

## SEPARATION OF IRON-NICKEL ALLOY NUGGET FROM LIMONITIC LATERITE ORE USING SELF-REDUCTION

P. Ju\*, K. Ryom, K. Hong

\* Kim Chaek University of Technology, Department of Metallurgical Engineering, Pyongyang, Democratic People's Republic of Korea

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### Abstract

To efficiently extract nickel from low grade limonitic laterite ore, a separation method of iron-nickel alloy nugget by self-reduction of coal composite limonitic laterite ore briquette was investigated. In this investigation, in order to decrease the separation temperature of iron-nickel alloy nugget, the selective reduction by control of C/O ratio was introduced and reductant added in the briquette was inadequate for the reduction of nickel and iron. Nickel was preferentially reduced in the reduction process, while iron was partially reduced due to the lack of reductant. After reduction, a certain amount of FeO existed in the reduced product. This residual FeO had a great role in the formation of low melting point slag, which could promote the formation and the separation of iron-nickel alloy nugget at relatively low temperature. In this investigation, the reduction experiments were all conducted at 1300°C. To evaluate the formation and the separation of iron-nickel alloy nugget in the reduction process, we observed the patterns of reduced products under different C/O ratio, CaO addition ratio and holding time conditions. And then the effect of C/O ratio and CaO addition ratio on nickel content of nugget and nickel recovery ratio were investigated. The results showed that 0.7 of C/O ratio, 8% of CaO addition ratio and 40min of holding time were suitable for the separation of iron-nickel alloy nugget from limonitic laterite ore. Nickel and iron content of the nugget and the nickel recovery ratio were 4.75%, 89.51% and 85%, respectively. Nuggets were easily separated from slag by crushing and screening. This separation method could be applied to any limonitic laterite ore by adjusting C/O ratio and CaO addition ratio.

**Keywords:** Limonitic laterite ore; Iron-nickel alloy nugget; Selective reduction; Self-reduction

### 1. Introduction

Two types of nickel ores such as nickel sulfide ore and nickel laterite ore are widely used in nickel production. Nickel sulfide ores are typically derived from volcanic or hydrothermal processes, while laterite ores are formed near the surface following extensive weathering of ultramafic rocks and they occur abundantly in tropical climates around the equator [1]. With the increasing demand of nickel, the decreasing reserve of nickel sulfide resources and the advantages of exploring nickel laterite, much attention has been paid to the efficient utilization of laterite ores [2].

Nickel laterite ores generally consist of three main layers [3]. The layer close to the surface is the limonitic ore body, the layer near to the bottom of the ore body is the saprolitic ore body, and the limonitic ore can be separated from the saprolitic ore by a transition zone. Because of the low nickel content and variable distribution in nickel laterite ores, these ores cannot be easily concentrated by current technologies [4].

Limonitic laterite ores with high iron content are usually used in hydrometallurgical process such as high pressure acid leaching [5], while saprolitic laterite ores containing small amount of iron, and higher levels of nickel, silica and magnesium are generally subjected by pyrometallurgical process, including reduction-smelting process to produce ferronickel alloy. The main pyrometallurgical process is rotary kiln-electric furnace (RKEF) process. However, the energy consumption of this process is high due to the high ratio of slag to metal. Therefore, many processes with low energy consumption have been researched.

Recently, considerable research efforts are being directed at developing new techniques for extracting nickel from the nickel laterite ores. The widely used method of concentrating the nickel content of these laterite ores is the selective reduction of the contained metal oxides at moderate temperature to form a metallic phase, which may then be separated from the gangue by magnetic separation. Several previous studies have explored this selective reduction method and the effects of temperature, time, reductant type

\*Corresponding author: zbx197121@163.com



and CaO content on the nickel content of magnetic separation products and the recovery ratio of nickel were investigated [6, 7]. In these methods, nickel was mainly enriched into a ferronickel phase with the reduction of nickel oxide [8].

The most promising results correlate with reduction conditions which would produce the largest ferronickel particles, increased reduction temperatures and times have been shown to result in the larger ferronickel particles, and the addition of sulfur have also resulted in an increase in the size of ferronickel particles formed [9, 10].

Ferronickel nugget separation method by direct reduction was also investigated [11]. In this method, the separated nuggets were very large and could be directly used in alloy steel production. However, the research was performed with the saprolitic laterite ore and high reduction temperature more than 1400°C.

