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CHARACTERIZATION OF IRON PRODUCTION FROM MILLS CALE BY CARBOTHERMIC REDUCTION

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Abstract

Mill scale is a byproduct of metal factory. There are many methods to recycle it for various applications. The aim of this research is to recycle mill scale to produce iron by reduction method with graphite as the reductant agent. The reduction process was carried out by milled the mill scale and graphite powder with weight ratio of 4 : 1 by using High Energy Milling with 4, 6, and 12 hours milling time variations. The powder then was characterized by X-Ray Diffraction (XRD), Vibrating Sample Magnetometer (VSM) and SEM-EDS. The XRD test result shows Fe_3C as a main phase. The other detected phases are carbon, magnetite, wustite and Iron as a minor phase. The percentage of detected iron content is increase with milling times at the amount of 6, 10.9, and 13 %. The remanence performance for the 4, 6, and 12 hours of milling time variation is 2.89, 3.39, and 4.98 emu/g, for the coercivity (Hc) is 209.58, 188.47, and 223.65 Oe, and the magnetic saturation number is 22.59, 30.7, 39.15 emu/g. From the Hc value, it is concluded that the powder has superparamagnetic behavior. The SEM-EDS results show that the distribution of Fe is more uniform on the surface of samples with the increasing of milling time.

Keywords: Millscale, by product, reduction, iron.

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1. Introduction

Iron is the tenth element in the universe and it is the fourth in the earth's crust. The ore of iron is extract to iron and it is not found in the free elemental form. The impurities must be removed by chemical reduction to obtain pure elemental iron [1].

Mill scale is a waste-product of metallurgical rolling mill in the steel production. About 13.5 metric tons/year is produced in the world. It is contain wustite (FeO), hematite (α-Fe_2O_3), and magnetite (Fe_3O_4). Mill scale can be as a useful resource when it is recycled. About 90% of millscale waste are recycled in iron and steel industry, whereas small parts can be used in cement plants and petrochemical industry [2-5]. The theme of previously research in an application of recycled millscale is the oxidized mill scale can be used to adsorb textile dye waste [6]. Some of the benefit of recycled mill scale are (i) to reduce the decimation of the earth's natural resources; (ii) to reduce the pollution; and (iii) to save energy indirectly [7].

Many methods to produce the iron from mill scale such as by metallothermic reduction [8-9], powder metallurgy [10-11], sintering, cold bonded agglomeration, injection, direct reduction and smelting reduction in order to utilize this waste material. The choice the recycle technique depends on factors such as environment, energy, policy, industrial statues, recycle strategy of industry, feasibility, technology readiness and economics [11].

In the coal based reduction process the mill scale particle is surrounded by the coal particles and the temperature is ranging from 850 to 950 °C. The carbon in coal is becomes CO and CO_2 gases during the reduction. Both coal and carbon monoxide are adsorb oxygen on surface of mill scale particle. The carbon in coal is react and reduce the iron oxide on mill scale surface. The CO gas is do same effect, but it is diffuse into particle surface and it is reduce it layer by layer. The oxygen bond in iron oxide is removed from the surface of mill scale in the form of carbon dioxide. The iron oxide is reduced from higher order to simpler one and last to metallic iron [12-16].

The new method in this research is the use of graphite as reductant agent that does not need high energy/temperature, only about 650 °C for reduction of the metal oxide [17] and graphite is more economically than the other reductant agent like coal. The reduction process in this research was used the High Energy Milling to produce the uniform powder result than only by heat treatment.

2. Experimental and Procedures

2.1 Materials

The reduction method was used a mill scale as source of raw material from PT. Krakatau Steel, Tbk. The commercial graphite was used as a reductant agent.

2.2 Instrument and equipment

The equipment analysis that used for this experiment are X-ray diffraction (XRD, Rigaku, Smartlab) for phase composition detection, Vibrating Sample Magnetometer (Dexing, VSM-250) for magnetic performance characteristic, and SEM-EDS (Hitachi SU-3500) for microstructure and elemental analysis of the studied sample powder. The other apparatus that used in this work are beaker glass, 400 mesh (<37 µm) sieve, A&D digital balance, and High Energy Milling (HEM) in 3D motion.

