Preliminary Study: Local Iron Sand Characterization of Titanomagnetite Type

Y E Gunanto[1], Maya P Izaak[1], L Cahyadi[1], E Jobiliong[1], and W A Adi[2]

[1]Department of Physics Education, Faculty of Education, University of Pelita Harapan, Karawaci, Tangerang 15811, Indonesia
[2]Center for Science and Technology of Advanced Materials, BATAN, Tangerang Selatan 15314, Indonesia

E-mail: yohanes.gunanto@uph.edu

Abstract. In this study, we have characterized the natural iron sand type of titanomagnetite taken from the coast of West Lampung. Prior to characterization, the natural iron sand type of titanomagnetite was milling with high energy milling (HEM) for 10 h. Scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) were used to characterize surface morphology and analysis of constituent elements of iron sands respectively. X-ray diffraction (XRD) was used for phase analysis and crystal structure. The morphology of the surface of the natural iron sand type of titanomagnetite had a variety of shapes and sizes of particles and contrasting colors. This is consistent with the result of characterization using energy dispersive spectroscopy where the element of iron sand consists of Fe (66.57 %), Mg (1.37 %), Si (0.57 %), Al (1.66 %), Ti (8.03 %), La (1.17 %) and O (20.64 %). These constituent elements did not stand on their own but bond to each other forming certain phases. Analysis by X-ray diffraction is generated that the phases formed on the iron sand of types titanomagnetite, consist of ilmenite ($FeTiO_3$), hematite (α-Fe_2O_3) and titanomagnetite ($Fe_{3-x}(MT,Ti)_xO_4$) phases with the content respective of 9.63 %, 19.09 % and 71.28 %, where MT = metal transition.

Keywords. Hematite, iron sand, ilmenite, and titanomagnetite.

1. Introduction

In this competitive globalization era, Indonesia has been intensively developing infrastructure in order to improve the economic level and to be a self-reliant country that does not rely on imports. Thus, supporting materials such as steel, cement and others are needed to develop the in infrastructure. This has an impact on the increasing need for basic materials in the form of iron sand as the base material for making steel or cement. The year 2025 is estimated to meet the steel industry required about 250 million tons of iron ore and 110 million tons of iron sand [1].

In Indonesia, natural iron sand resources totaling more than 2 billion tons are spread along the southern coast of Java, Sumatra, West Nusa Tenggara, and so on with the main content of iron oxide, titania, silica, and alumina [2]. Indonesia has great iron mineral resources, comprising primary iron ore (17 %), iron sand (8 %) and lateritic iron ore (75 %) [3]. Materials contained in the iron sand are magnetic and non-magnetic. Magnetic materials in the iron sand are drawn strongly by magnets, and some are weakly pulled. Strongly withdrawn consisted of titanomagnetite (Ti ~ 10%) and hematite (Fe:Ti ~ 1:1) content, while weakly pulled, the Ti content reached nearly 50 %.

In this study, natural iron sand of titanomagnetite type is produced from natural iron sand by using a separator of a weak permanent magnet around 1000 Oe. This stage is an important parameter that not conducted by previous researchers due to they still used the magnetic separator with magnet field high so that not obtained the titanomagnetite type perfectly. Thus only the iron sand of titanomagnetite type will attach to the permanent magnet. Some characterization will be done such as scanning electron microscope (SEM) is used to analyze surface morphology. To analyze the elements contained in the iron sand of titanomagnetite type used energy dispersive spectroscopy (EDS) and to analyze the phases use X-ray diffractometer (XRD).

