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RISET
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SYNTHESIS OF NICKEL CONTAINING PIG IRON (NCPI) BY USING LIMONITE TYPE OF LATERITIC ORE FROM SOUTH EAST SULAWESI

Pembuatan Besi Wantah Yang Mengandung Nikel Menggunakan Bijih Laterit Sulawesi Tenggara

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ABSTRACT Nickel containing pig iron (NCPI) is one of important materials for stainless steel and other iron-nickel alloys production. The natural source of NCPI in Indonesia is laterite ore. Large deposit of laterite ore has been found in South East Sulawesi. High grade laterite ore (saprolitic type of laterite ore) in this region has been used for ferronickel making, whereas low grade laterite ore (limonitic type of laterite ore) has not been processed, due to its too low nickel content. Through this recent research, low grade laterite ore has been utilized as raw material in nickel pig iron making experiment. Laterite ore was reduced by carbon at various temperatures. It has been found that reduction reaction increases with an increasing in temperature. At 1200 ^{o}C, metal phase has been formed significantly. The melting of reduced ore results in NCPI that contains 3.17 % nickel and 86.8 % iron. The analysis to NCPI morphology shows that microstructure of NCPI consist of iron-chromium layer and rich sulfur iron chromium grain in the matrix of iron nickel.

Keyword: laterite, nickel, pirometallurgy, pig iron.

ABSTRAK *Nickel containing pig iron* (NCPI) merupakan bahan baku penting dalam pembuatan baja tahan karat dan baja paduan lainnya. Sumber alami NCPI adalah bijih laterite. Cadangan bijih laterit dalam jumlah besar telah ditemukan di Provinsi Sulawesi Tenggara. Bijih laterit kadar tinggi dari wilayah ini telah diproses untuk menghasilkan ferronikel, sedangkan bijih laterit kadar rendah, karena kadar nikelnya yang terlalu rendah, tidak digunakan dalam pembuatan ferronikel. Dalam penelitian ini bijih laterit kadar rendah telah dicoba dimanfaatkan sebagai bahan baku dalam pembuatan NCPI. Terhadap bijih laterit dilakukan proses reduksi pada berbagai temperatur. Hasil pengamatan menunjukan bahwa reaksi reduksi meningkat seiring dengan naiknya temperatur proses. Pada temperatur 1200 ^{o}C telah tebentuk secara signifikan fasa logam. Hasil peleburan terhadap hasil reduksi menghasilkan NCPI dengan kadar nikel dan besi masing-masing 3,7 dan 86,8 %. Analisa morfologi terhadap hasil peleburan menunjukan bahwa NCPI yang dihasilkan mengandung lapisan kaya besi-kromiun dan butiran besi kromium yang kaya belerang dalam matrik paduan besi nikel.

Katakunci: *laterite, nickel, pirometallurgy*, reduksi, besi wantah.

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INTRODUCTION

Nickel and iron are the metals used as raw materials in the production stainless steel and special steel that contains nickel (LleweUyn *et al.*, 2000; Boland 2012). The natural source of nickel and iron in some countries, including Indonesia, is lateritic ore. The largest deposit of lateritic ore has been found in South East

Sulawesi. Some of this ore has been mined and processed to produce ferronickel, but another part of lateritic ore is low grade nickel layer that cannot be used in ferronickel making. Although this low grade layer cannot be used to make ferronickel, it can be used to make nickel containing pig iron (NCPI) (Lencou-Bareme 2010; Prasetyo 2011). This material is a main raw material in nickel based material production and it is also the additional material in stainless steel making (Lennon *et al.,* 2009; Stewart 2013). Although the quantity of low grade layer in lateritic ore is three times larger than high grade layer and some blast furnace plant in Indonesia has produced NCPI, the research to study this process in Indonesia has not been much intensively studied. Therefore, the aim of this recent research is to study the process of NCPI making by using this local low grade lateritic ore.

METHODS

Low grade nickel layer of lateritic ore was obtained from a mining site in South East Sulawesi, Indonesia. This low grade layer was ground and milled to obtain a 200 mesh (74 micron) particles. The milled layer was mixed with coal in weight ratio of coal to ore at 8:2 prior being pelletized. The pellet was put in muffle furnace to be heated at temperature range of 800-1200 ^{o}C. The oxigen needed by the process was obtained from the air, and therefore the atmosphere in the furnace was kept in air atmosphere without other gas addition.

