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Nd-rich Effects on Structural and MagneticProperties of $Nd_2Fe_{14}B$ Magnets

Syahrul Humaidi[1], Achiruddin[1], Kurnia Sembiring[1], Hanifah[1], Nenen Rusnaeni[2], Muljadi[2]

[1]Physics Department, FMIPA, University of Sumatera Utara, Medan, Indonesia

[2]Research Center for Physics, Indonesian Institute of Sciences, Tangerang Selatan, Indonesia

Email address:

syahrul1@usu.ac.id (S. Humaidi)

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Abstract: Hard magnetic material $Nd_2Fe_{14}B$ was synthesized via powder metallurgy technique. The magnetic powder $Nd_2Fe_{14}B$ was produced by mixing Neodymium powder (Nd), Iron powder (Fe), and Boron powder (B) in a vacuum and then followed by firing process at 720ºC. The $Nd_2Fe_{14}B$ powder produced was grained with addition of 0.3g and 0.7g Neodymium respectively. The powder was sintered at 720ºC. The characterization of magnetic properties by using VSM and phase analyse using XRD. SC-70 was used as detector with 40 kV and 30 mA (CuKα/1.5418A^o). The scan range was 10^o-90^o with a speed of 2 deg/min. The crystal structure of the samples were determined based on data obtained from XRD pattern. The tetragonal crystal structure of $Nd_2Fe_{14}B$ was confirmed with lattice parameters 'a' = 'b' = 8.707Å and 'c' = 12.203Å. The result showed that remanence magnetization (Mr). The maximum energy product $(BH)_{max}$ of $Nd_2Fe_{14}B$ was increased from 8,56 KGOe to 16,59 KGOe with adddition of 0,3g Nd. The BH_{max} reached 17,83KGOe when 0,7g Nd-rich was introduced.

Keywords: Magnetic Properties, Nd-rich, $Nd_2Fe_{14}B$ Magnets, Powder Metallurgy Technique

1. Introduction

Nd–Fe–B sintered magnets have been most widely applied in the electricmotors or hybrid vehicles due to the highest energy product, $(BH)_{max}$ [1]. Neodymium Iron Boron (NdFeB) magnet is a earth magnet consist of the Neodymium, Iron and Boron elements. The structure of the magnet is tetragonal crystal [18]. The classical $Nd_2Fe_{14}B$ sintered permanent magnets based rare-earth hard magnetic materials shows a high coercivity and large energy product. The researchs on the magnetic material tend to increase year by year around the world. Many experiments have been done to enhance the magnetic properties as well as the qualities of the stoichio-metric material addition. One of them is the substitution neodymium atoms to the basic structure [5, 6, 7, 8, 9]. The methods of the preparation also have been developed time to time. The newest nanocrystalline magnetic materials based on Nd-Fe-B alloys prepared by the rapid quenching of the liquid, known as exchange-coupled nanocrystalline composite hard magnetic materials [2, 3, 4, 5, 15].

One of the main magnetic property that must be considered is the coercivity. The coercivity mechanism of Nd-Fe-B sinteredmagnets is considered to be the nucleation of reversaldomain and interfacial microstructure between $Nd_2Fe_{14}B$. Thus, the Nd-rich phases influences the coercivity. In order to obtain theguiding principles for enhancement of coercivity, manyresearchers have observed the interfacial microstructure. Vialet al. [6] reported that the optimum annealing after sinteringdeveloped the smooth interface between $Nd_2Fe_{14}B$ and Nd-richphases. Sagawa et al. [7] reported the Nd-rich phasehave an fcc structureand Ramesh et al. [8] showed the Nd-richphase contains oxygen about 20%–50% [8]. According to Shinba et al. [9], the Nd-rich phase contains oxygen and the microstructure changes fromcrystallite to amorphous with decreasing the thickness of Nd-rich phase. Mo et al. [10] reported that the crystallinestructure of Nd-rich phase changes by amount of oxygen content.

The Nd rich phase is mainly composed of neodymium included iron and oxygen. Typically the oxygen is introduced during the processing stages. Some oxygen in the Nd-rich phase of Nd-Fe-B- type sintered magnets has been shown the

improvement of coercivity and corrosion resistance [16]. The ratio of Nd-rich phase increases the coercivity increases as the Nd rich phase is reduced the remanence increases [11]. In this paper, the Nd-rich effects on the structural and magnetic properties on $Nd_2Fe_{14}B$ magnets is reported. The addition of Nd-rich to $Nd_2Fe_{14}B$ magnet since Nd plays an important role in the densification process of NdFeB magnet. In other words, by this treatment (Nd-rich) a better characteristic of magnetic material can be produced. As such, we can modify the magnet in the future work.