2. Materials and methods

Iron sand was retrieved from Lampung, Indonesia. 100 g of iron sand was prepared and separated using magnetic separator with a low magnetic field about 1000 Gs to eliminate major impurity, so that obtained iron sand with Fe rich or called the type of titanomagnetite. The iron sand powders of titanomagnetite type were milled by using high-energy milling (HEM) type PW1000 in for 10 h at room temperature to obtain fine particles to facilitate the characterization and further processing. The analysis of phase qualitative and quantitative was carried out using X-ray diffractometer equipped (Pan Analytical) with a tube provided with the copper anode (CuKα). The Rietveld analysis was done by applying for GSAS program with the pseudo-Voigt function used to approximate of diffraction line profiles. The elemental composition analysis and surface morphology were observed by using dispersive spectroscopy (EDS) and scanning electron microscope (SEM), respectively

3. Results and discussion

Figure 1 shows the observed microstructure on iron sands of titanomagnetite type using scanning electron microscope. The surface morphological observations showed that the sample had a variety of shapes, particle size, and color contrast. This indicated that the suspected sample had a very diverse phase. The average size of the grains is between (5 to 10) μm. These results were much smaller than the results obtained Rusianto *et al.*, which is about 212 μm [4]. However, a photograph of this surface morphology cannot determine the phases contained in this titanomagnetite sandstone iron sample. Thus further analysis is needed to show that this type of titanomagnetite iron sandstone has a diverse phase, using x-ray diffraction analysis. To analyze the phases with x-ray diffraction, it is necessary to support the elementary data of this titanomagnetite iron sandstone by using energy dispersive spectroscopy (EDS).

Figure 2 shows the results of elementary analysis using energy dispersive spectroscopy on titanomagnetite iron sand rocks. The result of energy spectrum showed that the most dominant elements were oxygen (O), titanium (Ti) and iron (Fe) which were consecutively at 0.525 keV, 4.508 keV and 6.398 keV with wavelength Kα. This meant that these rocks contain iron-rich elements (Fe), but there were still some other elements contained in it in a relatively small amount. The elements contained in this titanomagnetite iron sandstone rock is presented in detail in Table 1.

Table 1 shows that the total iron content of iron sandstone of this type of titanomagnetite is 66.57 %. These results indicate that the iron content in the sand sandstone type titanomagnetite Lampung is more potential and economical to be further processed. The element content in this titanomagnetite iron sandstone is very complex. Some of the elements contained in the iron sand of titanomagnetite type did not stand alone most likely instead they bind to each other to form certain minerals according to the largest content of this iron sand of titanomagnetite type. Thus a structural analysis is needed to identify the phases present in the iron sand of titanomagnetite type. Mufti *et al.* obtain Fe content in the natural iron sand about 41.2 % before being extracted with a magnet [5].

IOP Conf. Series: Materials Science and Engineering **367** (2018) 012059 doi:10.1088/1757-899X/367/1/012059

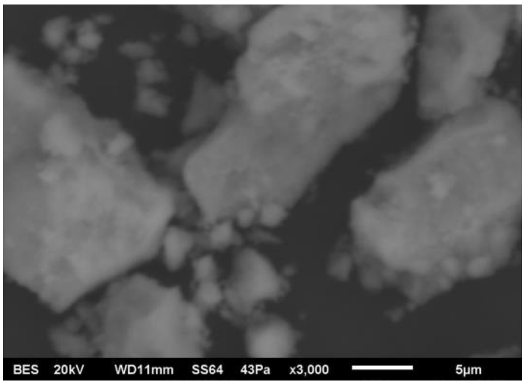

Figure 1. The surface morphology of iron sandstone samples of the titanomagnetite type