While, in the other previous studies [12, 13], iron nugget separation method by reduction of coal composite iron ore briquette was investigated. The reduction degree of briquette and FeO amount after reduction could be controlled by changing C/O ratio, and gangue in the briquette is combined with FeO thus forming the fayalite slag with low melting temperature. This slag ensured the separation of iron at relatively low temperature (1300°C). However, magnetic iron concentrate was used as main raw material in these studies and the product was iron nugget.

In the present work, iron-nickel alloy nugget separation method by self-reduction of coal composite limonitic laterite ore briquette was investigated at 1300°C, and the effects of the amount of added coal and CaO and holding time on the separation were the focus of this investigation.

## 2. Experiments

### 2.1 Raw materials

A laterite ore from Indonesia was used in this study. The chemical composition of nickel laterite ore is shown in Table 1, and its XRD pattern is shown in Fig. 1.

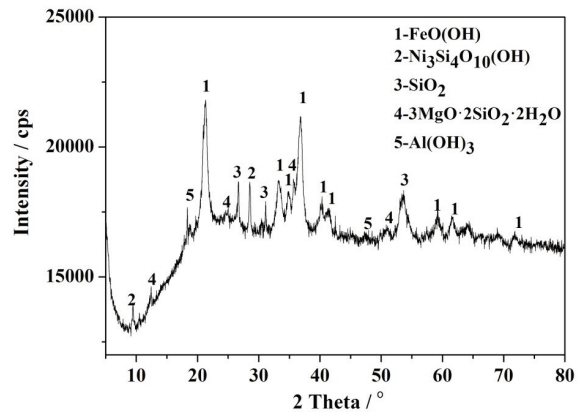
**Table 1.** Chemical analysis of nickel laterite ore (mass-%)

Ni	TFe	Al <sub>2</sub> O <sub>3</sub>	MgO	CaO	SiO <sub>2</sub>	LOI*
1.63	35.32	7.92	6.23	0.12	18.11	10.94

\*LOI: Loss of ignition

As shown in Table 1 and Fig. 1, the main mineral of the raw nickel laterite ore is goethite and this laterite ore is a limonitic laterite ore.

Powder coal was used as the reductant in the briquette and its proximate analysis and main chemical composition of ash are shown in Table 2. The chemical purity calcium oxide (CaO) powder was used as the flux in the briquette.



**Figure 1.** XRD pattern for nickel laterite ore

**Table 2.** Proximate analysis of coal and main chemical analysis of ash (mass-%)

proximate analysis			main chemical composition of ash		
C <sub>fix</sub>	V	A	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO
79.56	10.22	10.22	50.74	41.91	5.35

### 2.2 Methods

The raw laterite ore was dried at 100°C for 24 h to remove free water, and then crushed using a laboratory jaw crusher and also ground to -200 mesh using a ring pulverizer. While the coal was crushed to -100 mesh.

The powder coal and CaO reagent were added into the laterite ore, and the mixture was blended. The amount of added coal was represented as C/O ratio, which was defined as a fixed carbon to removable oxygen combined with iron and nickel molar ratio in the mixture. The amount of added CaO reagent was represented as CaO addition ratio, which was defined as a weight percentage of added CaO reagent in the mixture.

The mixtures were subsequently briquetted at 20MPa into cylindrical briquette with 20mm of diameter and 18 mm of approximate height and the weight of a briquette was about 7g.

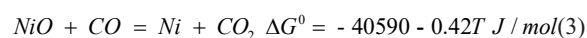
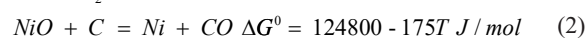
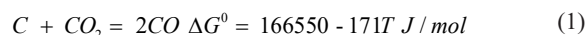
Reduction experiments were conducted in a horizontal tube furnace at 1300°C. Firstly, powder coal was charged on the bottom of refractory dish in 3~5mm of the thickness, this powder coal was just padding material, and several dried briquettes were then placed on it. The main role of padding material is to prevent the separated liquid slag adhering to bottom refractory, to provide carbon source for supplemental reduction and carburizing of iron and to assure the reducing atmosphere around the briquettes.

The refractory dish with the briquettes was placed in the furnace and held at 1300°C for a certain time. In the reduction process, the melting and separation of

slag and the cohesion of iron-nickel alloy were conducted in the reduced product. Some FeO in the separated slag was continuously reduced by padding material. After the holding time, the refractory dish with product was taken out from the furnace, then subsequently cooled to the room temperature under the argon atmosphere. Product was crushed and then separated into iron-nickel alloy nuggets and slag by screening.

