

# DIRECT REDUCTION AND EXTRACTION OF IRON FROM NICKEL SMELTING SLAG COUPLING OF PREPARATION OF CEMENTING MATERIALS USING GANGUE COMPOSITION

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Aiming at the properties of Fe and SiO<sub>2</sub> in nickel slag, the process of preparing DRI by direct reduction nickel slag from coal base was proposed, and the component of gangue is used as raw material to prepare C<sub>2</sub>S(belite) and C<sub>3</sub>S (alite), which is a comprehensive utilization of nickel slag. The reduction reaction of iron coupling of the reaction of cementitious materials was realized through thermodynamic calculation and experiment. The reduction roasting products of nickel slag with iron, C<sub>3</sub>S and C<sub>2</sub>S as the main phase were obtained by reasonable batching and temperature control technology of reduction roasting reaction.

**Keywords:** direct reduction iron, extraction, nickel slag, calcium silicate, x-ray analysis

## INTRODUCTION

Nickel slag is the industrial waste residue. Because of its complex mineral composition, nickel slag has not been effective comprehensive utilized. The available utilization mostly is stack, which wastes ground, and causes hidden dangers for environmental pollution [1]. Nickel slag as the production of metal nickel waste residue, the comprehensive utilization of it can not only reduce environmental pollution, but also has good economic and social benefits [2].

The comprehensive utilization of nickel slag at domestic and abroad mainly includes recovery and extraction of nickel, cobalt, copper, iron, precious metals and other valuable metals [3], the filling material for underground mines [4], the alloy production [5], the production of glass and ceramics [6], production of brick and block cement and cement mixed materials, such as building material [7], the production of slag cotton and road construction [8]. The use of nickel slag has the following problems: (1) the content of valuable metal elements in slag, especially iron is low, (2) The mineral structure is not suitable for blast furnace smelting, (3) The reduction of ore is inefficient, (4) The direct separation effect is not satisfied.

The reduction iron and gangue comprehensive utilization were proposed in this research. “One burning two mill” technology was used to obtain direct reduction of iron which could supply for furnace. The residual could be used to make raw materials. The comprehensive

utilization of nickel slag under low energy consumption was realized by considering the direct reduction of iron oxides and the gelation of Gangue.

## EXPERIMENTAL

### Materials

The nickel slag used in the test was from Jinchuan company. Because of the high content of glass phase, its appearance shows glass luster. Nickel slag’s moisture content is 3 %, and shows a black flake or spherical shape. Its morse hardness is between 6,0 – 6,5, bulk weight for 2 100 – 2 600 kg/m<sup>3</sup>. The X-ray diffraction (XRD) of nickel slag was showed in Figure 1. The Empyrean type of the Dutch Barnard company was used to measure X-ray pattern, and its scanning condition was 8°/ min. The VEGAIXMU type of Czech Tescan was used to test Scanning electron microscope (SEM). The chemical composition of hematite

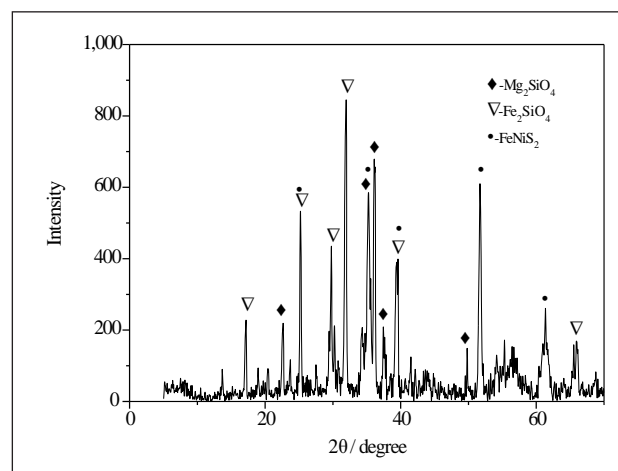


Figure 1 XRD of nickel slag.

