#### **CHAPTER IV**

## PROCESSING OF IRON NUGGET FROM LOW GRADE IRON ORE

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### 4.1 Abstract

The production of raw metals has been changed for several years. Blast furnace and gas-based direct reduction have been used to produce industrial metals from iron ore, but they have many drawbacks such as high capital cost, a lot of slag in the product, and environmental problems. A solution for these issues is a new technology known as IT Mark Three (Itmk3). This study evaluates the ITmk3 process to produce iron nuggets and the parameters that control the quality of the iron nuggets, such as weight ratios of feeds, the reduction time, and the reduction temperature. The goal of this work is to find the optimal conditions to produce the iron nuggets from a low grade iron ore (40% iron content). The drop test results show the suitable condition (endure up to 6 drops) for making a pellet, with a diameter of 4 cm and a height of 2 cm, by using the mole ratio of Bentonite Fe = 0.035. For the reduction, the suitable conditions for making iron nugget are the mol ratios of C/Fe = 1.6, Limestone/Al<sub>2</sub>O<sub>3</sub>+SiO<sub>2</sub> = 0.65, the reduction temperature of 1300 °C, and the reduction time of 60 minutes.

Keywords: Iron Nugget, ITmk3, Direct Reduction, Low grade iron ore

### 4.2 Introduction

The world steel markets, especially the iron market, have been changed for the several years due to growing demands. Additionally, a higher production cost comes from the transportation cost. The mass production method, which has been used by the iron and steel manufacturers, is no longer effective, as the rising cost condition becomes more critical today.

Kobe Steel and Midrex have been seriously concerned about this problem. They have developed various iron making processes. Typically, the iron making methods are the blast furnace (Kurunov et al, 2010) and the natural gas-based direct reduction (DR) (Harada and Tanaka, 2011) which have many disadvantageous issues: high capital and maintenance costs (Seki, 2008), a lot of slag in the product. and environmental problems. The solution of these issues is a new technology known as IT Mark Three (ITmk3) (Rutherford, 2009).

ITmk3 has a number of advantages in the iron making. It uses iron ore fines and a non-coking coal (Anameric and Kawatra, 2004). It can be used to produce a high-quality pig iron from lower-grade iron ores. There is no FeO attack to the refractory. Slag is clearly separated from the metal. The reduction and slag separation occur within one step. There is no hot metal handling required. The plant is easy to start up and shut down, and it is relatively safe to operate. (Negami, 2001, Dash and Das, 2009, Harada, et al, 2005)

Producing iron with ITmk3 process. A binder are required to improve pellet green strength (Kawatra and Ripke, 2002). The binder is needed to improve the strength of pellet by the cohesive force of the viscous binder (Forsmo, et al, 2008). The most common binder is bentonite, in which an addition of 10%wt of water is usually made (Forsmo, et al, 2006).

During the ITmk3 process, the following reactions take place which result in iron nugget formation: thermal decomposition of coal, reduction of iron oxide, slag formation, caburization, iron melting, and slag separation (Anameric and Kawatra, 2004). The reaction steps are given as follows (Haque and Ray, 1995, Nascimento, et al, 1997, 1998 and 1999, Zervas, et al, 1996):

$Coal \leftrightarrow CO, CO_2, H_2, N_2, C_nH_m, C$	(1)
$C + O_2 \leftrightarrow CO_2$	(2)
$CO_2 + C \leftrightarrow 2CO$	(3)
$C + \frac{1}{2}O_2 \leftrightarrow CO$	(4)
$CH_4 + CO_2 \iff 2CO + 2H_2$	(5)
$C + H_2 O \iff CO + H_2$	(6)
$Fe_3O_4 + C \iff 3FeO + CO$	(7)
$FeO + C \leftrightarrow Fe + CO$	(8)
$Fe_3O_4 + CO \leftrightarrow 3FeO + CO_2$	(9)
$FeO + CO \leftrightarrow Fe + CO_2$	(10)
$Fe_3O_4 + H_2 \leftrightarrow 3FeO + H_2O$	(11)
$FeO + H_2 \leftrightarrow Fe + H_2O$	(12)
$CaCO_3 \leftrightarrow CaO + CO_2$	(13)
$CaO + SiO_2 \leftrightarrow CaSiO_3$	(14)
$SiO_2 + 2C \iff Si + 2CO$	(15)
$FeO + SiO_2 \leftrightarrow FeSiO_3$	(16)
$FeSiO_3 + CaO \leftrightarrow CaSiO_3 + FeO$	(17)
$FeS + CaO + C \leftrightarrow CaS + FeO + CO$	(18)
$3Fe + C \leftrightarrow Fe_3C$	(19)