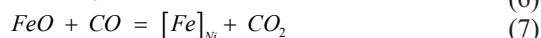
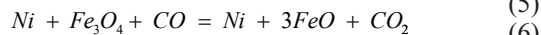
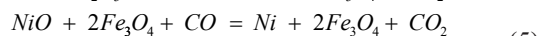
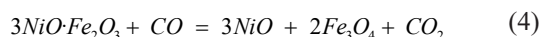
### 2.3 Thermodynamic basis

In reduction process of the coal composite laterite ore briquette, the main reactions of nickel oxide to metallic nickel are as follows [14]:



The initial temperature of NiO reduction by fixed carbon is about 440°C at the atmospheric pressure and the standard Gibbs free energy of reaction (3) is negative at the standard atmospheric pressure. Therefore, NiO is easily reduced by CO.

In fact, limonitic laterite ore consists of various components such as NiO, Fe<sub>2</sub>O<sub>3</sub>, Fe<sub>3</sub>O<sub>4</sub>, etc. Therefore, other reactions simultaneously take place in reduction process [15].



That is, nickel is preferentially reduced than iron in the reduction process.

The separation of metal nugget and slag after reduction of coal composite limonitic laterite ore briquette at low temperature of 1300°C cannot be achieved by conventional methods. To decrease the separation temperature of nugget, we used the selective reduction by control of C/O ratio in this study. The C/O ratio of coal composite limonitic laterite ore briquette is lower than theoretical requirement for the reductant. Therefore, in the reduction process, nickel is preferentially reduced, while iron is partially reduced due to the lack of the reductant. Thus, the slag system could be considered as FeO-SiO<sub>2</sub>-CaO system containing a little of MgO and Al<sub>2</sub>O<sub>3</sub> owing to the existence of FeO unreduced.

The calculated liquid region of the FeO-SiO<sub>2</sub>-CaO system under the temperature of 1300°C and different oxygen partial pressures is shown in Fig. 2 [16].

As shown in Fig. 2, the extensive liquid region existing in FeO-SiO<sub>2</sub>-CaO system. It is favorable for formation of liquid slag at 1300°C.

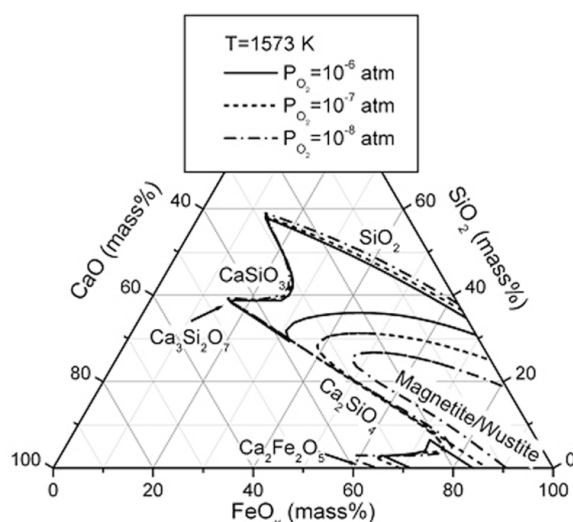


Figure 2. Liquid region of FeO-SiO<sub>2</sub>-CaO system at 1300°C and different oxygen partial pressure [16]

While the thermodynamic calculation results for the effect of MgO and Al<sub>2</sub>O<sub>3</sub> on the liquid region of the FeO-SiO<sub>2</sub>-CaO system at 1300°C [16] show that existence of MgO or Al<sub>2</sub>O<sub>3</sub> changes the position of liquid phase region, but there are almost no changes in the area of liquid region. This means that, liquid slag can be formed at 1300°C by controlling the FeO amount in the briquette after reduction. It is favorable for the formation and the separation of iron-nickel alloy nugget at 1300°C.