2.3 Experiment

Each samples (mill scale and graphite) was milled by disk mill for 10 seconds. Then it's was sieved by 400 mesh (<37 µm) sieve. It's was mixed with 4:1 weight ratio of mill scale to graphite. The mixed powder then was milled by High Energy Milling (HEM) in 3D motion of 100 ml of oval container, with 10:1 of ball to mill scale powder weight ratio, and milling time variations of 4, 6, 12 hours. The powder then was evaluated by X-ray diffraction with 2θ range of 10-90° with 2°/minutes of scan speed, Vibrating Sample Magnetometer, and SEM-EDS analysis.

3. Results and Discussion

The XRD test result of milled powder with 4, 6, and 12 hours of milling times variation is showed in Figure 1. It was also compared with the original peak of mill scale (before). The phase analysis was by Ritveld refinement by the High Scores Plus software analysis. The XRD peaks of the raw material of mill scale sample powder (before) consists of wustite, magnetite, and hematite. Meanwhile, the powder after milled consists of Fe_3C as main phase, then followed by carbon, magnetite, wustite, and iron as a minor phase with notations of triangle, rhombus, star, circle, and square respectively, as shown in Figure 1. The percentage composition of wustite is decrease with increasing of milling times i.e., 6.9; 6.9; and 5.0 %. Meanwhile the percentage composition of iron is increase with increasing milling time, i.e., 6.0; 10.9; and 13.0 %.

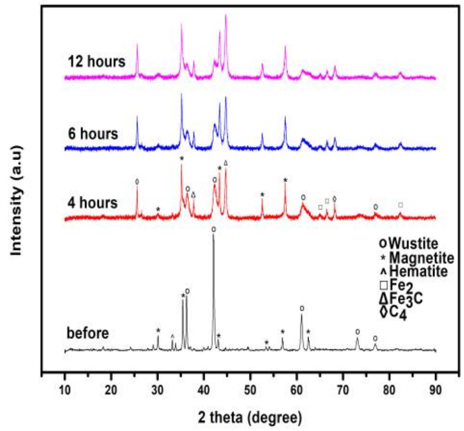

Figure 1. *XRD peaks result of mill scale reduction powder*

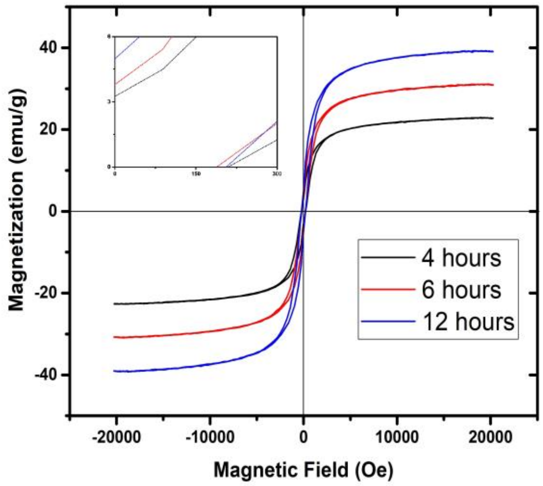

Figure 2. *VSM plot result of reduced mill scale powder*

The magnetic performance characteristic is obtained from the VSM test, as shown in Figure 2. The VSM test was measured from magnetic field from the range of 20 to -20 kOe at room temperature. The remanence number for the 4, 6, and 12 hours of milling time variations are 2.89, 3.39, and 4.98 emu/g, for the coercivity (Hc) are 209.58, 188.47, and 223.65 Oe, respectively, and the magnetic saturation number in this work is 22.59, 30.7, and 39.15 emu/g. Thus, from the VSM plot, it can be concluded that all reduced powder has such a superparamagnetic (narrow hysteresis loop) behavior at room temperature where the Hc value is near zero.

