reduced into metallic Cu and has dissolved into metallic Fe to form binary Fe–Cu alloy. This could be understood by the theory of solid solution, as proposed by Hume-Rothery et al.43,44 In addition, the separation of elemental Si from Fe indicates that the component SiO₂ is not reduced during the reduction process, which is determined by the thermodynamics at the experimental temperature. At the same time, the Fe–Cu alloy is surrounded by elemental Ca, Si, Mg and Al, which suggests the generation of more complicated phases.

3.3. Assessment on Iron-extracting from Nickel Slag by Aluminum Dross

3.3.1. Analysis on the Reduced Product

Recovery degree of iron and copper is characterized by Eqs. (10)–(11).

\[
R_{Fe} = \frac{M_{RP} \times w(Fe)}{M_{NS} \times w(TFe)_{NS}} \times 100\% \quad \text{............ (10)}
\]

\[
R_{Cu} = \frac{M_{RP} \times w(Cu)}{M_{NS} \times w(Cu)_{NS}} \times 100\% \quad \text{............ (11)}
\]

where \(R_{Fe}\) and \(R_{Cu}\) are the recovery degree of Fe and Cu, respectively (%), \(M_{RP}\) is the mass of reduction product (kg), \(w(Fe)\) and \(w(Cu)\) are the weight fractions of Fe and Cu in reduced product, respectively (%), \(M_{NS}\) is the mass of nickel slag (kg) and \(w(TFe)_{NS}\) and \(w(Cu)_{NS}\) are the weight fractions of total Fe and Cu in nickel slag, respectively (%).

Although the droplets dispersed in slag or adhered to the inner surface of crucible are difficult to be collected, the visible droplets together with iron ingot are included in the chemical analysis. After reaction for the determined time, the quenched slag samples are grinded into powders, and magnetic separation is adopted to separate the Fe–Cu alloy from the slag waiting for the further analysis. Figure 9 shows the dependence of recovery degree of iron and copper on reaction time. It increases evidently in the former 10 min, followed by a slow evolution then. Also, compared with Fe, Cu recovery degree is much higher at the same reaction time. For instance, it is 70.56% and 60.33% for Cu and Fe at 5 min, respectively, which can be originated from the different reaction potentials of oxides Cu₂O and ‘FeO’ with metallic Al. \(\Delta G^\theta\) for the related two aluminothermic reactions is \(-486.8\) kJ·mol\(^{-1}\) and \(-324.5\) kJ·mol\(^{-1}\), respectively. As a consequence, iron and copper in nickel slag could be well reduced by aluminum dross after reaction time.
Table 4. Chemical compositions of the reduced product (wt%).

<table>
<thead>
<tr>
<th>Reaction time (min)</th>
<th>Elements</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Fe</td>
</tr>
<tr>
<td>5</td>
<td>99.47</td>
</tr>
<tr>
<td>10</td>
<td>99.49</td>
</tr>
<tr>
<td>30</td>
<td>99.49</td>
</tr>
<tr>
<td>60</td>
<td>99.49</td>
</tr>
<tr>
<td>120</td>
<td>99.50</td>
</tr>
</tbody>
</table>

The recovery degree of these two elements is 94.35% and 97.89%, respectively.

Chemical compositions of the reduced product at different reaction time are analyzed by XRF. The results shown in Table 4 indicate that the reduced product from the current process is the crude Fe–Cu–(Ni) alloy. It’s of particular concern that, the change in chemical compositions of the alloy is negligible with reaction time increasing from 5 to 120 min. The content of sulfur in the alloy is lower than 0.03 wt%, which demonstrates the reduced product of Fe–Cu–(Ni) alloy has a higher purity. As the studies by Liang et al., the main constituents of weathering resistant steel are Fe, Cu and Ni, therefore, the produced alloy may be utilized as a raw material in making weathering resistant steel.45–47

3.3.2. Secondary Slag Stabilization

Given the presence of other components as drawbacks, hazardous element in secondary slag is mainly Cu. In the course of reduction process, about 97.89 wt% Cu has transferred to the crude Fe–Cu–(Ni) alloy, and the content of the copper left in secondary slag is only about 0.03 wt%. Therefore, the clean slag could be used as the raw materials in cement industries or glass-ceramic productions.48,49 Furthermore, the changes in slag compositions result in the generation of more complicated phases, which signifies a higher slag stabilization. Figures 10(a) and 10(b) show the evolution of slag compositions and mineral phases with reduction process, respectively. In the quasi-quaternary CaO–SiO₂–FeO–Al₂O₃ (−9 wt% MgO) slag system, the dominant phases of slag have transited from merwinite (Ca₃MgSi₂O₈), gehlenite (Ca₂Al₂SiO₇) to CaMg₂Al₁₆O₂₇ phase, which is the primary phase of secondary slag. What’s more, the compositions of secondary slag in the center of crucible have been detected and shown in the figure. As shown in Fig. 10(b), the main phases of secondary slag are confirmed as CaMg₂Al₁₆O₂₇, anorthite (CaAl₂Si₂O₈), gehlenite (Ca₂Al₂SiO₇), spine (MgAl₂O₄) and akermanite (Ca₃MgSi₂O₇). Additionally, different from the modified slag, FeO peaks in secondary slag have disappeared, which signifies the complete reduction reaction. Compared with the iron-bearing phases, the stronger attraction of cations (Ca²⁺ and Mg²⁺) and orthosilicate (and aluminate) anions in these produced phases leads to the more stable structure and the higher stabilization of secondary slag.34,35

4. Conclusions

The approach of recycling nickel slag by aluminum dross is studied, and the processes of network modification and reduction are investigated successively. The following conclusions can be drawn based on the experimental results.

(1) Thermodynamics analysis suggests that CaO should be the optimum modifier over the oxides MgO and Al₂O₃ to adjust nickel slag. Basicity of the modified slag is determined as 1.0 upon the a(‘FeO”) and the melting points of initial and secondary slags. Element mapping analysis of
modified slag shows that ‘FeO’ has been separated from the structure of fayalite and hortonolite, which is helpful to the further reduction of ‘FeO’.

(2) Composition analysis on metal phase shows that copper has been reduced into metallic Cu and has dissolved into metallic iron to form Fe–Cu alloy. After reaction time of 120 min, the recovery degree of these two elements is 94.35% and 97.89%, respectively.

(3) With reduction process, the dominant phases of secondary slag have transited from merwinite (CaMgSi2O6), gehlenite (Ca2Al2SiO7) to CaMg2Al2O7 phase. Compared with the iron-bearing phases, the main phases of secondary slag have the more stable structure, leading to a higher stabilization of secondary slag.

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