Thermal decomposition of coal is shown in reaction 1. Reducing gas regeneration reactions are represented by reactions 2 through 6, taking place in the same reactor in which the iron nuggets are produced. Reaction 3 of the reducing gas regeneration reaction is known as the Boudouard reaction (Rao, 1971). The direct reduction reactions, in which solid carbon reacts with solid magnetite and wusite, are represented by reactions 7 and 8. The indirect reduction reaction, carbon monoxide reacts with solid magnetite, is represented by reaction 9. The indirect reduction reaction, where carbon monoxide reacts with wusite, is represented by reaction 10. This reaction is the rate controlling step for iron ore reduction (Ghosh and Tiwari, 1970, Srinivasan and Lahiri, 1977, Mourao and Capocchi, 1996). Other endothermic reactions, where hydrogen is the reducing agent are represented by reactions 11 and

12. Some of the slags forming reactions are represented by reactions 13 through 18. Reaction 19 is the carbon diffusion into the metal. It occurs at a lower melting temperature of the metal (Anameric, et al, 2008).

Cheawchanpattanagone (2010) studied about processing of iron nugget from low grade iron ore. The iron ore containing 55% of iron content. The suitable conditions for making iron nugget are by using the molar ratios of C/Fe = 1.53, Limestone/Al<sub>2</sub>O<sub>3</sub>+SiO<sub>2</sub> = 0.75, and Bentonite/Fe = 0.02 with the reduction temperature of 1425 °C and the reduction time of 20 minutes.

Anameric and Kawatra (2007) studied the ironmaking process, they used reduction temperatures from 1400 to 1500 °C and 50% Fe of iron ore. The pig iron nugget contained 95-97% of Fe by using the reduction temperature of 1475 °C and the reduction time of 28 minutes.

For this thesis, the lower grade of iron ore was used. The iron ore contained about 45% of iron content. The study of hardness of the pellet was carried out by the drop test. The reduction temperature was minimized to provide energy saving.

The purpose of this investigation is to study and optimize the parameters for making iron nuggets from a low grade iron ore using the ITmk3 process on a laboratory scale. This study is focused on 5 parameters, they are the reduction time, the reduction temperature, the molar ratios of Bentonite/Fe, the molar ratios of C/Fe, and the molar ratios of Limestone/Al<sub>2</sub>O<sub>3</sub>+SiO<sub>2</sub>.

## 4.3 Experimental

#### Raw Materials

Iron ore from Phu Khoud was the source of iron oxide for the iron production. In the present case, a low grade Iron ore has % Fe of 41.62%, (XK-03). The wt % of elements of the XK-03 are tabulated in Table 2. Reductant is a substance used to reduce oxygen in an iron ore. In the present work, the FIRST coal has a fixed carbon content of 65.3%. Limestone, from Petch Thai Chemical Co., Ltd. (99.96% wt CaCO<sub>3</sub>), is a flux used to separate iron from slag or other impurities. The sodium bentonite from Dhebkaset Industry Co., Ltd. is a binder used in our experiments.



**Figure 1** The sample preparation: (a) Grinding of raw material; and (b) Mixing and the pellet preparation.

## Sample Preparation

### Grinding of Raw Material

The XK-03 and FIRST coal were grinded by a cylindrical ball mill. The diameter and length of the chamber were 70 cm and 100 cm respectively. The media was 1 kg of spherical metallic ball, with 15 balls. The critical speed was 60 rpm. The product was screened by a mesh 20 (300  $\mu$ m) and the oversize was grinded again. The grinding step is shown in Figure 1a. The particle size after grinding was measured by PSA (Malvern, Mastersizer X).