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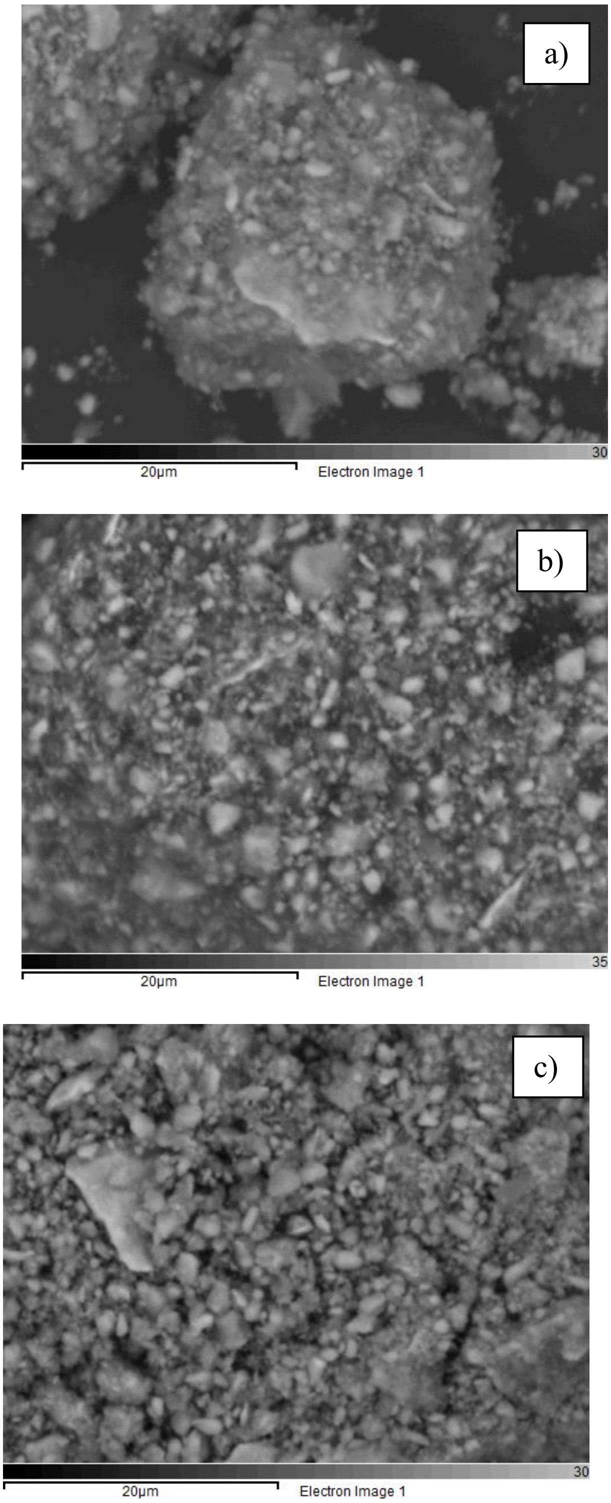

Figure 3. *BSE mode of SEM image of a) 4 hours, b) 6 hours, and c) 12 hours milling time of mill scale powder*