R. Shi, Y. Cui, J. Zhao, X. Li, C. Zou. School of Metallurgical Engineering, Xi’an University of Architecture and Technology, China. R. Shi (Email: Shiruimeng@163.com)  
Y. Yu. School of Materials Science and Engineering, Shanghai University, China

Table 1 Experimental composition of nickel slag / %

|         |                                |                  |      |      |                                |
|---------|--------------------------------|------------------|------|------|--------------------------------|
| Name    | TFe                            | SiO <sub>2</sub> | MgO  | CaO  | Al <sub>2</sub> O <sub>3</sub> |
| Content | 49,28                          | 32,89            | 7,92 | 3,37 | 2,27                           |
| Name    | Cr <sub>2</sub> O <sub>3</sub> | CuO              | NiO  | TiO  |                                |
| Content | 0,17                           | 0,16             | 0,12 | 0,12 |                                |

powder was analyzed by fluorescence spectra, and the results were shown in Table 1.

From Table 1, it was showed that the slag contains a high Fe content, and also contains a part of SiO<sub>2</sub>, MgO, Al<sub>2</sub>O<sub>3</sub> and other oxides, and most of these present in the form of glass-state silicate.

## Experimental Analysis

(1) Nickel smelting slag, calcium solvents, additives and reducing agent were placed in proportion to mixture. After adding to the reduction furnace, the samples were preheated, heated heat preservation, deep reduced, cement clinker sintered and cooled. Finally, the samples were quenched.

(2) The samples were treated with magnetic separation. Consequently, the iron-containing was used as the raw material of blast furnace or steelmaking, and other parts were added as cement.

## RESULTS AND DISCUSSION

### Thermodynamic study

Main reactions during reduction roasting of nickel slag was shown in Table 2. Reaction module in the FactSage thermodynamic software was used to calculate the standard Gibbs free energy of the possible reactions of nickel slag in the roasting reduction process. The reactions between is 0 °C - 1 450 °C, interval 10 °C, respectively were shown in Figure 2(a). The relationship between temperature and the standard Gibbs free energy of each reaction was plotted with origin and shown in Figure 2(b).

It can be seen in Figure 2(a), that the reaction equation (2) cannot react spontaneously in the temperature between the 500 °C - 1 450 °C, but the reaction (3) and (4) could react spontaneously and produce calcium silicate and calcium silicate. According to Figure 2(b), when temperature was in 1 300 °C - 1 800 °C, reaction (5) would be spontaneous, calcium silicate combined with calcium oxide, and then calcium silicate was formed. Combined with Figure 2(a) and (b), to create a calcium silicate-oriented gel material, it needed to heat up to 1 300 °C - 1 800 °C. In order to further study the reaction system of Fe<sub>2</sub>O<sub>3</sub> and CaO and SiO<sub>2</sub>, the phase diagram of Fe<sub>2</sub>SiO<sub>4</sub>-CaO-SiO<sub>2</sub> ternary system in 1 350 °C was calculated by using the phase module of FactSage software (shown in Figure 3).

It could be seen in Figure 3 that at 1 350 °C, the ternary system Fe<sub>2</sub>O<sub>3</sub>-CaO-SiO<sub>2</sub> could generate Ca<sub>2</sub>Fe<sub>2</sub>O<sub>5</sub>,

Table 2 Main reactions during reduction roasting of nickel slag

| Number | reaction   |
|--------|--|
| (1)    | Fe <sub>2</sub> SiO <sub>4</sub> +2C=2Fe+SiO <sub>2</sub> +2CO               |
| (2)    | Fe <sub>2</sub> SiO <sub>4</sub> +2CO=2Fe+SiO <sub>2</sub> +2CO <sub>2</sub> |
| (3)    | 2 CaO+ SiO <sub>2</sub> = Ca <sub>2</sub> SiO <sub>4</sub>                   |
| (4)    | 3 CaO+ SiO <sub>2</sub> = Ca <sub>3</sub> SiO <sub>5</sub>                   |
| (5)    | CaO+ Ca <sub>2</sub> SiO <sub>4</sub> = Ca <sub>3</sub> SiO <sub>5</sub>     |

