Magnetic and Physical Properties Modification using Sintering Temperature Variations in the Process of Making Barium Hexaferrite Permanent Magnet

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Abstract: This paper discusses the modification of magnetic hysteresis and particle size of barium hexaferrite permanent magnet using sintering temperature variatons. The materials used were Barium Carbonate (BaCO3) and Hematite (Fe2O3) with a stochiometric ratio of (1: 6), obtained by the dry miling mixing process for 6 hours. Then this material was calcined at 1100°Celsius for 30 minutes and sieved to pass through the 200 mesh filter. The sample is compacted by 3wt% additive shellac and 5 Ton pressing to form a pellet with diameter of 5 mm. The next process is samples were sintered at temperatures of 900°C, 1000°C and 1100°C for 30 minutes. The magnetic hysteresis of this sample were then determined using VSM and microstructure analysis was determined using SEM. The results showed that the average Hmax and particle size increase for temperatur of 900°Cto 1000°C and decrease from 1000°C to 1100°C. At sintering temperature of 1000° C a granullar fusion was formed while new phase had not been formed, whereas at a sintering temperature of 1100°C a new phase had been formed which causes coercivity decreased. It was concluded that the optimal modification of magnetic properties and particle size was obtained at sintering temperature of 1000°C.

1 INTRODUCTION

Permanent magnets are the one of primary component for modern machinery equipment in various fields such as automotive machinery, electronic equipment and energy. Industrial applications require permanent magnet components with certain specifications to run the machining system. Because Indonesia as a developing industrial country, permanent magnets is demanded so high that it have to import such permanent magnet components. Thus, the local magnet industry is needed to meet domestic magnetic demand (P. Sardjono et all.,2012).

In electric machinery, a permanent magnet is a passive component in producing a magnetic field, which allows work without electric current supplied to coil or solenoid to maintain the magnetic field. The induced magnetic in the permanent magnet material will be maintained, so that when the electric current is terminated the magnetic field of the permanent magnet material remain stored (D. Jiles, 1991). Ceramic permanent magnets replace electromagnets in many applications and widely used as permanent magnets in electric motors, generators and speakers (S. Collocot, 2007).

One of the materials to produce ceramic type of permanent magnet is barium. Barium is a silvery white metal formed in nature in various forms commonly in compound forms. This material is found in nature in two forms of material, namely barium sulfate and barium carbonate which are deposits deposited on earth mantle (Clement International Corporation, 1992). The chemical properties of barium material i.e. melting point at 720°C, boiling point 1,640ºC, and density of 3,51 gcm^3 (Sunarya, S. A. 2009). This magnetic properties of the material after magnetization is permanent (M. I. Alif, 2012,), mechanical properties are very strong and not easily corroded (Snoek, 1947). In addition, mix of barium carbonate and oxide ferrite produces a permanent magnet barium hexaferrite (Priyono, 2001). The use of M.hexaferit-based barium magnets, i.e., as a microwave absorber in the aircraft cabin (D. P. Efhana et all, 2013), and a permanent magnet based on Ba/Sr-ferrite are used as measuring instruments on water meters (I. Yusan, et all,2012). Although it is very potential as a mineral material to produce magnets, in Indonesia, this

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material is a steel industry waste that has not been managed optimally.

In this research, barium ferrite permanent magnet manufacturing was performed by using powder metallurgy method. The raw material are Barium Carbonate ($BaCO_3$) and Oxide Ferrite (Fe_2O_3) with a stoichiometric ratio of 1:6, using the milling process. The next process is calcination, compaction, and sintering treatment. The heat treatment process has generally been known to have a negative impact on magnetic properties, but this process cannot be avoided in the process of metallurgy powder in order to make a strong magnet that can be utilized in machinery. Sintering is a heat treatment process that is employed to produce a dense material by adjusting the treatment for components of metal or ceramic powder. For this reason, a study of sintering conditions is needed to obtain high quality permanent magnet materials.

The next phase of this research is analysis using a scanning electron microscope (SEM) method to determine the microstructure. While the magnetic properties of the sample was observed through hysteresis curve analysis using Vibrating Sample Magnetometer (VSM).

2 RESEARCH METHODOLOGY

The powder metallurgy method is used in this research The stoichiometric ratio for barium carbonate and oxide ferrite is (1: 6) was determined. The first process of making samples is the materials were scaled using digital scales. After weighing according to the desired composition the material was mixed using ball milling in a wet state, so that the mixture obtained had a high homogeneous level. The next processeswere calcination, compaction, and sintering heat treatment. Powder material before calcination is shown in Figure 1 (a). Figure 1 (b) is a ball milling device for mixing for 6 hours. The calcination process was then carried out to form the crystalline phase of barium hexaferrite with a temperature of 1100ºC with a holding time of 30 minutes. Figure 1 (c) shows the material after calcination process.