2. Experiment

Hard magnetic material "$Nd_2Fe_{14}B$" was synthesized via powder metallurgy technique. The process in manufacturing magnetic powder $Nd_2Fe_{14}B$ conducted by mixing Neodymium powder (Nd), Iron powder (Fe), and Boron powder (B) with high purity level >99.99% (2N) in a vacuum and then followed by sinteringprocess at 720ºC in vacum furnace. Digital balance was used to measure the precise masses of these elements according to the calculated values of %-mole. The $Nd_2Fe_{14}B$ powder produced was grained with addition of Nd 0.3 gram and 0.7 gram respectively. The powder was then sintered at 720ºC in vacum furnace. The characterization of magnetic properties by using VSM, microstructure analysis using SEM-EDX and phase analysis using XRD. The crystal structure of the magnets was determined by X-ray diffraction (XRD) with Cu-kα radiation (λ= 1.5405 Å) at room temperature.

3. Results and Discussion

3.1. X-Ray Diffractometery

X-ray diffraction (XRD) analysis was used for determination of crystal structure of the samples. XRD patterns (as presented in Figure 1) were plotted by using data obtained from X-ray diffractometer and matched with ICDD (International Center for Difraction Data) $Nd_2Fe_{14}B$ card (card number 04-004-9492). For each peak, interplaner spacing '*d*' was calculated using Bragg's law ($n\lambda = 2d\ sin\theta$) and was compared with interplanar spacing '*d*' in ICDD database. The calculations of '*d*' for particular peaks, corresponding to the reflections from the (310), (311), (312), (224), (331) and (531) planes for samples. These results verify the crystal structure of Nd_2Fe_{14} Bmaterialsis a tetragonal system.

The lattice parameters for all samples were determined using XRD Software (Cell). The *hkl* values and corresponding to 2θ values of all prominent peaks were used in the Software program and lattice parameters can be listed. The value of the lattice parameter as well as the crystalline size can be presented in Table 1. These values of lattice parameters are in good agreement with the standard values for Nd2Fe14B magnets.

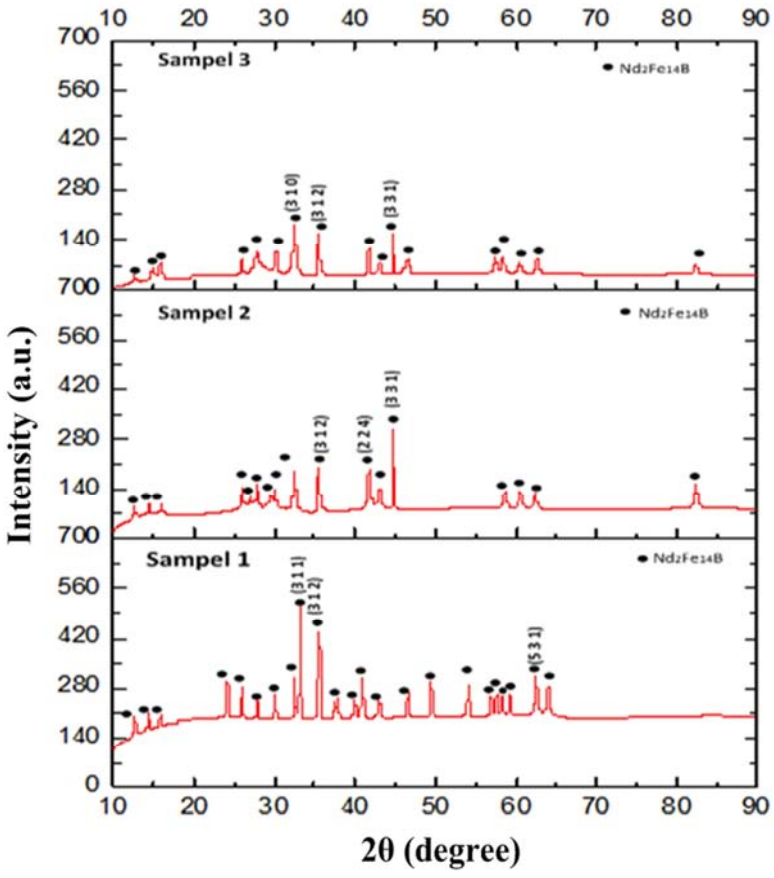

Figure 1. *XRD patterns of $Nd_2Fe_{14}B$ (Sample 1), $Nd_2Fe_{14}B$ + 0.3 gram Neodymium (Sample 2), and $Nd_2Fe_{14}B$ + 0.7 gram Neodymium (Sample 3).*

Figure 1 shows the XRD patterns of the magnet. From Figure 1 it can be seen that the maximum peaks is in the range of 2θ= 30°- 40°. These results indicate that the samples are dominantly by $Nd_2Fe_{14}B$ phase. The samples also show a sharp peak that indicate our samples are in high crystallinity degree. The crystallite sizes of samples were calculated from most intense peaks in the XRD patterns using Scherrer formula:

$$\text{Crystallite Size } (t) = \frac{k\lambda}{BCos\theta_B} \quad (1)$$

Here, '*k*' is Scherrer constant and its value is '0.94' for our material, '*B*' is full width at half maximum (FWHM) of respective peaks, '*θB*' is Bragg's angle and '*λ*' is wavelength of Cu-K-αradiation (1.5405 Å) used during the XRD analysis of the samples [12, 13]. The calculated values of crystallite size for samples are given in Table 1. From Table 1 it can be seen that the crystallite size of Nd2Fe14B1 magnet decreased from 0.310 µm to 0.148 µm, with increasing ofNd-rich from 0.3 gram to 0.7 gram.