#### Mixing and the Pellet Preparation

XK-03, FIRST Coal, Limestone, and Bentonite were mixed by using mole ratio as tabulated in Table 1. Water of 10% by weight of the mixture was added. The mixture was mixed until it became homogenous phase. The mixture was compressed by a cylindrical mold (4 cm of diameter) for making pellets. The pellets were compressed at 6,000 psi, 2 minutes and then dried at 80 °C for 24 hours. The pelletizer step is shown in Figure 1b. The sizes of the pellets are shown in Figure 2.



Figure 2 Size of the pellet after pelletizer.

[				Mol ratio	Weight (g)				
Experiment	No.	Fe	C/Fe	Limestone/ Al <sub>2</sub> O <sub>3</sub> +SiO <sub>2</sub>	Bentonite/Fe	XK-03	FIRST Coal	Limestone	Bentonite
	1-2				0.016				2 03
1	3-6	1	1.6	0.49	0.075	200	44.91	47.52	3 39
	7-10				0.085				4.74
2	1-3	1	1.6	0.40	0.038	200	44.01	47.52	4.74
2	4-6	1	1.0	0.49	0.082	200	44.91	47.32	10.17
3	1-4	1	1.6	0.49	0.038	200	44.91	47.52	4.74
	1		1.4				38.38		
	2		1.6				43.86		
4	3	1	1.8	0.75	0.035	200	49.34	73.26	4.34
	4	]	2.0				54.83		
	5		2.2				60.31		
	1	1		0.45				43.95	
	2			0.55				53.72	
	3			0.65				63.49	
5	4	1	1.6	0.75	0.035	200	43.86	73.26	4.34
	5			0.85				83.03	
	6			0.95				92.79	
	7			1.05				102.56	
6	1-3	1	1.6	0.65	0.035	200	43.86	63.49	4.34
	1-3				0.025				3,10
7	4-6	1	1.6	0.65	0.035	200	43.86	63.49	4.34
	7-9				0.045				5.58

**Table 1** The mol ratios and the weight of the raw materials in the mixture

This work is separated in seven experiments. The molar ratios and the weight of mixtures are tabulated in Table 1. The experiment 1 and 2 are study of the effect of the molar ratio of Bentonite/Fe on the hardness of the pellet. The ratio of Bentonite/Fe was varied to determine the optimal condition provided by drop test. For the other components, such as molar ratios of C/Fe and Limestone/Al<sub>2</sub>O<sub>3</sub>+SiO<sub>2</sub>, the optimal condition from previous work (Cheawchanpattanagone, 2010) were used. Followed by experiment 3, this experiment is to study the effect of the reduction time and the reduction temperature from 30 to 60 minutes of reduction time and 1200 to 1300 °C of reduction temperature.

Experiment 4 is study of the effect of molar ratio of C/Fe. The molar ratio of C/Fe were varied from 1.4 to 2.2 using the optimal reduction time and the reduction temperature from experiment 3. Experiment 5 is the study the effect of molar ratio of Limestone/Al<sub>2</sub>O<sub>3</sub>+SiO<sub>2</sub>. The molar ratio of Limestone/Al<sub>2</sub>O<sub>3</sub>+SiO<sub>2</sub> was varied from 0.45 to 1.05 by using the other parameters from previous experiment.

After the optimum condition was observed, the experiment 6 was carried out to provide another result on the additional reduction time from 45 to 90 minutes. The hardness of the pellet was tested again by the drop test. The molar ratio of Bentonite/Fe was varied from 0.025 to 0.045 by using other parameters from previous experiments.

#### Drop Test of the Pellet

The pellet was dropped to the ground (polished stone floor) from one meter high at room temperature until it breaks. From experiment 1 and 2, the molar ratio of Bentonite/Fe was varied to determine the strongest condition of the pellet.

### Reduction

The pellets were reduced by a furnace (Protherm Furnace, model PLF130/6). The pellets were fed at room temperature and heated to a desired temperature, which was held on during reduction period. After the reduction period, the samples were cooled down for 24 hours. In Experiment 3, the reduction temperature and soaking time were varied as shown in Table 2 to minimize the raw materials usage as tabulated in Table 1.