## 3. Results and discussion

### 3.1 Separation of iron-nickel alloy nugget

#### 3.1.1 Effect of C/O ratio

In order to investigate the effect of C/O ratio on the separation of iron-nickel alloy nuggets, the experiments were carried out with different C/O ratio. CaO addition ratio was 10% and holding times were

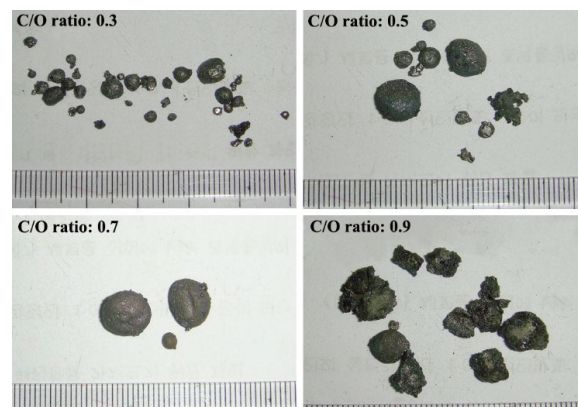
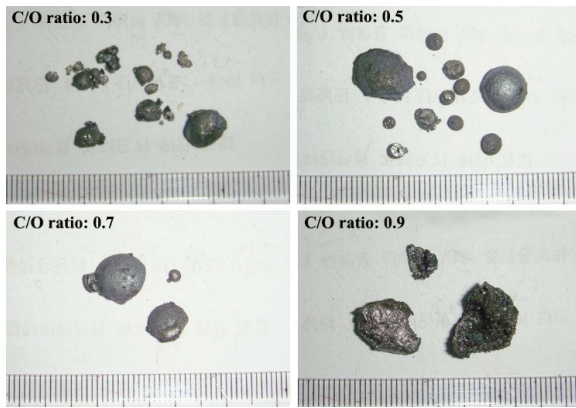


Figure 3. Morphologies of separated iron-nickel alloy nuggets or products after reduction at different C/O ratio (CaO addition ratio was 10% and holding time was 40 min)





**Figure 4.** Morphologies of separated iron-nickel alloy nuggets or products after reduction at different C/O ratio (CaO addition ratio was 10% and holding time was 60 min)

40 and 60 min respectively. Two briquettes were used in each experiment. Morphologies of separated iron-nickel alloy nuggets or products are shown in Fig. 3 and Fig. 4.

As shown in Fig. 3 and Fig. 4, the effect of C/O ratio on the cohesion and the growth of iron-nickel alloy nuggets was remarkable. However, holding time had a little effect. When C/O ratio was 0.9, iron-nickel alloy nuggets were not all formed and slag was partially melted on the surface of the briquettes. Moreover, products still appeared in a sponge state. When C/O ratio was 0.3, the slag and metal were separated. But the iron-nickel alloy nuggets were not cohered and grown, and they still existed in small sizes, their sizes were 1~4mm and 2~6mm at 40min and 60min of holding time, respectively. When C/O ratios were 0.5 and 0.7, the big iron-nickel alloy nuggets with 7~9mm of diameter were formed from each briquette, and some of the small nuggets with diameter of 2~3mm were formed as well.

These are the possible reasons: first, the high C/O ratio decreases FeO content of the reduced product so that the liquid slag cannot be easily formed at 1300°C; second, the low C/O ratio decreases the amount of reduced metal, which is unfavorable for the cohesive and growth of iron-nickel alloy nuggets.

There are two kinds of reduction reactions in the reduction experiment of this study: “internal reduction” (reduction by carbon added in briquette) and “external reduction” (reduction by carbon in padding material). In the first stage, nearly all of nickel and partial iron are reduced by internal reduction. At the end of this stage, the slag with low melting temperature is formed in the briquette. In the reduction process, inner temperature of the briquette cannot be reached at the temperature of atmosphere due to endothermic reduction reaction. After reduction, with the increase of inner temperature of briquette, the slag is gradually melted and separated from the reduced metal, and the nugget is formed. In

**Table 3.** Slag composition calculated after reduction

C/O ratio	Slag composition/%				
	Al <sub>2</sub> O <sub>3</sub>	MgO	CaO	SiO <sub>2</sub>	FeO
0.3	8	6.15	11.59	18.09	56.17
0.5	9.48	7.17	13.92	21.29	48.14
0.7	11.7	8.72	17.4	26.1	36.08
0.9	15.4	11.31	23.18	34.11	16

the next stage, partial FeO in the separated slag is reduced by external reduction at the interface between separated slag and padding material, formed metal particles are merged in to the nugget. Therefore, on the viewpoint of the separation, the composition of slag formed after internal reduction is very important.

In order to evaluate the possibility of liquid slag formation, the slag composition after reduction was calculated theoretically. Table 3 shows the slag compositions calculated after reduction under different C/O ratio conditions. The theoretical FeO content could be calculated according to the following assumptions: (1) 1mol of C removes 1mol of O; (2) the gas product is CO in the reduction process; (3) all of unreduced iron existed in the form of FeO.