Table 1. *Structural and Crystallite size of $Nd_2Fe_{14}B$.*

Sample	Lattice Parameter		ρ_x (gcm^{-3})	Unit Cell Volume (nm)	2θ (deg)	FWHM (deg)	Crystallite Size (µm)
	a = b (Å)	C (Å)					
Sample 1	8.707	12.203	7.763	0.925	33,17	0,1998	0,272
Sample 2	8.707	12.203	7.763	0.925	44,78	0,1814	0,310
Sample 3	8.707	12.203	7.763	0.925	32,49	0,3649	0,148

Table 1 presents the structural and crystallite size of the magnets. As it can be seen, the value of lattice parameter 'a' is 8.707 A^o for all samples. This result is in a good agreement with previous finding [17]. The X-ray Density (ρ*x*) of the material was determined using the following relation [13]:

$$\rho_x = \frac{nM}{N_A V} \quad (2)$$

In this relation, 'M' is molar weight of one formula unit, 'NA' is Avogadro number, '*n*' is number of formula unit (in our case; $n = 4$) and V is the Volume of Unit cell. These results imply X-ray density (ρx) 7.76 g/cm^3 which agrees well with the theoretically calculated value 7.763 g/cm^3 [14]. The value of crystallite size is optimum (0.310 µm)when we used 0.3 gr Nd. When we increased the amount of Nb-rich up to 0.7gr, the value of crystallite size decreased to 0.148 µm.

3.2. Scanning Electron Microscopy(SEM)

Scanning Electron Microscopyis a suitable technique to study the microstructure of thematerials which tells us about the the surface structure of the sample. To observe the grains clearly in the Scanning Electron Microscopy, the samples were coathing with Au. These results of Scanning Electron Microscopy, as shown in Figurte 2. The micro structural observations showed that the Nd-rich were uniformly distributed on $Nd_2Fe_{14}B$ magnets. These types of grain distributions illustrate the good mechanical properties of the material.

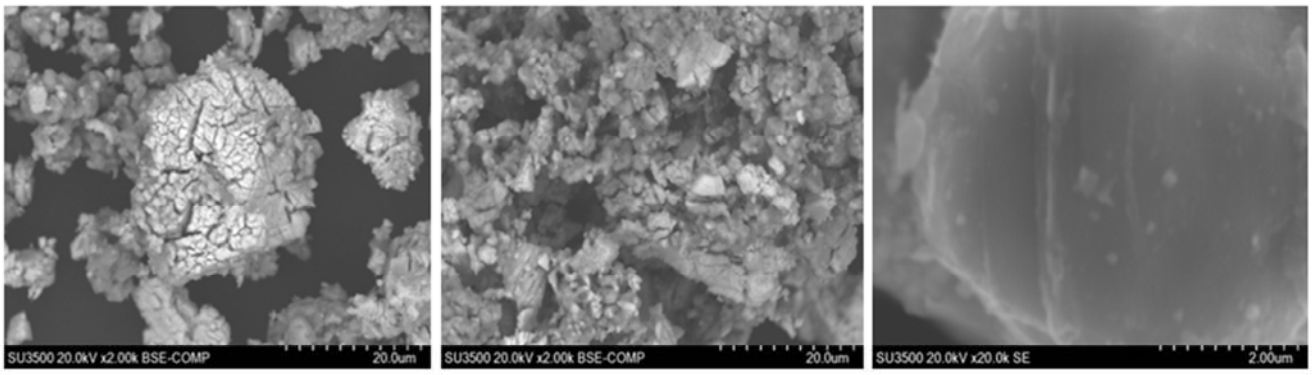

Figure 2. *Scanning Electron Microscopy of a. Nd2Fe14B b. Nd2Fe14B + 0.3 g Neodymium c. Nd2Fe14B + 0.7 g Neodymium.*

3.3. Magnetic Properties

M-H loops of the samples are shown in the Fig. 3 which are illustration of the data obtained from vibrating samples magnetometer, at room temperature. Magnetic properties such as saturation magnetization (Ms), coercivity (Hc) and maximum energy product (BH)max were calculated from these plots and their values are given Table 3. Magnetic flux density *B* for the energy product (BH)max was calculated by using the equation:

$$B = \mu o\,(H+M) \quad (3)$$

Here, µo is constant of permeability. B is the magnetic flux density.