#### **Characterization**

The XK-03 and the products were characterized for finding the wt % of elements by an energy dispersive X-Ray fluorescence spectrometer (Horiba, model 51-ADD0014), connected to a scanning electron microscope (Hitachi, model S-4800, 25 V, 20 mA). The particle sizes after grinding were measured by a particle size analyzer (Malvern, Mastersizer X). The XK-03 iron ore was also analyzed for its compositions by the X-Ray fluorescence (PANalytical, model AXIOS PW4400) and the wet chemical analysis at Thai Pride Cement Co., Ltd. FIRST coal was carried out to give the result as the proximate analysis.

## 4.4 Results and Discussion

### Raw Material Characterization

The characteristic of the raw materials are tabulated in Table 2 and 3.

**Table 2** Composition of the XK-03 iron ore: (a) XRF characterization; (b) EDXcharacterization; and (c) Wet chemical analysis

(a)						
Element	% wt					
Al	4.824					
Ca	0.07098					
Cs	0.5234					
Fe	41.62					
К	0.8889					
Mg	0.3062					
Mn	2.911					
Na	0.1928					
0	36.91					
Р	0.3598					
S	0.026					
Si	11.2					
Ti	0.1676					

	(b)
Elements	Average % wt
С	0.00
0	40.39
Al	4.54
Si	6.53
Mn	2.20
Fe	45.46
Zr	0.00
Ca	0.14
Р	0.28
K	1.11
Mg	0.00
Cl	0.00
Na	0.02
Zn	0.00
Ti	0.08
As	0.00
S	0.00
Cr	0.00

(c)						
Composition	% weight					
SiO <sub>2</sub>	20.1					
Al <sub>2</sub> O <sub>3</sub>	7 59					
Fe <sub>2</sub> O <sub>3</sub>	56.77					
CaO	0.80					
MgO	1.92					
SO3	0.00					
Na <sub>2</sub> O	0.39					
K <sub>2</sub> O	() ()()					
LOI	10.51					

For the characterization of XK-03, the XRF characterization shows the amount of Fe equal to 41.62%. The EDX characterization results give 45.46% of Fe in XK-03. The wet chemical analysis shows 56.77% of Fe<sub>2</sub>O<sub>3</sub>.

The XK-03 was characterized by XRF, EDX, and the wet chemical analysis. For the experiment, the XRF characterization results of XK-03 have been chosen to calculate the molar ratios and the weights of the component in the pellet mixture. This method can determine the % element from the whole sample or in bulk (EDX probes only a specific area of the sample), thus giving more accurate data.

Moisture	% Total Moisture	1.23
	% Inherent Moisture	0.97
	% Ash	23.84
Properties . Air dried basis	Volatile Matter	9.89
roperties. All uneu basis	% Fixed Carbon	65.30
	% S	0.49
	Gross Calorific Value (Kcal/kg)	6199.00

### Table 3 Proximate analysis of the FIRST coal

To calculate amount of coal used in the experiment, the amounts of carbon in coal and the amount of Fe in iron ore were investigated next. The amount of fixed carbon was used to convert the amount of coal to carbon. The proximate analysis of FIRST coal shows the % of fixed carbon to be equal to 65.30%.

#### Effect of the Bentonite/Fe Molar Ratio

The results of drop test (experiment 1 and 2) shows that ratio of Bentonite/Fe can affect the hardness of the pellet. All conditions were tested for 3 samples. At the molar ratio of Bentonite/Fe = 0.016, the pellets were broken from 1 up to 2 drops. At the molar ratio of Bentonite/Fe = 0.027, the pellets were broken from 1 up to 4 drops. At the molar ratio of Bentonite/Fe = 0.038, the pellets were broken from 3 up to 6 drops. At the molar ratio of Bentonite/Fe = 0.038, the pellets were broken from 3 up to 5 drops. The results show that the appearances of the pellet after drop test can be classified into 4 types as shown in Figure 2.

