Effect of forming process by using external magnetic field of bonded magnet made from NdFeB flakes to microstructure and magnetic properties

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Abstract. Research of fabricated bonded magnets NdFeB made from NdFeB flakes with variation of external magnetic field has been done. The materials preparation process begins with milling NdFeB flakes using High Energy Milling (HEM) for 60 minutes and mixing it with 5 wt % celula binder and performing compaction to form pellet with a pressure of 40 Kgf/cm² and then applying external magnetic field (0, 2000, 5000, 8000 and 11000 Gauss). The pellet samples were then dried using vacuum dryer with temperature of 100 °C for 1 hour. Characterization includes bulk density, measurement of magnetic properties with gauss meter, and Vibrating Sample Magnetometer (VSM). From the characterization results the best value was obtained on the external magnetic field orientation of 8000 to 11000 Gauss with a density value of 5.38 g/cm³, flux magnetic value of 465.9 – 467.1 Gauss, remanence value of 2.63 – 2.776 kGauss, and coercivity value of 1.905 – 1.925 kOe.

1. Introduction

High performance Nd-Fe-B-type bonded magnets have increasingly found new applications, such as the magnet in small motor (e.g. motor for power window), small generator, and component for flow meter [1, 2]. For preparation of bonded magnet, fine powder Nd-Fe-B-type, isotropic particles are required and they need to maintain the high permanent magnetic performance even when they are processed to fine particles [3]. The easiest way to prepare fine particles is probably crushing and milling of bulk or flakes of Nd-Fe-B-type precursor magnets. In addition to those materials, the HDDR-treated material may also be a promising candidate as a precursor material for fine particles. It is common, however, that the fine particles prepared by mechanical milling of the precursor materials lose significantly the coercivity [3, 4]. There are many sources of raw materials for making bonded magnet NdFeB such as ribbon flakes NdFeb, product from magnet quench with name code MQP-A, MQP-B, and etc.

The preparation of NdFeB powder is important step in manufacture of bonded magnet. The atmosphere of powder preparation process can influence the quality of powder, because NdFeB is easy to cause corrosion with oxygen to form oxide [5, 6]. There are some methods for making NdFeB fine powder, such as HDDR process method and mechanical alloying by milling process with hydrogen or argon gas atmosphere [7]. NdFeB magnet has high values of the remanence and coercivity, as well as
relatively high curie temperatures (312 °C) [8]. This type of magnetic alloys are identified as suitable for research and further development of magnetic composite materials with polymer matrix, and therefore it is called bonded magnets [7, 8]. There are several different process routes for bonded magnet production. The compression molding is the most common technique utilized for materials with thermo setting polymer matrices. Advantages of using bonded composite materials include their simple technology, possibility of forming their final properties, low manufacturing costs because of no costly finishing, and low material losses resulting from the possibility of forming any shape [9].

There are two kinds of bonded magnet, such as isotropic bonded magnet and anisotropic bonded magnet. Isotropic bonded magnet is a material made at forming process that is done without using an external magnetic field. It means that the direction of the domain of the particles from this material still leads randomly. It is however different for anisotropic bonded magnet, which is made at forming process using an external magnetic field. As a result, the direction of the domain of the particles from this material will be oriented in one particular direction. In general, the magnetic properties of an isotropic are greater than that of isotropic magnets [10]. In this research, we will make an isotropic permanent bonded magnet using NdFeB flakes and celuna as binder. The purpose of this research is to know the effect of external magnetic field to the microstructure and its magnetic properties.

2. Experimental works
A bonded magnet is made from NdFeB flakes by using a celuna as binder. The NdFeB powder preparation step was done by using High Energy milling (HEM) machine for milling of NdFeB flakes for 60 minutes. The resulting powder was then dried at 80 °C by using a vacuum dryer. Then the fine powder was analyzed by using XRD to observe the crystal structure. The dry powder was then mixed with 5% of the celuna binder and was formed as pellets by using an external magnetic field (0, 2000, 5000, 8000 and 11000 Gauss) and a pressure of 40 kg/cm². The resulting pellets were dried at 100 °C by using a vacuum dryer for 2 hours, and then all samples were magnetized by using impulse magnetizer. Characterization was performed including measurement of bulk density, flux magnetic, hysteresis curve, and microstructure.

3. Results and discussion
Sample NdFeB powder after wet milling for 60 minutes was analyzed by using XRD-Rigaku; figure 1 shows XRD patterns. The analysis using XRD aims to observe the phases formed on the sample powder. The peaks matched with JCPDS cards number 79-1994 were marked with circle which show that all peaks in the figure 1 are Nd₂Fe₁₄B phase.

![Figure 1. XRD patterns of NdFeB powder after wet milling for 60 minutes.](image)

The fine powder was measured distribution of particle size by using laser particle size analyzer (PSA), figure 2 shows PSA result of sample after milling 60 minutes. The fine powder NdFeB has mean diameter about 21.29 µm after milling using HEM for 60 minutes and the PSA curve as shown in figure 2 has a narrow distribution curve, which means that the NdFeB powder has a homogeneous particle size.
Figure 2. Distribution particle size of NdFeB powder after milling 60 minutes.

