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The Effect of Nd Content Variation on Crystal Structure and Microstructure to Improve Magnetic Properties Performance

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Abstract. $Nd_2Fe_{14}B$ magnet powder has been fabricated by mixing Fe, B, and Nd powder based on their stoichiometric proportion at 650°C for 2 hours. Heat treatment series were applied at 720°C for one hour and continued with annealing at 100°C for 4 hours on different Nd stoichiometric variations; 26.6 %wt, 32.6 %wt, and 40.6 %wt. The results obtained from the X-ray Diffraction (XRD) phase analysis of Nd-rich Nd-Fe-B alloy suggested that the alloy has monophasic composition with hard magnetic $Nd_2Fe_{14}B$ phase as a composition. The mean of the crystal grain size was determined using XRD analysis, while the microstructure and composition of the alloys were analyzed using the Scanning Electron Microscope – Energy Dispersive X-ray (SEM-EDX). The magnetic properties characterization were determined using the Vibrating Sample Magnetometer (VSM), which indicates that the sample with 40.6 %wt Nd was able to achieve the highest remanence of 446.50 G and BHmax of 17.83 kGOe. Despite the high remanence result, the coercivity and BHmax value of the third sample was still lower than the commercial's but has adequate potential value.
Keywords : Nd-Fe-B alloys; Nd-rich; phase composition; crystallite size

1. Introduction

Neodymium Iron Boron (NdFeB) is well known as rare earth alloy magnets made from neodymium, iron and boron elements and formed the $Nd_2Fe_{14}B$ tetragonal crystalline structure which has high coercivity and large energy product [1]. The permanent magnet Nd-Fe-B has become an important part in the everyday life as its being used in electric motors, generators, and electronics equipment that has been developed by Japan Company since 1982. The Industries demand of permanent magnets in Indonesia is very high while 100% permanent magnet products in Indonesia are imported with 80% of them came from China since they produced low cost global [2].

Therefore, research in permanent magnet material field to increase magnetic performance such as magnetic induction remanence (Br), coercivity (Hc), and maximum energy product (BHmax) is done in order to produce local permanent magnet product [3,4,5]. The stoichiometric alloys are investigated by some intergranular exchange between the grains of the same hard magnetic phase. One of these studies are the enhancement of non-ferromagnetic phase densification through enrichment Nd (Nd-rich) in order to distinct the separation between $Nd_2Fe_{14}B$ magnetic grains. Phase composition and grain size which has direct influence on magnetic properties were observed on different Nd contents to investigate the change in structure while fabricating the $Nd_2Fe_{14}B$ alloy powder for the permanent magnet.

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2. Experimental

The $Nd_2Fe_{14}B$ powder were prepared in series of heat treatment at 300ºC (1 hour), 650ºC (2 hours), and 720 ºC (1 hour), before being dropped to100 ºC. Powder Nd, Fe, and B with a purity of 99.99% were used as starting materials, and the composition of each powder was calculated using the stoichiometric ratio with 26.6 %wt Nd; 72.32%mol Fe; and 1%mol B (sample 1). The sample 2 and sample 3 have Nd-rich with 32.6 %wt and 40.6 %wt respectively. Phase composition was determined by using X-ray diffraction (XRD) whereas mean crystal grain size was calculated from the XRD data. The microstructure and composition analysis were characterized by using SEM-EDX and the magnetic properties were determined by using VSM.

3. Results and Discussion

The XRD patterns result of the $Nd_2Fe_{14}B$ powder (Sample 1), and the Nd-rich $Nd_2Fe_{14}B$ alloys powder (sample 2 and sample 3) are presented on figure 1.