As shown in Table 3, FeO content of the slag changed in the very wide extent from 16% to 56.17% under different C/O ratios. When FeO content was high, fayalite slag with low melting temperature could be formed in the reduced product. It was favorable for the separation of nugget and slag.

The experimental results show that determination of the optimum C/O ratio is very important in order to separate the iron-nickel alloy nugget.

### 3.1.2 Effect of CaO addition ratio

In this study, the growth of the nugget depends on the solid state diffusion of reduced metal in molten slag. Therefore, it is very important to reduce the viscosity of molten slag. The addition of CaO could decrease the viscosity of slag, slag with low viscosity can be formed by controlling CaO addition ratio. It is favorable for the separation of nugget at 1300°C.

In order to investigate the effect of CaO addition ratio on the separation of nugget, the experiments were carried out with different CaO addition ratio. C/O ratio was 0.7 and holding time was 40min in this experiment.

Experimental results show that when CaO addition ratio was less than 4%, the nugget was not separated (see Fig. 5). However, when CaO addition ratio was more than 6%, the nuggets were separated, and morphologies of separated nuggets and slags are shown in Fig. 6 and Fig. 7.

As shown in Fig. 6, although iron-nickel alloy nugget was separated at 6% of CaO addition ratio, the

nugget was still hidden in the slag due to insufficient separation. Moreover, when CaO addition ratios were 8% and 10%, the nuggets were separated from the slag completely (see Fig. 7).

With the increase of CaO addition ratio, the separation was considerably improved. High CaO addition ratio was favorable for the separation of the nugget. This means that it is possible to separate the iron-nickel alloy nugget directly at 1300°C by controlling C/O ratio and CaO addition ratio of coal

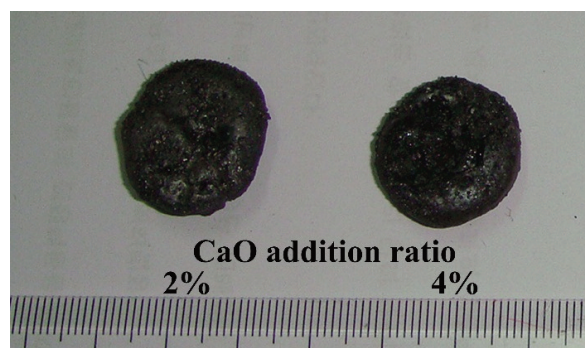


Figure 5. Morphologies of products after reduction (CaO addition ratios were 2% and 4%)

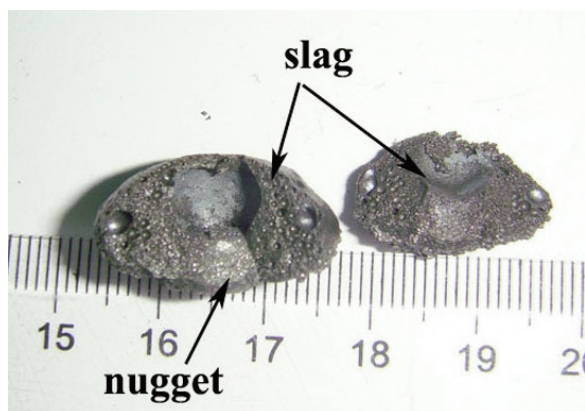


Figure 6. Morphologies of separated iron-nickel alloy nugget and slag (CaO addition ratio was 6%)

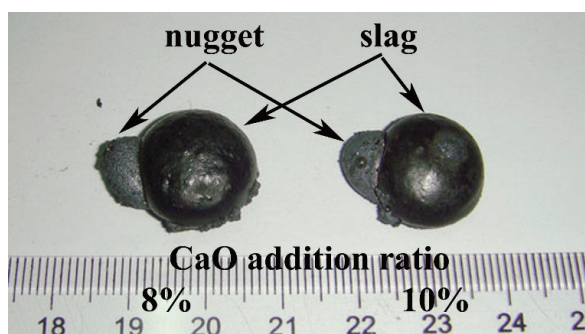


Figure 7. Morphologies of separated iron-nickel alloy nuggets and slags (CaO addition ratios were 8% and 10%)

composite limonitic laterite ore briquette.

Thus, in the viewpoint of separation, the suitable ranges of C/O ratio and CaO addition ratio were 0.5~0.7 and 8~10%, respectively.