From results of experiment 1 and 2, with the drying condition at 80  $^{\circ}$ C, 20 hours and one meter height, the pellets were broken from 3 up to 6 drops. The best result is with molar ratio of Bentonite/Fe = 0.035. Figure 2 shows the different appearances of the pellets after dropping them to the floor.



**Figure 3** Appearances of the pellet from drop test: (a) Perfect pellet, sample No.2-2 after  $1^{st}$  drop time; (b) Nicked pellet, sample No.2-2 after  $2^{nd}$  drop time; (c) Crack pellet, sample No.2-3 after  $2^{nd}$  drop time; and (d) Broken pellet, sample No.2-4 after  $3^{rd}$  drop time.

# Effect of the Reduction Time and the Reduction Temperature

The weight of the pellet after the reduction, wt % Fe of iron nuggets, and % yield of experiment 3 and 6 are tabulated in Table 4.

**Table 4** Experiment 3 and 6; the weight of the pellets after reduction, wt % Fe ironnugget from EDX and % yield

No.	%wt Iron ore in mixture	%wt Fe in mixture	Dried weight of pellet (g)	Fe input (g)	%Fe Nugget from EDX	Iron Nugget (g)	Fe Output (g)	%Yield
3-1	67.30		162.22	45.44	-	-	-	-
3-2		28.01	161.34	45.19	-	-	-	-
3-3		28.01	162.81	45.60	51.17	68.43	35.02	76.78
3-4			160.33	44.91	67.35	53.28	35.88	79.90
6-1			162.81	43.48	75.64	49.32	37.31	85.8
6-2	64.17	26.71	161.97	43.26	86.60	41.42	35.87	82.91
6-3			156.4	41.77	90.67	35.03	31.76	76.04

It can be seen from Table 4 that wt % Fe increases with increasing reduction time. The conversion from hematite to magnetite and finally to pig iron increases and it affects wt % Fe in iron nugget. At a high reduction time, the % yield decreases because amount of iron nugget is lower compared to the input of Fe.

For the samples No.3-1 and 3-2, it shows that the reduction does not occur at 1200 °C. It does not produce any iron nugget or slag because the reduction needs a higher reduction temperature.

The experiment shows the optimal reduction time and temperature for producing iron nuggets are from the sample No.3-4 (the best % yield and complete separation) with 60 minutes and 1300 °C. Then the experiment 4 and 5 were carried out to determine the best ratio of feed mixtures. After getting the best ratio, the reduction time was considered again in experiment 6.



Figure 4 For Experiment 3 and 6: (a) reduction time vs. weight; and (b) reduction time vs. wt % of Fe.

From Figure 4, an increase the reduction time increases the wt % of Fe and decreases the weight of iron nuggets. From experiment 3, at the reduction time of 30 minutes, the weight of iron nugget is 68.43 g and contains 51.17% of Fe. When the reduction time increases to 60 minutes. The weight of iron nugget decreases from 68.43 to 53.28 and wt % Fe increases from 51.17% to 67.35%. From experiment 6, at the reduction time of 45 minutes, the weight of iron nugget is 49.32 g and contains

75.64% of Fe. Increasing the reduction time to 90 minutes, the weight decreases to 35.03 g and wt % Fe increases to 90.67%.

Cheawchanpattanagone (2011) studied of processing iron nugget at higher temperature (1375 to 1500 °C) at 50 minutes with molar ratios of C/Fe = 1.72, Limestone/Al<sub>2</sub>O<sub>3</sub>+SiO<sub>2</sub> = 0.89, and Bentonite/Fe = 0.02. It produced iron nugget, which contained more % of Fe (90 to 93% of Fe) because the reduction temperature, % of Fe in iron ore, and molar ratios of feeds are different. Higher reduction temperature and better grade iron ore can provide more wt % Fe of iron nugget.

## Effect of the Molar Ratios of the C/Fe and Limestone/Al<sub>2</sub>O<sub>3</sub>+SiO<sub>2</sub>

All pellets were reduced at the reduction temperature 1300 °C and the reduction time of 60 minutes. The weights of pellets after the reduction, % Fe of iron nuggets, and % yield of experiment 4 and 5 are tabulated in Table 5. The % elements of products are shown in Figure 5.