It was observed that saturation magnetization (Ms) and coercivity (Hc) of the materials were increased due to the decrease in crystallite size. Hence, magnetic behavior of the samples were straightforwardly affected by the alteration in crystallite size of the material [14]. M-H loops of the samples showed an decrease in coercivity (Hc) from 274.60Oe to

261.10Oe and increase in maximum energy product (BH)max from 8.56KGOe to 17.83 KGOe. It is observed that saturation magnetization (Ms), Coercivity (Hc) and energy product (BH)max are directly affected by changes in crystallite size of the materials. Moreover, our values for Ms, Hc, and (BH)max are much better than previous reports which shows that by appropriate Nd-rich, good magnetic properties can be obtained in NdFeB magnets for required applications. The hysteresis curve of applied magnetic field of $Nd_2Fe_{14}B$ as presented in Figure 3 below.

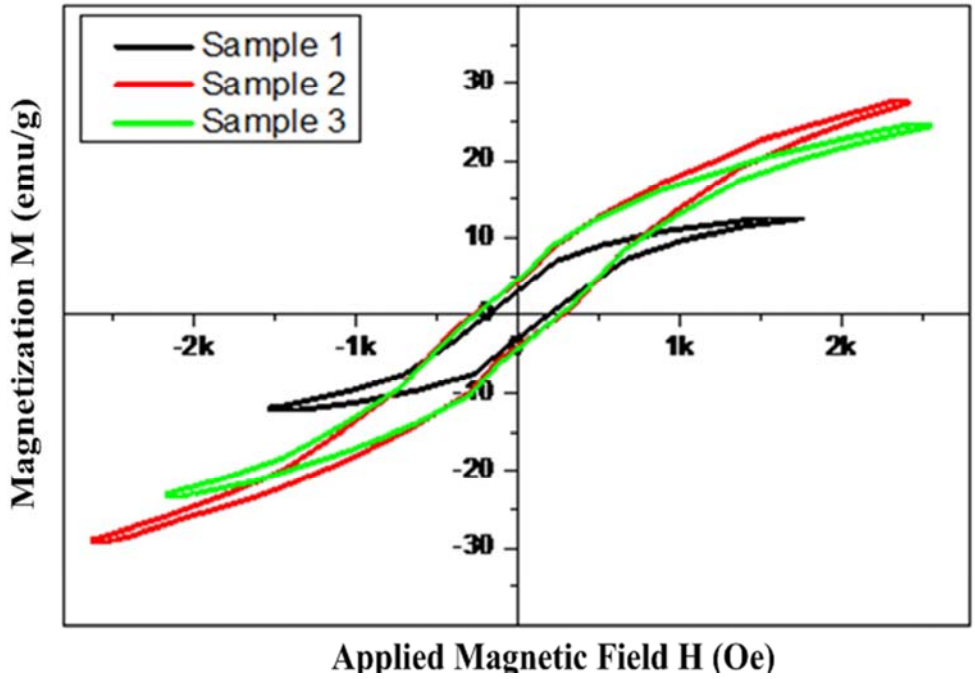

Figure 3. *M-H loops for $Nd_2Fe_{14}B$, $Nd_2Fe_{14}B$ + 0.3 gram Neodymium (Sample 2), and $Nd_2Fe_{14}B$ +0.7 gram Neodymium (Sample 3).*

The shape of the hysteresis curve denotes that our samples are in hard magnetic class. Based on Figure 3 we can get the magnetic parameters of $Nd_2Fe_{14}B$ magnet as can be listed in the Table 2. As it can be seen the value of H_c increases with increasing of Nd up to 274 Oe when we use 0.3 g Nd. However, the value of Hcdecreased to 261 Oe when Nd is increased up to 0.7 g. Other properties like magnetic remanenence and BH_{max} increased with increasing of Nd contents.

Table 2. *Magnetic parameters of $Nd_2Fe_{14}B$.*

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

4. Conclusion

Hard magnetic materials $Nd_2Fe_{14}B$ were prepared via powder metallurgy. The tetragonal crystal structure of Nd2Fe14B was confirmed with lattice parameters *a* =*b*= 8.707Å and *c* =12.203Å. The microstructural observations showed that the Nd-rich were uniformly distributed on $Nd_2Fe_{14}B$ magnets. M-H loops of the samples showed a decrease in coercivity (Hc) from 274.60Oe to 261.10Oe and increase in maximum energy product (BH)max from 8.56KGOe to 17.83 KGOe. It was observed that saturation magnetization (Ms), Coercivity (Hc) and energy product (BH)maxwere directly affected by the change in crystallite size of the materials.

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