### 3.2 Nickel content and nickel recovery ratio

#### 3.2.1 Effect of C/O ratio

In order to investigate the effect of C/O ratio on the nickel content of nugget and the nickel recovery ratio, some experiments were carried out under the control condition with varying C/O ratio. CaO addition ratios were 8 and 10%, holding time was 40min. The experimental results are shown in Fig. 8.

The nickel recovery ratio was calculated by the following expression:

$$\text{nickel recovery ratio} = \frac{\text{amount of nickel in nuggets}}{\text{amount of nickel in briquettes}} \times 100$$

As shown in Fig. 8 (a), the nickel content is the highest at 0.6 of C/O ratio. Under the condition of fixed CaO addition ratio, the nickel content of nugget and the nickel recovery ratio were mainly related to

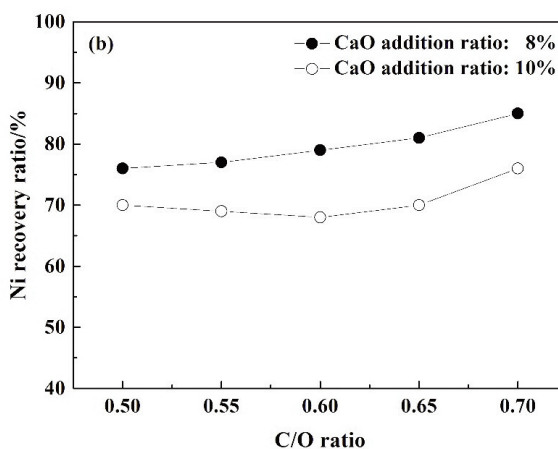
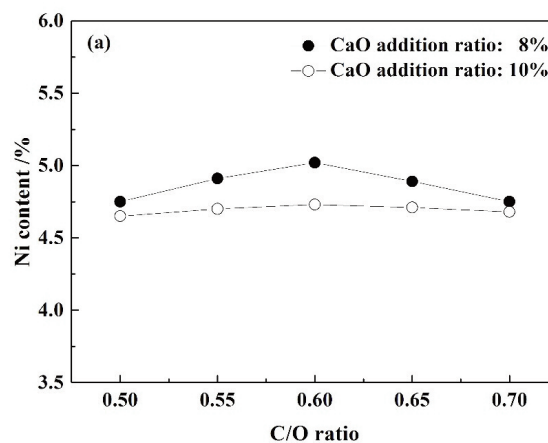


Figure 8. Effect of C/O ratio on nickel content (a) and nickel recovery ratio (b)

the amount of reduced iron. The low C/O ratio resulted in less internal reduction of iron. It was unfavorable for collection of nickel. However, it was favorable for the external reduction of iron due to the high FeO content of slag, and the increase of the total amount of reduced iron resulted in the low nickel content. With the increase of C/O ratio from 0.5 to 0.6, more amount of nickel was collected due to the increase of iron reduced by internal reduction. Because the external reduction of iron was deteriorated, the total amount of reduced iron was decreased and the nickel content was increased. However, with the increase of C/O ratio from 0.6 to 0.7, the total amount of reduced iron was increased due to increasing of internal reduction of iron, and the nickel content was decreased.

However, the nickel recovery ratio was continuously increased approximately with the increase of C/O ratio (see Fig. 8 (b)). When CaO addition ratio was 8% and C/O ratios were 0.5, 0.6 and 0.7, the nickel recovery ratios were 76%, 79% and 85%, respectively. The high C/O ratio resulted in the larger amount of iron reduced by internal reduction,

which was favorable for the collection of nickel. The above experimental results show that 0.7 of C/O ratio is suitable for this separation method.

### 3.2.2 Effect of CaO addition ratio

Under the control condition with varying CaO addition ratio, the experiments were carried out. C/O ratio were 0.6 and 0.7, while the holding time was 40min. The experimental results are shown in Fig. 9.

As shown in Fig. 9, with the increase of CaO addition ratio from 6% to 8%, the nickel content and the nickel recovery ratio increased. And, with the increase of CaO addition ratio from 8% to 10%, both decreased.

Under the condition of fixed C/O ratio, the amount of reduced iron was almost constant and the nickel content and the nickel recovery ratio were mainly related to the conditions of collecting the nickel particles. Here, the viscosity of slag was the important condition.

When CaO addition ratio was 8%, the viscosity of the separated slag could be lower than the other CaO addition ratios. It was favorable for the collection of nickel particles. This is possible due to the fact that the nickel content and the nickel recovery ratio at 8% of CaO addition ratio are higher than the other CaO addition ratios.