Table 5 Experiment 4 and 5; the weight of pellets after reduction, % Fe from EDXand % yield

No.	%wt Iron ore in mixture	%wt Fe in mixture	Dried weight of pellet (g)	Fe input (g)	%Fe Nugget from EDX	Iron Nugget (g)	Fe Output (g)	%Yield
4-1	63.30	26.34	162.41	42.78	77.51	47.76	37.02	86.53
4-2	62.22	25.89	161.08	41.71	84.87	43.03	36.52	87.55
4-3	61.17	25.46	163.13	41.53	83.15	36.90	30.68	73.87
4-4	60.16	25.04	162.19	40.61	80.38	39.94	32.10	79.05
4-5	59.19	24.63	160.33	39.50	77.58	32.85	25.49	64.53
5-1	68.46	28.49	158.95	45.29	68.18	58.79	40.08	88.51
5-2	66.24	27.57	157.80	43.51	73.88	53.15	39.27	90.26
5-3	64.17	26.71	160.02	42.73	83.94	46.19	38.78	90.73
5-4	62.22	25.89	160.33	41.52	80.23	46.02	36.92	88.92
5-5	60.38	25.13	162.32	40.79	82.19	38.96	32.02	78.50
5-6	58.65	24.41	164.44	40.14	79.47	41.43	32.92	82.02
5-7	57.02	23.73	163.56	38.81	73.64	39.50	29.09	74.94

It can be seen from Table 5, the amount of carbon was varied from sample No.4-1 (lower ratio of C/Fe) to sample No.4-5 (higher ratio of C/Fe) and the amount of Limestone was varied from sample No. 5-1 (lower ratio of

Limestone/Al<sub>2</sub>O<sub>3</sub>+SiO<sub>2</sub>) to sample No. 5-4 (higher ratio of Limestone/Al<sub>2</sub>O<sub>3</sub>+SiO<sub>2</sub>). Adding carbon (coal) affects the wt % Fe in iron nugget. At a low amount of coal, the reaction is not complete. Some of hematite is not converted to pig iron and still remains in the product. When increasing the amount of coal, wt % Fe increases until it reaches the optimum condition. Adding amount of coal decreases wt % Fe in iron nugget because the excess moles of coal in the product and it also decreases the % yield.



**Figure 5** (a) % Element of iron nugget vs. molar ratio of C/Fe of experiment 4; and (b) % Element of iron nugget vs. molar ratio of Limestone/Al<sub>2</sub>O<sub>3</sub>+SiO<sub>2</sub>.

In Figure 5a, samples No. 4-1 and 4-2 of experiment 4, it shows an increase of the molar ratio of C/Fe increases the wt % Fe. The molar ratio of C/Fe = 1.4 gives 77.51% Fe and it reaches the maximum point at the molar ratio of C/Fe = 1.6 giving 84.87% Fe. Samples No. 4-3 to 4-5, an increase in the molar ratio of C/Fe induces the reaction to be a complete separation. Adding the excess moles of FIRST coal decreases the wt % Fe from 84.87% (C/Fe = 1.6) to 77.58% (C/Fe = 2.2), as shown in Figure 5. The best ratio of C/Fe is from the sample No.4-2 (the best of % Fe and % yield) with the ratio of C/Fe = 1.6. For the wt % of C, an initial state shows a decrease of C because the reaction uses C as the reductant. After the reaction reaches the optimum point at molar ratio C/Fe = 1.6, % C increases.

The Figure 5b shows the increase of wt % Fe in the products occurs when increasing the molar ratio of Limestone/Al<sub>2</sub>O<sub>3</sub>+SiO<sub>2</sub>. Initial states show the increase of % Fe because limestone is combined with impurity in the iron ore and separated in

a form of slag, and wt % Fe increases from 68.18% (Limestone/Al<sub>2</sub>O<sub>3</sub>+SiO<sub>2</sub> = 0.45) to 83.94% (Limestone/Al<sub>2</sub>O<sub>3</sub>+SiO<sub>2</sub> = 0.65). Further increase in the molar ratio, wt % Fe is still constant. Increase of the moles of limestone decreases the % Fe from 83.94% at optimum point of 73.64% at Limestone/Al<sub>2</sub>O<sub>3</sub>+SiO<sub>2</sub> = 1.05.