Powder material produced from the calcination process is then sieved using 200 mesh size filter in order to obatin homogen particle size powder. Then 2% additive material was added as a binder. At the compaction stage the material that was initially in the form of granulars, was compacted to form a solid specimen as pellets with a pressure of 5 tons. The specimen was shown in Figure 2. Furthermore the specimens were subject to sintering heat treatment with variations in temperature of 900°C, 1000°C, and 1100°C with a holding time of 30 minutes.

The sintering treatment was conducted to perform granule fusion and to reduce porosity. Heating variations is for temperature of 900°C, 1000°C, and 1100°C with a holding time of 30 minutes. It was expected that a bonding process between magnetic fragments had been occured without changes ofmagnetic phase, so that a solid and hard magnetic material is obtained (Strant, Wahlfarth, et al, 1952). Holding time 30 minutes after sintering process was performed to eliminate residual stress so that the material does not crack easily and then improves its coercivity. Furthermore, test of magnetic properties using a vibrating magnetometer sample (VSM) was conducted to obtain hysteresis curve of each sample. The microstructure of sample was investigate susing scanning electron microscopic (SEM) test.

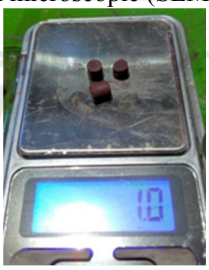


Figure 2: Specimens after compaction.

(a) (b) (c)

Figure 1: (a) powder before calcination; (b) ball milling machine; (c) after calcination.

3 RESULTS AND DISCUSSION

Results shows the effect of sintering temperatures on magnetic properties and microstructure of the magnetic samples. Temperature variation results in changes in the properties of magnetic materials both magnetic properties and physical properties. These changes include coercivity, maximum energy products of magnetic properties, and microstruc-ture.

3.1 Characterization of Magnetic Properties

Characterization of magnetic properties was based on values contained in the hysteresis curves of are shown in fig. 3. Figure 3 shows the hysteresis curve produced from barium hexaferrite material for three sintering temperature variations, namely: 900°C, 1000°C, and 1100°C and holding time 30 minutes.

Tabel 2 shows that at temperature of 900°C the H_{max} value of 1835kA/m (23.058 kOe) at test temperature of 25.3°C, Hc 449.8kA/m (5,653 kOe) and 53,10emu/g of magnetic saturation were obtained. While at temperature 1000°C H_{max} value of 1836kA/m (23.076 kOe) at test temperature of 25.0°C. Hc 467.9 kA/m (5,880 kOe)were obtained and at 1100°C Hmax value of 1832 kA/m (23.016 kOe) at test temperature 24.9°C. Hc 443.1 kA/m (5,569 kOe) were obtained. The results of Hmax for each samples is shown graphically in fig. 4. Figure 4 shows graphs of VSM test results.

Figure 4. shows the effect of sinters temperatures on magnetic coercivity. It was find that from the hysteresis curve, the effect of sintering temperature on magnetic coercivity at sintering temperature 1100^{O}C has the smallest value of 1832 kA/m (23,016 kOe), while at 1000^{O}C has value of 1836 kA/m (23,076 kOe). The highest value from hysteresis curve is for sintering temperature of 1000^{O}C. In addition, during the sintering process morphological of the particles are possibly changes. Morphological changes in particles is not only change the density between granules but also microstructure changes.

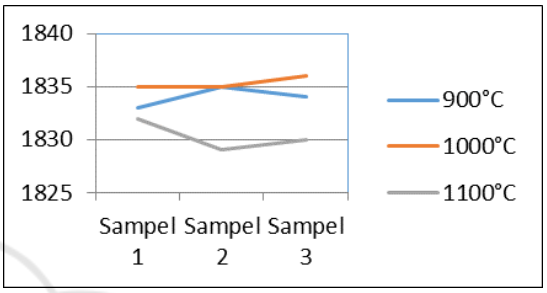
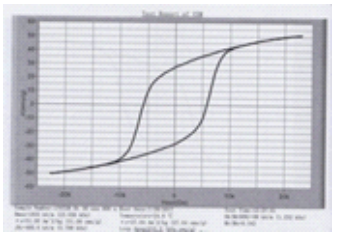


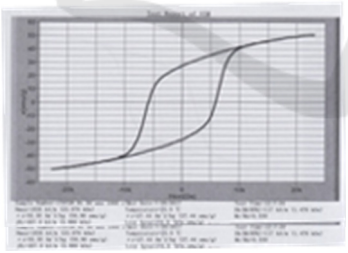
Figure 4: Graph of H_{max} values.