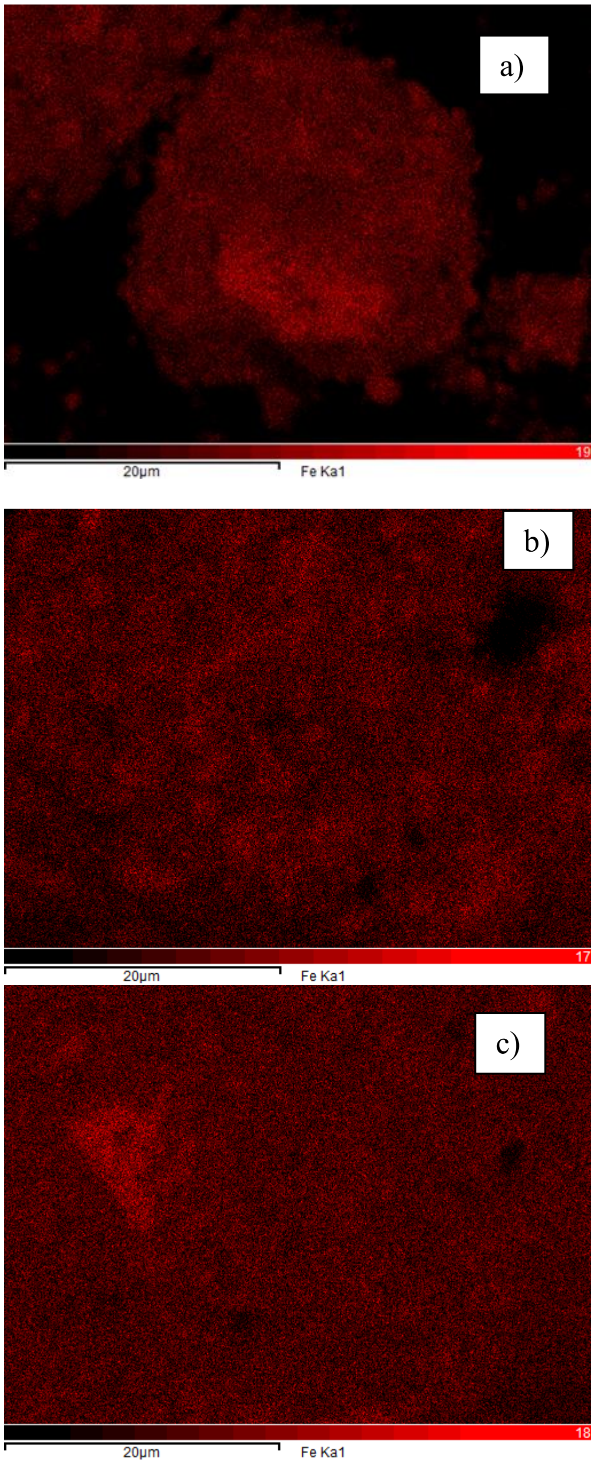

Figure 4. *Fe denoised maps of an RGB component composite image of EDS mapping of a) 4 hours, b) 6 hours, and c) 12 hours of milling time of mill scale powder*

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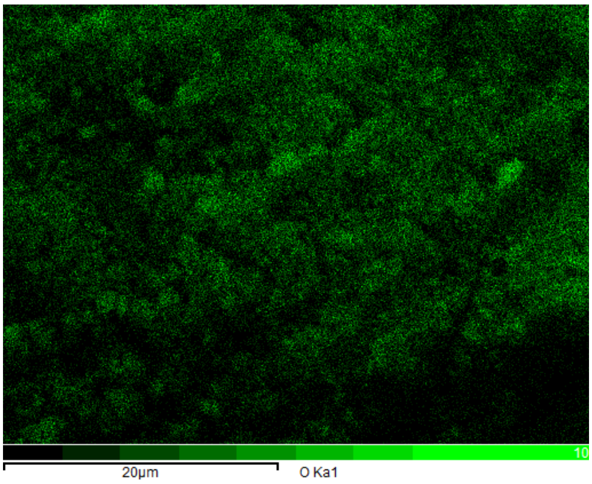

Figure 5. *O denoised maps of an RGB component composite image of EDS mapping 12 hours milling time of mill scale powder*

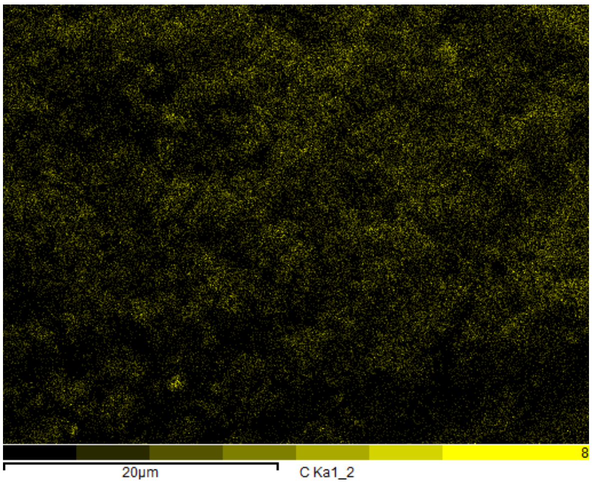

Figure 6. C denoised maps of an RGB component composite image of EDS mapping of 12 hours milling time of mill scale powder

In the SEM-EDS powder test, carbon tape was employed to examine the distribution of Fe on the external surface of milled powder, EDS mapping measurement was carried out on the surface of milled powder on carbon tape. Figure 3 shows the microstructure of the reduced mill scale powder. From Figure 3, it is known that the particle size of the powder is less than 20 microns and the powder has more uniformly grain size with the increase of milling time variations. Figure 4 shows the 2D elemental EDS mapping for Fe that was obtained from the reduced mill scale sample. it is clearly shown that the distribution of Fe is more uniform on the surface with higher of milling time.

4. Conclusions

The iron production is successfully done by carbothermic reduction of mill scale with graphite as the reductant agent and the XRD characterization depicted still showing some impurities on it. However, the iron composition increases with the increasing of milling time of reduction process.

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