NdFeB fine powder was formed by using pressure at 40 kgf/cm² and external magnetic field was applied at 0, 2000, 5000, 8000, and 11000 Gauss. The resulted pellets were dried at 100 °C in vacuum dryer, then all samples were magnetized by using impulse magnetizer. Subsequently, bulk density and flux magnetic of the bonded magnet were measured for all samples and the results were shown at figure 3. The bulk density value of the bonded magnet NdFeB tends to be constant with an external magnetic field variation of about 5.13-5.14 g/cm³. When compared with the theoretical density value of NdFeB that is about 6-7 g/cm³ [11], then the density obtained from this experiment only reaches about 78.92–79.08% from theoretical density. The density of the samples made in this experiment has not yet reached a high density (> 90%), which is strongly influenced by the pressure of compaction used; the compression pressure of 40 kgf/cm² is probably not enough and the compaction pressure should be much higher. Nevertheless, the magnetic properties, especially the flux magnetic value, are greatly influenced by the value of the external magnetic field.

The highest flux magnetic value achieved is about 465.9–467.1 Gauss with applied external magnetic field 8000–11000 Gauss. When compared with other author's research results, where NdFeB permanent bonded magnets have magnetic flux values of 40-60% of the remanence value (Br) that should be 1000-2000 Gauss for NdFeB bonded magnets [12, 13]. This is due to the raw material factor used in the experiment, as seen from the XRD analysis (figure 1) where the intensity of the NdFeB peaks seems to be still too low. This is causing the flux magnetic to remain very low. The effect of providing external magnetic fields on the forming process does not have a significant effect on flux magnetic value.

The properties of magnetic materials are a manifestation of the hysteresis phenomena, where hard magnetic materials have greater values of hysteresis [14]. Magnetic properties of an isotropic bonded magnets are affected by the fraction of magnetic powder and external magnetic field used [11]. One of the most important characteristics of the rare-earth magnetic material is high values of remanence and coercivity, which have direct influence on high values of maximal energy product [9]. The result of hysteresis curve analysis for bonded magnet NdFeB as shown in figure 4 shows that the magnitude of the external magnetic field used affects the area of the hysteresis curve. The larger the external magnetic field, the VSM hysteresis curve tends to be slightly enlarged. The samples prepared by using external magnetic field pressing were measured using VSM, whereas the samples prepared without using external magnetic field pressing were not. Magnetic flux measurement results and hysteresis curve at figures 4a to 4d indicated that the magnetic flux value and hysteresis curves are almost the same for all samples. From the hysteresis curve shown in figure 4 properties of isotropic bonded
magnet NdFeB such as remanence and coercivity can be obtained.

![Graph showing bulk density and flux magnetic values as a function of external magnetic field.](image)

**Figure 3.** Bulk density value and flux magnetic value as function of external magnetic field.

![Graphs showing hysteresis curves for isotropic NdFeB bonded magnet samples.](image)

**Figure 4.** The hysteresis curve of an isotropic NdFeB bonded magnet samples made with external magnetic fields: (a) 2000 Gauss; (b) 5000 Gauss; (c) 8000 Gauss; (d) 11000 Gauss.
Table 1 shows the remanence and coercivity values of the isotropic bonded magnetic samples made from NdFeB flakes and the variations of external magnetic application. When compared to the magnet (Br, Hc) bonded magnetic NdFeB value of another author having Br about 6-8 kGauss and Hc about 4-10 kOe [7-9], the bonded magnet resulting from this research is still lower, possibly due to different raw materials result. The starting material needs to be heated first to obtain more perfect crystallization.

**Table 1. Value of remanence (Mr) and coercivity (Hc) for bonded magnet NdFeB.**

| External Magnetic Field, Gauss | Mr (emu/g) | Mr (kGauss) | Hc (kOe) |
|-------------------------------|------------|-------------|---------|
| 2000                          | 37.86      | 2.550       | 1.783   |
| 5000                          | 38.88      | 2.570       | 1.903   |
| 8000                          | 40.77      | 2.632       | 1.925   |
| 11000                         | 42.68      | 2.776       | 1.905   |

SEM micrographs of isotropic bonded magnet Nd-Fe-B sample made with celuna binder and external magnetic field 8000 Gauss and 11000 Gauss are presented in figure 5. It shows that the shape and size of NdFeB particles is in homogeneous. The largest particles have size of about 4-5 μm, and there is an agglomeration of smaller particles of about 0.5-1 μm as shown in figures 5a and 5b. The celuna binder is not clearly visible and possibly envelops the entire surface of the particle. The effect of applied external magnetic field is not clearly seen, so the change of remanence value and coercivity in various applied external magnetic field is not too large.

**Figure 5.** SEM micrographs of anisotropi bonded magnet NdFeB made with celuna binder and external magnetic field (a) 8000 Gauss and (b) 11000 Gauss.

**4. Conclusion**

A bonded magnet NdFeB can be made from NdFeB flakes and celuna binder; the highest magnetic properties are achieved at sample bonded magnet with applied external magnetic field of 8000–11000 Gauss. Evaluation of microstructure using SEM cannot see clear effect of applied external magnetic field. The magnetic properties of resulted samples bonded magnet NdFeB are flux magnetic value = 465.9–467.1 Gauss, remanence value = 2.63–2.776 kGauss, and coercivity value = 1.905–1.925 kOe.
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