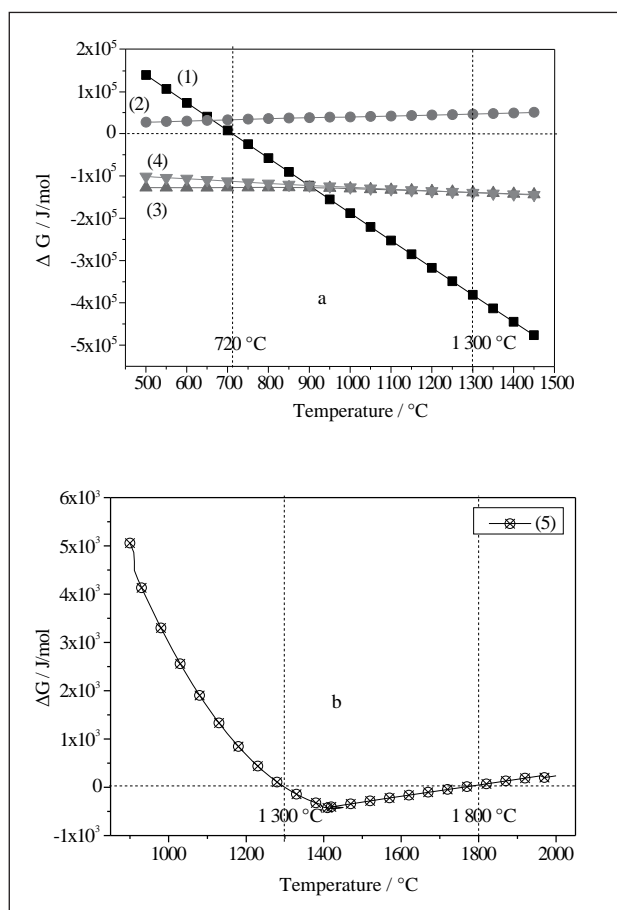


Figure 2 ΔG-T of nickel slag reduction reaction (see Table 2).

Table 3 The compositions of experimental reactants

| Nickel slag | CaO   | C    |
|-------------|-------|------|
| 51,59       | 43,44 | 4,97 |

which becomes the impurities in the gel material and also conduces to iron reduction, When Fe<sub>2</sub>O<sub>3</sub> content was less or zero (such as the blue part of the Figure 3), C<sub>3</sub>S could be generated a lot. Therefore, the Fe<sub>2</sub>O<sub>3</sub> should be reduced to Fe to prevent Ca<sub>2</sub>Fe<sub>2</sub>O<sub>5</sub> generated.

The equipment module of FactSage software was used to calculate the experimental reactions of preparation of cementitious materials such as calcium silicate and calcium silicate. Nickel slag was used as raw material, carbon powder as reducing agent, calcium oxide as additive in the experiment. The compositions of reactants were showed in Table 3. The phases changes in 500 - 1 500 °C, with an interval of 50 °C, was showed in Figure 4.

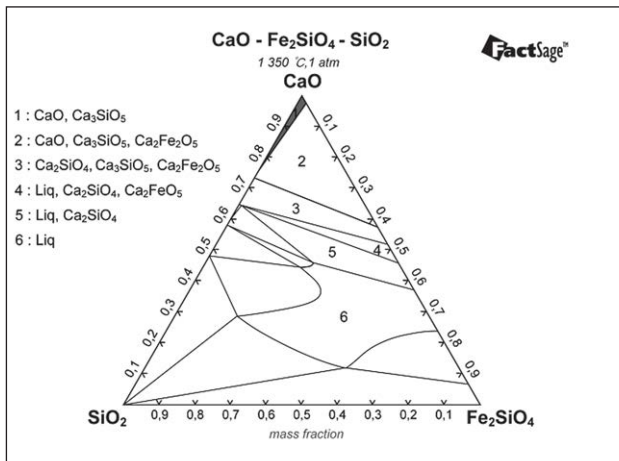


Figure 3 The phase diagram of Fe<sub>2</sub>SiO<sub>4</sub>-CaO-SiO<sub>2</sub> ternary system.