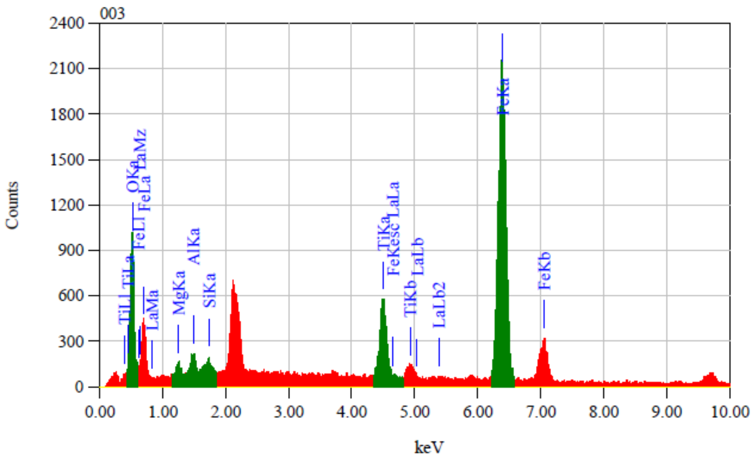

Figure 2. elementary analysis using energy dispersive spectroscopy

Table 1. The result of elementary analysis using energy dispersive spectroscopy

No.	Element	Content (% weight)
1.	Iron (Fe)	66.57 ± 0.35
2.	Magnesium (Mg)	1.37 ± 0.22
3.	Silicon (Si)	0.57 ± 0.16
4.	Aluminum (Al)	1.66 ± 0.18
5.	Titanium (Ti)	8.03 ± 0.22
6.	Lanthanum (La)	1.17 ± 0.61
7.	Oxygen (O)	20.64 ± 0.21
8.	Calcium (Ca)	ND
9.	Chromium (Cr)	ND
10.	Vanadium (V)	ND

*) ND: not detected and limit detection around 1 wt.%

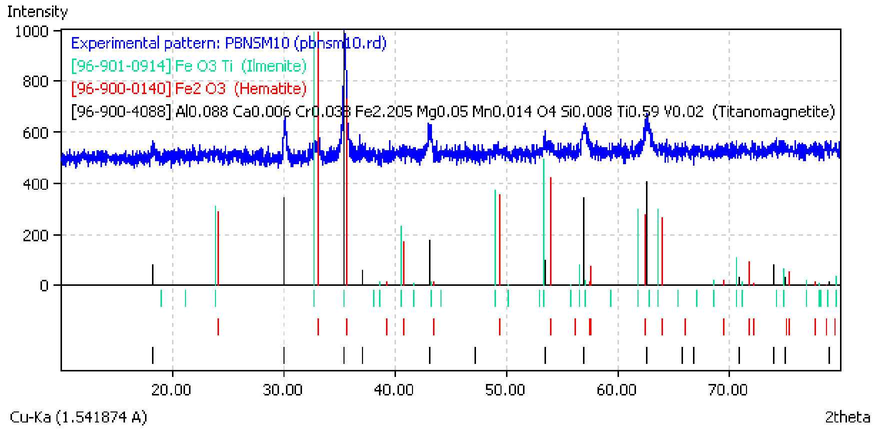

Figure 3. X-ray diffraction pattern on the iron sand of titanomagnetite type samples

Figure 3 shows the results of the measurement and identification of x-ray diffraction patterns of titanomagnetite iron sandstone samples. This revealed that there are formed Bragg diffraction peaks which were suspected to be multi-phase. The phase identification refers to the results of the Yamanaka study, ICDD 96-901-0914 [6], Blake, 96-900-0140 [7], Stout, ICDD 96-900-4088 [8], which are the ilmenite, hematite, and titanomagnetite. Thus, based on the results of the analysis of the X-ray diffraction pattern shows that the amount of mineral phase content contained in this iron sand of titanomagnetite type consist of ilmenite ($FeTiO_3$), hematite (α-Fe_2O_3) and titanomagnetite ($Fe_{3-x}(MT,Ti)_xO_4$) phases with the content respective of 9.63 %, 19.09 % and 71.28 %, where MT (metal transition) as shown in Table 2.

Based on the results of x-ray diffraction analysis in Table 2, then the next steps can be done for processing of iron sandstone of this type of titanomagnetite. For further research, it will focus on a local natural resource that can be used as a permanent magnetic material ingredient. And based on the initial identification of mineral content in this titanomagnetite iron sandstone, the most efficient use is processing for purification into the Fe_3O_4 raw material to reach more than 99 % Fe_3O_4. The Fe_3O_4 material can be used as a permanent ferrite based magnet material.