The quantity of iron and nickel in the low grade layer of lateritic ore was measured through wet analysis by using Atomic Absorption Spectrometry (AAS). It is found that the ore contains 1.35 % nickel and 29.5 % iron. Since the nickel content in the as received sample is less than 1.6 %, this sample is classified as low grade nickel ore. X-Ray Diffraction (XRD) analysis using CuK-alpha radiation was applied to as received low grade layer and heated ore to reveal the phases exist after heating.

The morphology of heated pellet was analyzed through Scanning Electron Microscope - Energy Dispersive Spectroscopy (SEM-EDS) equipment. The XRD analysis was also applied to the heated sample.

After being smelted, metal and slag phase was separated mechanically by a hammer. The morpholiological analysis to metal phase was done through SEM-EDS. The composition of nickel containing pig iron was analyzed through Optical Emission Spectroscopy (OES). Meanwhile, the slag phase due to its less importance was not analyzed.

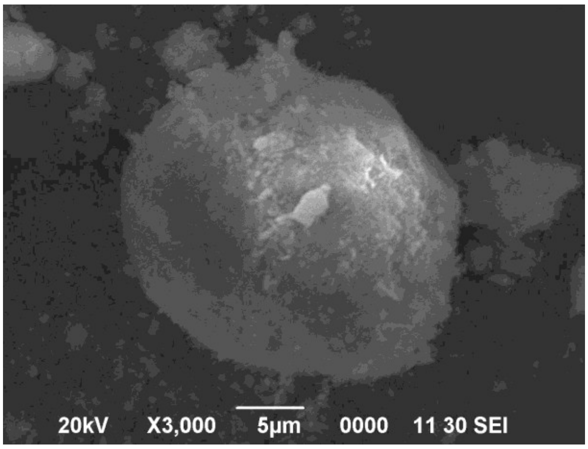

Figure 1. Typical morphology of particle in low grade lateritic ore.

RESULT AND DISCUSIONS

The typical morphology of ground and milled sample is shown in Figure 1. The result of EDS analysis of some area in this ore (Table 1) shows that, beside iron and oxygen as main element, there are also other elements in small quantity, including nickel. The minerals that compose the ore must be combination of these elements.

The minerals identified in the ore, which was shown in Figure 2, are goethite (FeOOH), magnesium silicate hydroxide ($Mg_3Si_2O_5(OH)_4$), hematite (Fe_2O_3) and quartz (SiO_2). The distribution of these minerals in the sample can be traced through mapping of each element within sample, as shown in Figure 3. Oxygen that spreads in the same area with only silicon indicates that those are the quartz area, whereas the oxygen that spreads in the same area with both silicon and magnesium indicates that those area is filled with magnesium silicate hydrate. Meanwhile based on this element mapping, iron

Table 1. EDS analysis of some area in limonite Sample.

Element	% Weight			
	Area 1	Area 2	Area 3	Average
O	58.0	41.3	49.9	49.7
Na	0.8	0.7	0.8	0.8
Mg	12.7	12.7	12.8	12.7
Al	1.3	1.3	1.4	1.3
Si	15.5	17.3	17.0	16.6
Ca	0.2	0.3	0.3	0.3
Cr	0.6	1.1	0.9	0.9
Fe	9.5	21.5	14.9	15.3
Co	0.3	0.4	0.0	0.2
Ni	1.1	3.2	2.0	2.1

should be in the form of oxide or oxy-hydroxide and nickel is in oxide form. Nickel spreads in the entire ore. Nickel, in the oxide form, is possibly trapped physically in the network of magnesium silicate hydroxide and iron oxy-hydroxide.

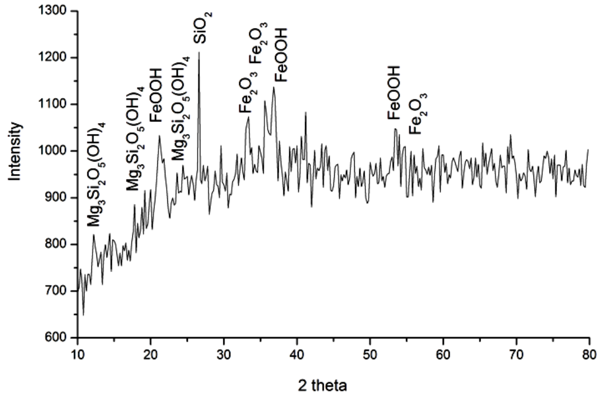

Figure 2. XRD pattern of low grade layer of lateritic.

The spreading of nickel and its being trapped in the network of magnesium silicate hydroxide and iron oxy-hydroxide makes it very difficult to be separated through conventional mineral dressing. Based on this fact, the process of mineral dressing was not applied and the low grade layer of lateritic ore was directly processed through reduction and smelting process.