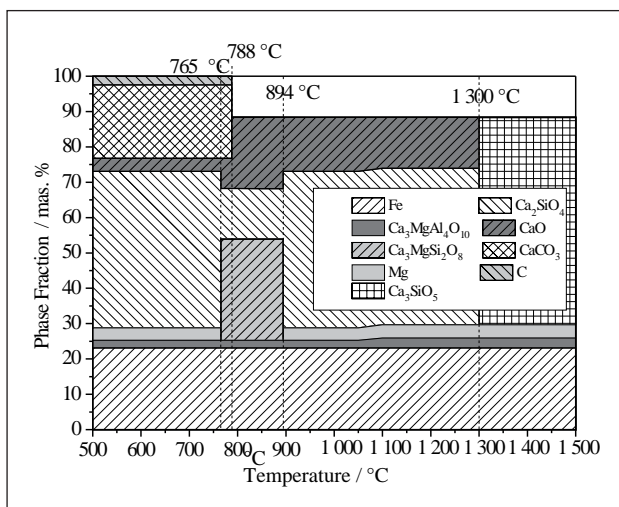


Figure 4 Phases changes between 500 – 1500 °C.

### Synergistic experiment of preparation of cementitious materials and direct reduction of iron

The experiment was carried out with the alkalinity of 2,6, the roasting temperature of 1480 °C and lasted for 40 min, the reducing agent content of 15 %, and the particle of 200 meshes. When the furnace cooled to room temperature, the sample were taken out. After detection of iron reduction rate of 62,9 %. With strong magnets to check the magnetic of sample and XRD examination of the object phase, as shown in Figure 5. SEM analysis of the roasting product was shown in Figure 6.

From Figure 5, it can be seen that with continuous heating, there was no calcium silicate production in reaction, only iron and C<sub>2</sub>S were produced. It could be caused by the problem of the dynamics of producing calcium silicate. From Figure 6, it could be known that the iron was in the form of particle package, whose outer layer was oxides of solid soluble O, Al, Si, Ca and so on. Iron was mixed with cementitious material, which need to be separated.

Table 3 The compositions of experimental reactants

| Nickel slag | CaO   | C    |
|-------------|-------|------|
| 51,59       | 43,44 | 4,97 |

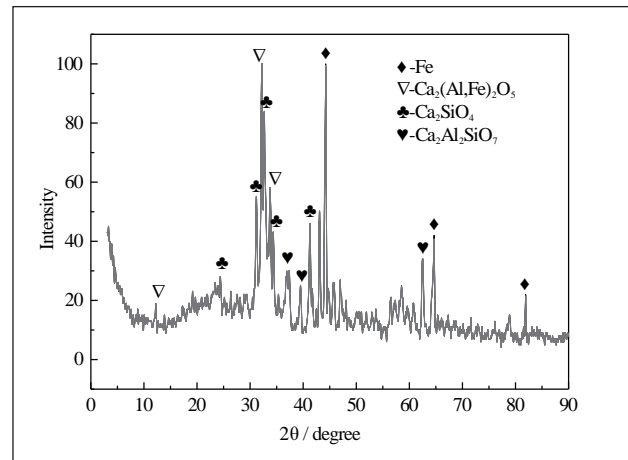


Figure 5 XRD of nickel slag after roasting.

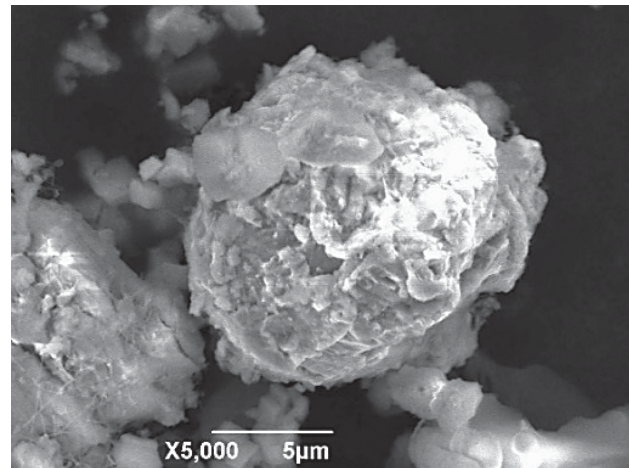


Figure 6 SEM image of nickel slag after roasting.