In general, mineral analysis results can be classified into two parts, namely magnetic materials and non-magnetic materials. Minerals classified as magnetic materials are maghemite minerals, titanomagnetite iron sands, and goethite. Meanwhile, the minerals belonging to non-magnetic materials are minerals staurolite, quartz, and lime. However, to get the raw material of Fe_3O_4 up to reach more than 99 % level, the extraction process of iron sandstone of this type of titanomagnetite should be made so that the phases other than iron can be separated and expected to be obtained by a material with relatively high iron content. The results obtained by Jalil *et al.*, Fe_3O_4 content in iron sand taken from the coast of Syiah Kuala about 85 % [9].

Table 2. The mass fraction of the mineral phases contained in the titanomagnetite iron sandstone rock

No.	Mineral	Empirical Formula	Mass Fraction (%)
1.	Ilmenite	$FeTiO_3$	9.63 ± 0.004
2.	Hematite	α-Fe_2O_3	19.09 ± 0.005
3.	Titanomagnetite	$Fe_{3-x}(MT,Ti)_xO_4$ (MT: Transition Metal)	71.28 ± 0.006

*) MT : Mn, Cr, V, Ni, etc.

4. Conclusions

Iron sand of titanomagnetite type has been obtained from natural iron sand using a weak magnetic separator of 1000 Oe. To increase Fe content, iron sand of titanomagnetite type was milled for 10 h using high energy milling (HEM). The result of surface morphology by using SEM found that the average size of the grains is about (5 to 10) μm. The elements contained in natural iron sand were obtained by energy dispersive spectroscopy (EDS) analysis, consisting of: Fe (66.57 %), Mg (1.37 %), Si (0.57 %), Al (1.66 %), Ti (8.03 %), La (1.17 %) and O (20.64 %). The results of the analysis using X-ray diffraction (XRD) have been obtained that the phase contained in the natural iron sand of the type of titanomagnetite, namely ilmenite ($FeTiO_3$), hematite (α-Fe_2O_3) and titanomagnetite ($Fe_{3-x}(MT,Ti)_xO_4$) phases with the content respective of 9.63 %, 19.09 % and 71.28 %, where MT (metal transition).

Acknowledgements
This work was supported by Higher Education Research Grants 2017 No. DIPA: SP DIPA-042.06.1.401516/2017, Development of Iron Oxide Nanoparticles from Iron Sand Processing in Support of National Industry

References
[1] Performance Report of the Ministry of Industry Year 2015, 2016.
[2] Ministry of Industry Republic of Indonesia, Profile of Steel Industry, 2014
[3] Rochani S et al 2008 The current status of iron minerals in Indonesia *Indonesian Mining Journal* **11** 1 – 17
[4] Rusianto T *et al* 2012 The potential of iron sand from the coast south of Bantul Yogyakarta as raw ceramic magnet materials *Journal of Technology* **5** 62 – 69
[5] Mufti, N., Munfarriha, U., Fuad, A., & Diantoro, M. (2016, February). Synthesis and photocatalytic properties of Fe_3O_4@TiO_2 core-shell for degradation of Rhodamine B. In AIP Conference Proceedings (Vol. 1712, No. 1, p. 050009). AIP Publishing.
[6] Yamanaka T, Komatsu Y, and Nomori H 2007 Electron density distribution of $FeTiO_3$ ilmenite under high pressure analyzed by MEM using single crystal diffraction intensities Locality: synthetic Sample: P = 3.6 Gpa *Physics and Chemistry of Minerals* **34** 307 – 318
[7] Blake R L *et al* 1966 Refinement of the hematite structure *American Mineralogist* **51** 123 – 129
[8] Stout M Z and Bayliss P 1975 Crystal structure of a natural titanomagnetite *The Canadian Mineralogist* **13** 86 – 88
[9] Jalil Z *et al* 2017 Magnetic behavior of natural magnetite (Fe_3O_4) extracted from beach sand obtained by mechanical alloying method *AIP Conf. Proc.* 1862 **030023** 1 – 4