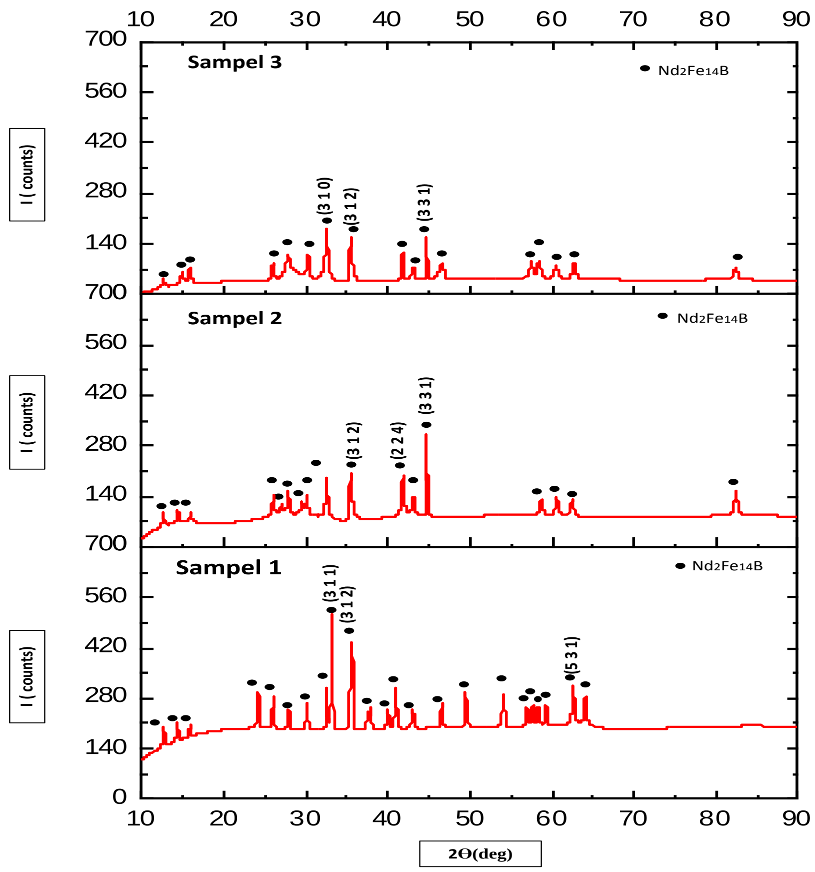

Figure 1. XRD patterns of the $Nd_2Fe_{14}B$ powder (sample 1) and $Nd_2Fe_{14}B$ powder Nd-rich (sample 2 and 3).

$Nd_2Fe_{14}B$ crystal structure analysis using XRD aims to observe the phases that are formed on the sample powder. The peaks matched with NdFeB cards numbered 04-004-9492 were marked with black round which show that the third sample in the figure 1 above are $Nd_2Fe_{14}B$ crystalline alloy powder. At the same time, the phase composition and crystalline structure produced shows that the alloy has almost monophasic composition with $Nd_2Fe_{14}B$ dominating the magnetic phase both in stoichiometric and Nd-rich sample. The mean crystallite grain size was calculated using the Scherrer formula from the highest peak of the XRD patterns (the mean grain sizes were calculated only for $Nd_2Fe_{14}B$ phase).

Table 1. "Crystallite size" of the $Nd_2Fe_{14}B$ powder (sample 1) and $Nd_2Fe_{14}B$ powder Nd-rich (sample 2 and 3)

Sample	"Crystallite Size" (µm)
1	0,272
2	0,310
3	0,148

The crystallite sizing results in sample 1, sample 2, sample 3 are respectively around to 0.272 µm, 0.310 µm and 0.148 µm. In sample 2, the mass of Nd element is raised by 23%, crystal planes diffracted are reduced and the crystalline grain size increased by 1.5 times. In the third sample, the mass of Nd element is raised by 53%, crystal planes diffracted are the same as in sample 2 but the crystalline grain size reduced by half.

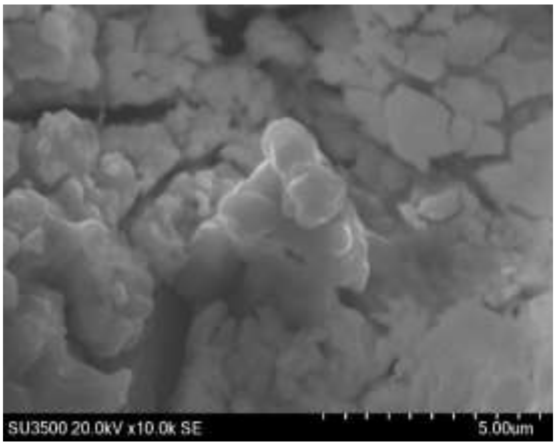

Figure 2. SEM micrographs of $Nd_2Fe_{14}B$ powder Nd-rich (sample 2).

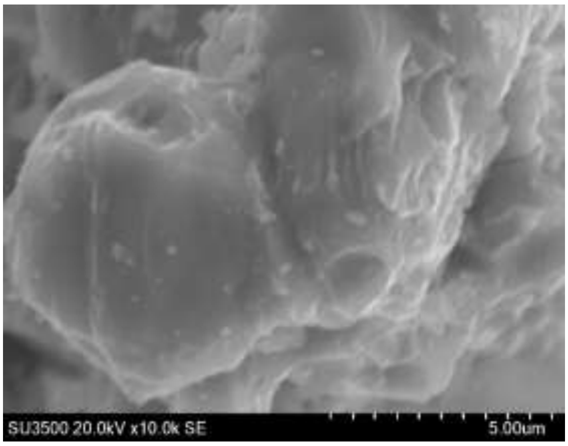

Figure 3. SEM micrographs of $Nd_2Fe_{14}B$ powder (sample 1).

Figure 2 shows white color which show Nd element on the surface of the $Nd_2Fe_{14}B$ alloy grains. The whiter the color show greater concentration of Nd (see Table 2).

Table 2. The Nd content by EDX Result

% atom Element	Sample 1	Sample 2	Sample 3
Nd	0.41	24.44	34.40

Meanwhile, figure 3 shows the $Nd_2Fe_{14}B$ crystalline surface morphology by SEM. Dark gray color indicates the $Nd_2Fe_{14}B$ tetragonal structure formed in stoichiometric state. The magnetic property of the powder was tested by using vibrating sample magnetometer (VSM) and is presented on figure 4. The result shows that the NdFeB magnets is categorized as hard magnet material powder by having 301 G in sample 1 and 413 G in sample 2 and 447 G in sample 3 of remanence (Br) value. Saturated remanence is enough well around 4 kG as it can be seen in Table 3. The enhancement of Nd content such as coercivity (Hc) and BHmax value is expected to increase after the compaction treatment.