Anameric and Kawatra (2004) investigated the production and properties of pig iron nuggets. The reduction time and the reduction temperature were 22 minutes and 1450 °C, respectively. It produced iron nuggets, as characterized by XRF with 96.49% of Fe. In comparison, the wt % Fe in this study is less compared to Anameric and Kawatra's study because the reduction temperature is less and the raw materials are different.

Comparison with Cheawchanpattanagone (2011), the best result of the reduction showed 94% of Fe in iron nugget and using molar ratios of C/Fe = 1.53, Limestone/Al<sub>2</sub>O<sub>3</sub>+SiO<sub>2</sub> = 0.75 and Bentonite/Fe = 0.02 with the reduction condition of 1425 °C and 20 minutes from iron ore that contains about 55% of Fe content. In comparison, the wt % of Fe in this study is less compared to Cheawchanpattanagone's study because of the lower grade of iron ore, the different ratio of feeds, and the lower reduction temperature.

### Phase Separation of the Product

In term of phase separation, the product can be classified into three types as shown in Figure 6.



**Figure 4.6** Appearances of the product after reduction: (a) Product No.3-1, 1200 °C and 30 minutes; (b) Product No.5-6, 1300 °C and 60 minutes; and (c) Product No.4-3, 1300 °C and 60 minutes.

It can be seen from Figure 6a that the separation is a direct reduction. The iron reduction is not complete. The metal and slag do not separate. It occurs in the reduction with a low temperature. The reaction does not occur. Figure 6b, shows the separation is a partial separation. The metal and slag separate partially. It occurs in the reduction with a high amount of limestone. Excess of limestone is an impurity which obstructs the separation of product. Figure 6c shows that the separation is a complete separation. The iron is reduced completely. The metal and the slag separate perfectly. It occurs with the optimum condition of the reduction time, the reduction temperature, and the ratios of mixture.

## Micro-analysis of iron nugget

The result of X-Ray Diffraction Spectrometer, XRD (Rigaku D/max; model 2000) of product No.5-3 is shown in Figure 7.



Figure 7 XRD diffraction pattern of iron nugget No.5-3.

For the XRD result, the X-Ray intensity of iron nugget No.5-3 is shown in Figure 7. The iron, iron carbide, hematite and magnetite were detected as represented by the X-Ray intensity peaks. The peaks at the angles = 44.674 and 65.023 represent the iron and the peaks at the angles = 37.768, 45.068, 49.212, and 58.357 represent the iron carbide. The peaks at the angles = 33.153, 54.091, 62.451, and 63.991 represent the iron oxide (hematite). The peaks at the angles = 35.423 and 73.950 represent the iron oxide (magnetite) (Aronniemi, 2004, Kugler, 2003). This means the iron nugget is produced mostly in the form of iron and iron carbide but some of hematite and magnetite do not convert to iron due to the low operating temperature used in the reduction process. The quality of iron content is improved from 41.62% to 83.94% as confirmed by the EDX results.

The XRD reference patterns of any components are shown in Appendix J (p. 238-241).

## 4.5 Conclusion

The product of the reduction can be classified into three types of phase separation: (1) Complete separation; the iron ore is reduced completely, the iron nugget and the slag are separated from each other; (2) Partial separation, the metal and the slag separate partially; and (3) Direct reduction, the reduction of iron ore is not yet complete, the metal and slag is not separated. The drop test results show the suitable condition for making a pellet. The condition for making the pellet, with a 4 cm in diameter and 2 cm in height, is at the molar ratio of Bentonite/Fe = 0.035. The experiments show the suitable conditions for making iron nugget from a low grade iron ore are at the mixture molar ratios of the mixture as C/Fe = 1.6, Limestone/Al<sub>2</sub>O<sub>3</sub>+SiO<sub>2</sub> = 0.65, the reduction temperature of 1300 °C, and the reduction time of 60 minutes.

#### 4.6 References

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