3.2 Microstructures

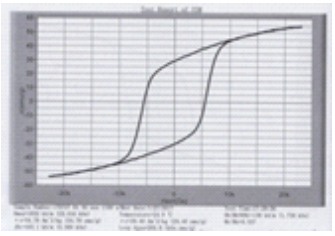
Microstructure pictures from the SEM process are shown in fig. 5. The magnification used is 5000 times for each samples with sintering temperature of 900°C, 1000°C, and 1100°C and a holding time of 30 minutes.



(a) Sintering of 900°C



(b) Sintering of 1000°C

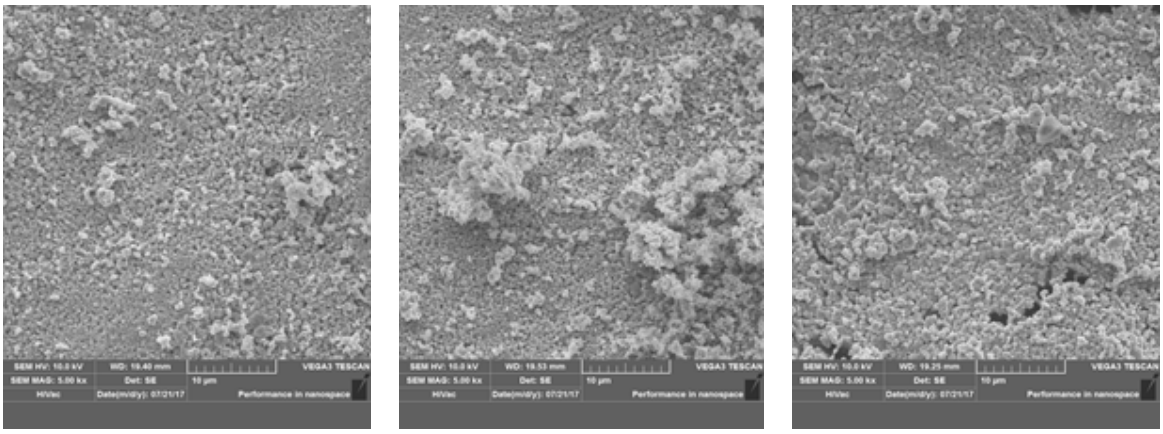


(c) Sintering of 1100°C

Figure 3: Hysteresis curves.

Table 2: Magnetic properties of barium ferrite magnets for temperature variations of sinterings.

No	*Heat Temperature*	*Material composition*	*Hmax*	*Hc*	*Magnetic Saturation*
1	900^{O}C	*barium carbonate* : *oxide ferrite* (1:6)	1835kA/m (23.058 kOe)	449,8kA/m (5,653 kOe)	53,10emu/g
2	1000^{O}C	*barium carbonate* : *oxide ferrite* (1:6)	1836kA/m (23.076 kOe)	467,9 kA/m (5,880 kOe)	53,10emu/g
3	1100^{O}C	*barium carbonate* : *oxide ferrite* (1:6)	1832kA/m (23.016 kOe)	443.1 kA/m (5,569 kOe).	55,20emu/g



(a) Temperature of 900°C (b) Temperature of 1000°C (c) Temperature of 1100°C

Figure 5: Microstructure photographs.

The magnetic analysis and microstructure analysis of barium hexaferrite material were perform-ed. It was obtain that H_{max} value is 1835 kA/m (23,058 kOe) at 900°C, and increasesto 1836 kA/m (23,076 kOe) at a temperature of 1000°C, then decreases to 1832 kA/m (23,016 kOe). Microstructure analysis was conducted the microstructure photographs for samples with sinter temperature of 900°C is shown in fig. 5. (a), sinter temperature of 1000°C is shown in fig. 5(b), and sinter temperature of 1100°C is shown in fig. 5(c). It can be seen that grain size increasesfor temperature 900°C to 1000°C than it decrease in grain size for temperature 1100°C. At sintering temperature of 1000°C, formation of a structure of granules fusion was indicated and then the magnetic coercivity increases. These results are in accordance with the reference journal which states that at high sintering temperature then coercivity increases and at temperature of 1100°C it decreases (Shi, T, S. & Grile. D, 2012,).

4 CONCLUSION

From the discussion of the research, it can be conclude that VSM test results shows that sintering temperature increases than Hmax values increases. The highest Hmax value is obtain-ed at sinter temperature of 1000°C and decreases at a temperature of 1100ºC. Therefore the optimum modification of hysteresis magnetic was at sinter temperature 1000ºC. SEM test results show that sintering temperature increases than the grain size increase. The largest grain size is achieved at a temperature of 1000°C. Therefore the optimum modification of grain size was at sinter temperatur of of 1000°C.

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