Therefore, the suitable CaO addition ratio for the separation of the nugget could be estimated as 8% in this study.

### 3.2.3 Analytic results of iron-nickel alloy nuggets

Under the suitable condition with 40 min of holding time, 0.7 of C/O ratio and 8% of CaO addition ratio, the separation experiment was carried out. Iron-nickel alloy nuggets were easily separated from the slag by crushing and screening the reduced product. Fig. 10 shows XRD pattern for produced nugget.

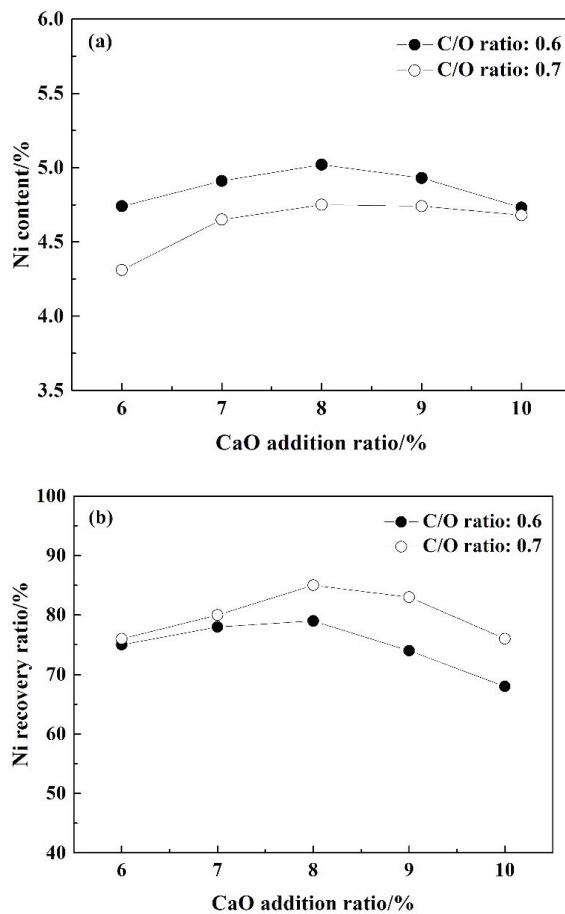


Figure 9. Effect of CaO addition ratio on nickel content (a) and nickel recovery ratio (b)

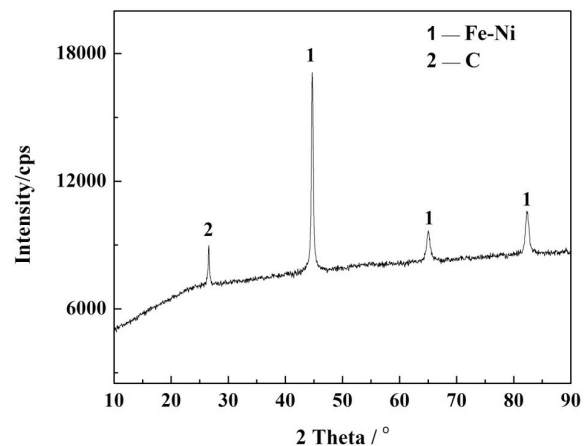


Figure 10. XRD pattern for iron-nickel alloy nugget



The analytic result show that the main phases of iron-nickel alloy nugget are Fe-Ni and C, and there are almost no slag components.

The SEM image and the EDS results of the nugget are shown in Fig. 11 and Table 4.

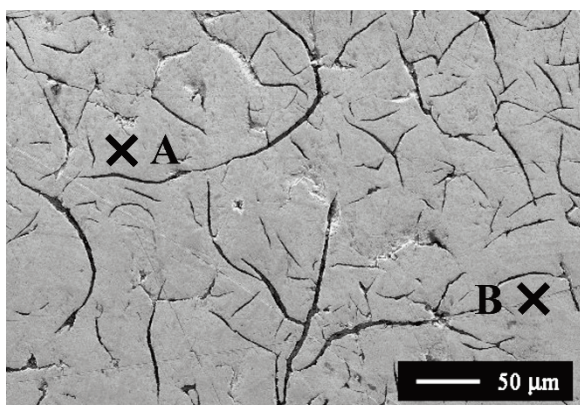


Figure 11. SEM image of iron-nickel alloy nugget

Table 4. EDS results of iron-nickel alloy nugget (mass-%)

Point	Content/%		
	Fe	Ni	C
A	90.7	4.9	4.4
B	91.1	4.6	4.3

As shown in Fig. 11, there were no distinct differences in the phase, but there were only some cracks. These cracks could be formed due to the cohesion of semi-molten metal particles.