The XRD pattern of the pelletized samples that was heated is shown in Figure 4. Some phases can be identified such as fosterite (Mg_2SiO_4),

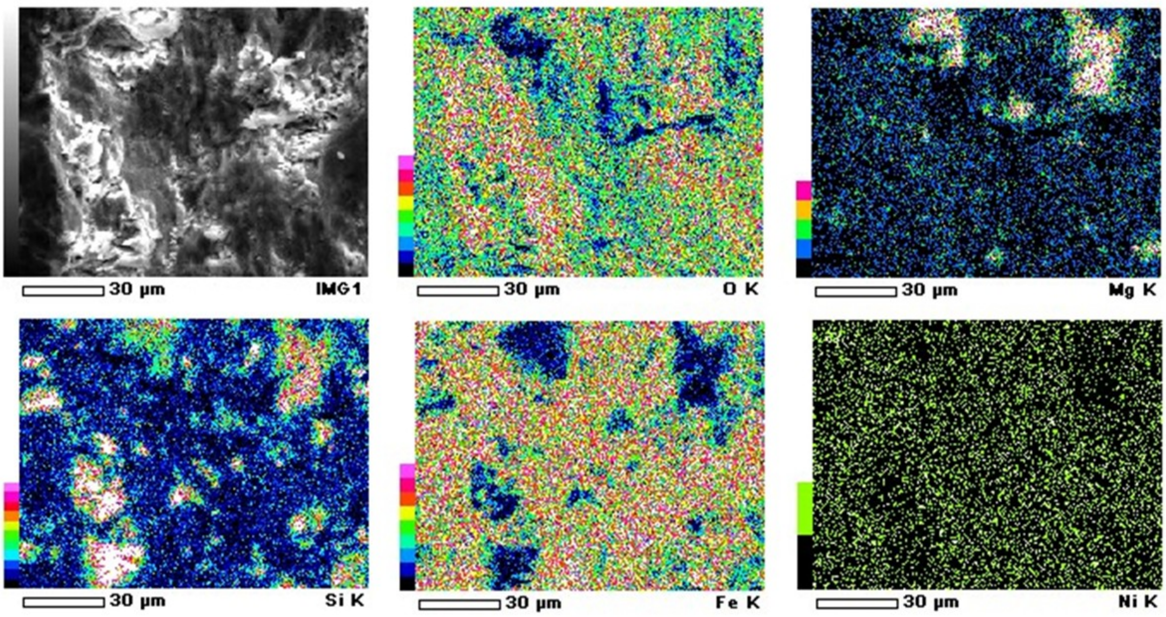

Figure 3. The mapping of elements in the low grade layer of lateritic ore.

enstatite ($MgSiO_3$), quartz (SiO_2), magnetite (Fe_3O_4), and iron-nickel intermetallic (Fe_xNi_y). At 800 ^oC, magnesium silicate hydroxide seems to lose its hydroxide part to become fosterite and enstatite. This result agrees with previous work that reports the decomposition of magnesium silicate at high temperature (Damir *et al.,* 2005, Li *et al.,* 2009).

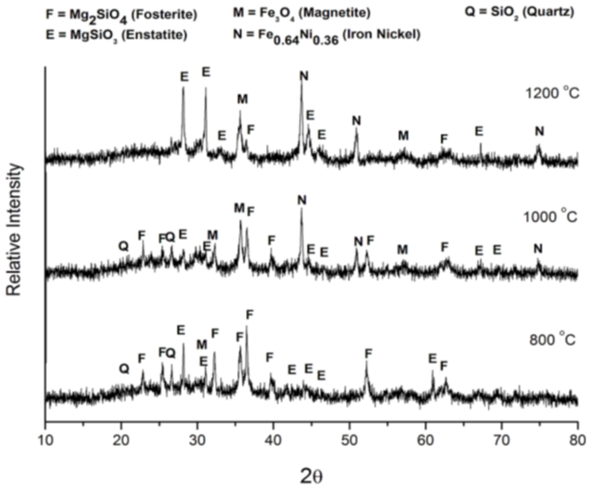

Figure 4. XRD patterns of heated samples.

At 1000 ^oC, intermetallic compound of iron-nickel has been found in the heated samples. The formation of metal becomes more rapid at 1200 ^oC, indicating by high peak of intensity corresponding to iron nickel intermetallic. Meanwhile, fosterite seems to decomposed again to become enstatite. Based on XRD patterns in Figure 4 and other relevant literature (Rhamdhani *et al.,* 2009, Yıldırıma *et al.,* 2012, Babich*et al.,* 2008), the reaction taking place at 800-1200 ^oC can be written as follows.