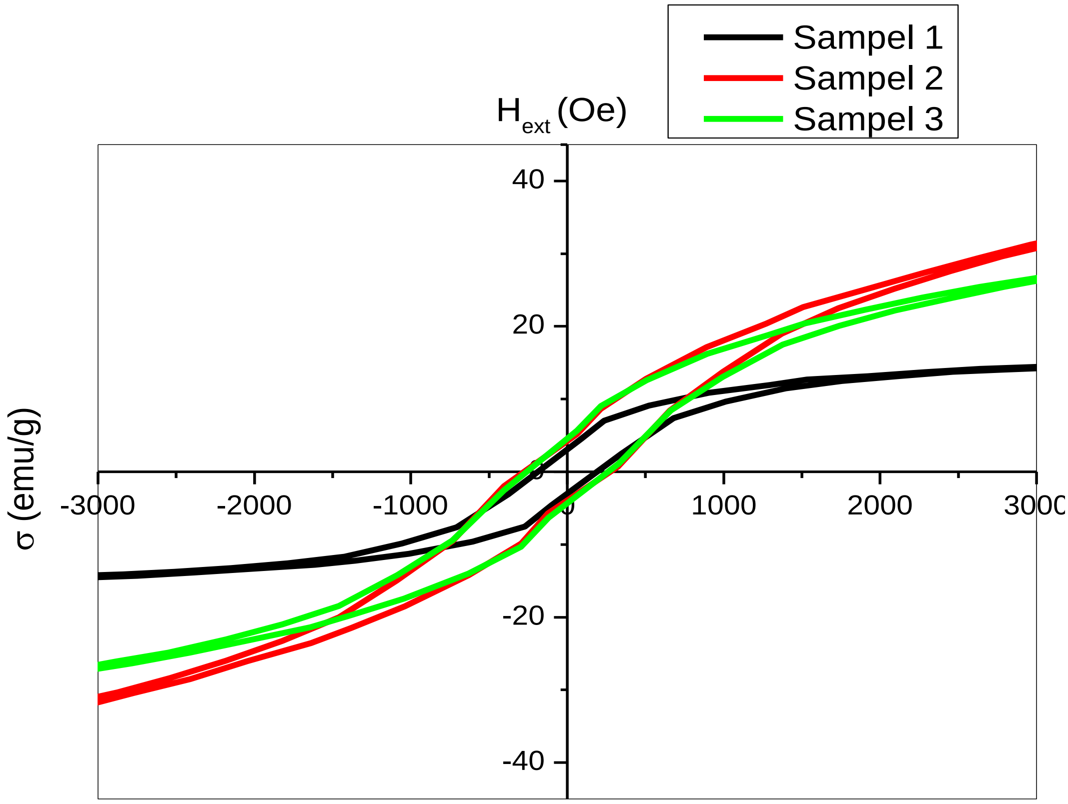

Figure 4. Hysteresis loops of of the $Nd_2Fe_{14}B$ powder (sample 1 black) and $Nd_2Fe_{14}B$ powder Nd-rich (sample 2 and 3).

Table 3. Magnetic Properties of samples 1, 2 and 3 from hysteresis loops

Sampel	Br (G)	Bs (G)	Hc (Oe)	BH_{max} (KGOe)
Sampel 1	301,24	1584,22	188,89	8,56
Sampel 2	413,36	4431,92	274,60	16,59
Sampel 3	446,50	3496,01	261,10	17,83

The heat treatment mechanism has direct influence on the formation of $Nd_2Fe_{14}B$ alloys crystallized grains [5, 6]. The remanence-enhancement effect is not yet increase because the ideal conditions to achieve effective ferromagnetic exchange between the grains of magnetic phases are not fulfilled and the grain size must be below 100 nm [7,8]. Although for sample 3 appear that crystallite size become smaller but the optimal magnetic state as the distinct separation between $Nd_2Fe_{14}B$ magnetic grains and the identification phase composition must be detailed again.

4. Conclusion

$Nd_2Fe_{14}B$ magnetic alloys powder has been fabricated by mixing the Fe, B, and Nd powder with heat treatment series on different Nd stoichiometric variations; 26.6 %wt, 32.6 %wt, and 40.6 %wt. The results obtained from the XRD phase analysis of Nd-rich Nd-Fe-B alloy suggested that the alloy has monophasic composition with hard magnetic $Nd_2Fe_{14}B$ phase as a composition. The mean crystal grain size was determined by using XRD analysis tend to approach nanocrystalline alloy powder, while the microstructure and composition of the alloys were analyzed using the SEM-EDX. The magnetic properties was determined using the Vibrating Sample Magnetometer (VSM) which indicates that the sample with 40.6 %wt Nd was able to achieve the optimal magnetic state with highest remanence of 446.50 G and BHmax of 17.83 kGOe. Despite the high remanence result, the coercivity and BHmax value of the third sample was still lower than the commercial's but has adequate potential value.

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