The SEM image and the EDS result for two points selected randomly show that the nugget was sufficiently separated from the slag, and the composition was relatively uniform in the whole section.

The results of the chemical analysis show that the contents of iron and nickel were 89.51% and 4.75% respectively, and the nickel recovery ratio was about 85%.

#### 4. Conclusions

An iron-nickel alloy nugget separation method by reduction of coal composite limonitic laterite ore briquette at relatively low temperature of 1300°C was investigated in the present study, and the conclusions could be summarized as follows:

(1) Experimental results show that it is possible to separate the iron-nickel alloy nugget directly at relatively low temperature of 1300°C by the control of C/O ratio and CaO addition ratio of coal composite limonitic laterite ore briquette.

(2) The effect of C/O ratio and CaO addition ratio

on cohesion and growth of iron-nickel alloy nugget was very remarkable, and the suitable C/O ratio and CaO addition ratio for this method were 0.5~0.7 and 8%~10%, respectively.

(3) 0.7 of C/O ratio and 8% of CaO addition ratio were suitable for the high nickel content and nickel recovery ratio. After reduction the nugget was easily separated from the slag by crushing and screening. Under the suitable condition, the contents of iron and nickel of separated nugget were 89.51% and 4.75% respectively, and the nickel recovery ratio was about 85%. There were no slag components in the nuggets.

This method can be applied to any limonitic laterite ore by adjusting C/O ratio and CaO addition ratio.

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## ODVAJANJE GRUMENA LEGURE GVOŽĐA I NIKLA IZ LIMONITNE LATERITNE RUDE PUTEM SPONTANE REDUKCIJE

P. Ju\*, K. Ryom, K. Hong

\* Kim Ček tehnološki univerzitet, Odsek za metalurško inženjerstvo, Pjongjang, Demokratska Narodna Republika Koreja

### Apstrakt

Da bi se postiglo efikasno dobijanje nikla iz limonitne lateritne rude niže klase ispitivana je metoda odvajanja grumena legure gvožđa i nikla putem spontane redukcije briketa koji se sastojao od uglja i limonitne lateritne rude. Za potrebe ovog istraživanja korišćen je postupak selektivne redukcije tokom kojeg je kontrolisan odnos C/O da bi se smanjila temperatura odvajanja grumena legure gvožđa i nikla, a redukcijsko sredstvo koje je korišćeno nije bilo adekvatno za redukciju nikla i gvožđa. Redukcija nikla je imala prednost tokom redukcionog postupka, dok je gvožđe delimično redukovano usled nedostatka redukcijskog sredstva. Nakon redukcije ustanovljeno je postojanje određene količine FeO u proizvodu koji je nastao ovim putem. Preostali FeO je imao značajnu ulogu u uspostavljanju niske tačke topljenja šljake, što je uticalo na stvaranje i odvajanje grumena legure gvožđa i nikla na relativno niskoj temperaturi. Tokom ovog istraživanja svi redukciono eksperimenti su izvedeni na 1300°C. Da bi se procenilo stvaranje i odvajanje grumena tokom redukcionog postupka posmatrani su modeli redukcijskog proizvoda sa različitim odnosom C/O, dodatkom CaO tokom različitog vremena držanja. Nakon toga su ispitivani efekti različitih odnosa C/O i dodavanja CaO na sadržaj nikla u grumenu, kao i na procenat dobijanja nikla. Rezultati su pokazali da je odnos C/O 0,7, uz prisustvo 8% CaO tokom perioda od 40 min pogodan za odvajanje grumena legure gvožđa i nikla iz limonitne lateritne rude. Sadržaj nikla i gvožđa u grumenu i procenat dobijenog nikla su procentualno iznosili 4,75%, 89,51% i 85%. Grumeni su se lako odvajali od šljake drobljenjem i ekraniranjem. Ovaj metod odvajanja se može primeniti za bilo koju limonitnu lateritnu rudu prilagođavanjem odnosa C/O i dodavanjem određene količine CaO.

**Ključne reči:** Limonitna lateritna ruda; Grumen legure gvožđa i nikla; Selektivna redukcija; Spontana redukcija