$2Mg_3Si_2O_5(OH)_4 \rightarrow 3Mg_2SiO_4 + SiO_2 + 2H_2O + O_2$ (1)
$Mg_2SiO_4 + SiO_2 \rightarrow 2MgSiO_3$ (2)
$2FeOOH \rightarrow Fe_2O_3 + H_2O$ (3)
$3Fe_2O_3 + CO \rightarrow 2Fe_3O_4 + CO_2$ (4)
$Fe_3O_4 + CO \rightarrow 3FeO + CO_2$ (5)
$FeO + CO \rightarrow Fe + CO_2$ (6)
$NiO + CO \rightarrow Ni + CO_2$ (7)
$Fe + Ni \rightarrow FeNi$ (8)

The exact temperature of each reaction equation above was not studied in this research, but from XRD analysis (Figure 4) it is obvious that reaction (1) to (4) takes place at temperature 800 ^oC or lower, whereas reaction (6) and (7) takes place at 1000-1200 ^oC.

After being heated, the sample that has been heated at 1200 ^oC was smelted at 1500 ^oC to obtain nickel pig iron. After being smelted, metal phase and slag phase was found to be two different phases that can be distinguished. The separation of these phases can be done mechanically by using a hammer. The % weight of nickel and iron in the metal phase (nickel containing pig iron) are 3.17 % and 86.8 % respectively. The morphology of nickel containing pig iron (NCPI) synthesized from low grade lateritic ore is shown in Figure 5.

There are 3 (three) main phases can be found in nickel contain pig iron: (A) The phase that contains solid solution of nickel in iron, (B) The phase that contains solid solution of chromium and (C) Carbon in iron phase that contains sulfur as inclusion. Due to the low carbon content in phase (A), the composition of this phase looks like nickel – chromium steel. Nickel chromium steel is famously known for its good property at elevated temperature, easy to be work hardened, excellent weldability and high ductility at cryogenic temperature. On the other hand phase (B), due to its high content of carbon and chromium, looks like chromium cast iron. Cast iron is brittle phase but the existence of chromium makes it corrosion resistant. Thus this material is the composite of ductile and brittle phase, which gives quite good mechanical property. Phase (C), since it contains sulfur inclusion, becomes the main disadvantage of this material. Sulfur can cause the rupture of material at elevated temperature. Therefore, it is necessary to do the refining process to remove sulfur from this material down to maximum level of sulfur allowed in steel. The maximum of sulfur content allowed in any steel is 0.004%. The conventional refining process is certainly be able to eliminate sulfur down to 0.04%.

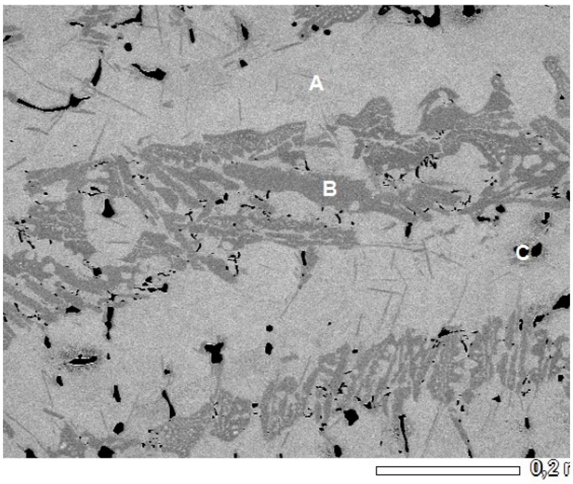

Figure 5. Morphology of Nickel Containing Pig Iron.

CONCLUSION

The formation of nickel containing pig iron (NCPI) from low grade laterite ore has been studied to give certain scientific information about its formation. Low grade nickel layer of lateritic ore consist of certain minerals such as goethite (iron oxy-hydroxide, FeOOH), magnesium silicate hydroxide ($Mg_3Si_2O_5(OH)_4$), quartz (SiO_2), and hematite (Fe_2O_3). Nickel is distributed in goethite and magnesium silicate network. The heating of the low grade layer results in the decomposition or reduction of certain minerals. Magnesium silicate hydroxide decomposes to fosterite (Mg_2SiO_4) and enstatite ($MgSiO_3$), goethite and hematite decomposes to magnetite. Magnetite and nickel oxide was reduced to produce metallic nickel and iron, and then at 1000-1200 ^oC both of these metals forms intermetallic of iron-nickel. The smelting of reduced low grade layer of lateritic ore result in nickel containing pig iron that consist of ductile phases similar to nickel-chromium steel and another brittle phases similar to chromium cast iron.

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