Comparing the forming mechanism of the C<sub>3</sub>S in the sintering process of cement raw meal, the section heating method, “Two-section method heats up”, was designed. The first section was to reduce Fe, whose reaction temperature was 800 °C - 1000 °C. In this temperature range and last for certain time, Fe<sup>2+</sup> could be reduced to iron. The purpose of the second heating was to set the temperature at the C<sub>3</sub>S-generated interval so that the calcium silicate could be fully to produced. Because of the temperature of producing C<sub>3</sub>S was 1350 °C - 1500 °C, so the second temperature used this interval.

50 g nickel slag was used in this experiment, with alkalinity of 2,6 and reducing agent 15 % content, particle 200 mesh. The roasting temperature of first section was set at 1000 °C, heat preservation 40 min, the temperature of second section was set at 1480 °C, heat preservation 90 min, with argon as shielding gas preventing iron oxidation. The material was water quench-

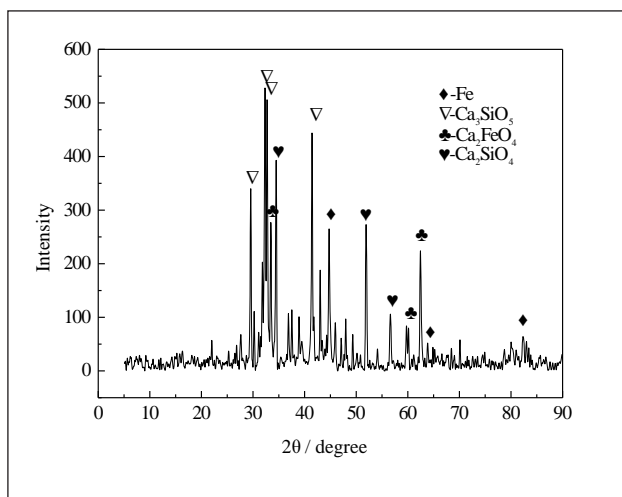


Figure 7 XRD of nickel slag after two section roasting.

ing quenched, the reduction rate of the iron is 72 %,  $C_3S$  was 55 % of the total material. XRD examination of the sample phases were shown in Figure 7.

It could be known from Figure 7 that calcium silicate and calcium silicate could coexist with Fe. Therefore, the two-section heating method could be used to reduce iron and prepare  $C_2S$  and  $C_3S$ .

## CONCLUSIONS

(1) Calcium silicate can be produced at low temperature,  $Ca_3MgSi_2O_8$  appears at 850 °C. Its stable existence area is smaller and disappears at 900 °C.  $Al_2O_3$  and CaO could produce calcium aluminate at 1 100 °C. When temperature was 1 350 °C - 1 400 °C, the content of f-CaO reduced a lot and combined with calcium silicate, calcium silicate, iron and calcium silicate could coexist.

(2) The continuous heating experiment shows that when the material particle size is 200 mesh, with the reductant ratio of 15 %, the alkalinity of 2,6, the reaction temperature of 1 480 °C, the reaction time of 40 min, the iron reduction rate is the highest of 62,9 %. But there is no  $C_3S$  produced.

(3) Stepped heating method: inert atmosphere, strong reduction environment, the temperature is 1 000 °C and holds 40 min, the temperature of second stage is 1 480 °C and holds 90 min. Iron could be obtained after material quenching to after room temperature and the reduction rate of iron is 72 %, cementitious components-calcium silicate component of 55 %.

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**Note:** The responsible translator for language English is doctor J. He - Xi'an University of Architecture and Technology, China