Effect of sintering temperature variations of barium hexa ferrite on physical properties and crystal structure

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Abstract. A Barium hexa ferrite BaFe\textsubscript{12}O\textsubscript{19} permanent magnet has been made by using raw materials barium carbonate BaCO\textsubscript{3} and hematite Fe\textsubscript{2}O\textsubscript{3}. The raw materials such as BaCO\textsubscript{3} and Fe\textsubscript{2}O\textsubscript{3} were mixed according to the composition mole ratio BaO:Fe\textsubscript{2}O\textsubscript{3}= 1:6, then calcined at 1050°C for one hour. The calcined powder was then crushed by using hand mortar and then mixed with WE-518 celuna as much as 3 % wt. The powder was compacted with 8 tons force to form a pellet. Further the pellets were sintered at a temperature of 1150°C, 1200°C and 1250°C with holding time one hour. The sintered samples were characterized such as: physical properties measurement (density and porosity) and micro structure analysis using XRD and magnetic properties testing measurement by vibration sample magnetometer. The characterization results show that the sample with sintering temperature of 1250°C had the highest density value of 1.43 g/cm\textsuperscript{3} and the lowest porosity value was 4.16 %. From the XRD results show that the sample that was sintered 1150°C had a single phase BaFe\textsubscript{12}O\textsubscript{19}, while the samples that had been sintered 1200°C and 1250°C were obtained in two phases, namely dominant phase is BaFe\textsubscript{12}O\textsubscript{19} and minor phase is Fe\textsubscript{2}O\textsubscript{3}. Of the three samples tested by VSM, samples with a sintering temperature of 1150°C have good conditions and magnetic properties greater than both. the sintering temperature affects the value of each category of magnetic properties of the sample.

1. Introduction
Nanostructured magnetic materials have been intensively studied, due to their applications in magnetic high-density recording media, sensors, and bio molecular separations. Driven by radar electronics, wireless technologies, and enormous progress in fundamental theoretical and experimental laboratory studies of hexagonal M type ferrites various properties, researchers have shown huge interest in it [1]. The hexagonal barium hexa ferrite (BaFe\textsubscript{12}O\textsubscript{19}) is known as a hard magnetic material and represents one of the mostly used materials in the area of magnetic recording media, which cannot be easily substituted by other magnets [2] and as a particulate media for magnetic recording as well as in microwave devices [3]. Barium hexa ferrite is a long lasting research area for nearly a century due to its high magneto crystalline anisotropy, chemical stability, excellent frequency, and curie temperature [4]. These materials have attracted because of excellence chemical stability, mechanical hardness and relatively strong magnetic properties like high magnetic anisotropy, high coercivity and saturation magnetization [5] and these materials provide dual functionality, since they possess gyro tropic
properties that allow for nonreciprocal microwave devices, and also provide magnetic self-biasing through the large uniaxial magneto crystalline anisotropy that makes them valuable as permanent magnet[4]. The large uniaxial anisotropy present in barium hexa ferrite (BaFe\(_{12}\)O\(_{19}\)) is coincident with the c-axis of the hexagonal unit cell, such that the easy axis of the magnetization lies along [6]. The ferromagnetic oxides such as ferrites and garnets exhibit dielectric and magnetic characteristics [7].

This paper reported about the effect of sintering temperature variations and of barium hexaferite on physical properties and crystal structure. It is hoped that this process will get more information about barium hexa ferrite ceramic magnets for development in the magnetic field.

2. Experiment

The process of making magnet BaFe\(_{12}\)O\(_{19}\) was done by using raw materials barium carbonate BaCO\(_3\) and hematite Fe\(_2\)O\(_3\). The raw materials such as BaCO\(_3\) and Fe\(_2\)O\(_3\) were mixed according to the composition mole ratio BaO:Fe\(_2\)O\(_3\)=1:6, then calcined at 1050°C for one hour. The calcined powder was then crushed by using hand mortar and then mixed with WE-518 celuna as much as 3 % wt. and compacted with 8 tons force to form a pellet. Further the pellets were sintered at a temperature of 1150°C, 1200°C and 1250°C with holding time one hour. The sintered samples were characterized such as: physical properties measurement (density and porosity), microstructure analysis using XRD and magnetic properties testing measurement by Vibration Sample Magnetometer (VSM).

3. Results and discussions

3.1. Density and Porosity

The level of density of a material can be determined from the sintered samples by using the Archimedes method. The result of the measurement of bulk density is shown in Figure 1.

![Figure 1. Bulk density curves as function of variations sintering temperature](image)

Figure 1 shows the highest density value at 1250 °C is 1.43 g/ cm\(^3\) and the smallest value at 1150 °C is 1.33g / cm\(^3\). This is in accordance with the theory which states that the greater the temperature, the greater the density value of a material. In general, the density of a material depends on environmental factors such as temperature and pressure.
Figure 2. Porosity curves as function of Variations sintering temperature

Figure 2 shows the highest porosity percentage shown at a temperature of 1150 °C, which is 11.11% and the smallest percentage of porosity shown at a temperature of 1250°C at 4.16%. This is in accordance with the theory that the relationship between porosity and sintering temperature is inversely proportional. The porosity value decreases if the sintering temperature increases.

3.2. XRD

The samples were tested using XRD to determine the phase formed and the composition contained in the sample. Here are the results of XRD analysis of BaFe$_{12}$O$_{19}$ samples with variations in sintering temperature (1150°C, 1200°C and 1250°C).

Figure 3. Results of XRD analysis for sintering temperature 1150 °C

Figure 3 shows that there are three phases such as: BaFe$_{12}$O$_{19}$ as dominant phase and Fe$_2$O$_3$, Fe$_3$O$_4$ as minor phase. For sintering temperatures of 1150 °C there are many peaks that form BaFe$_{12}$O$_{19}$ (Barium Hexa ferrite), Fe$_2$O$_3$ and Fe$_3$O$_4$ phases. The major phase in this sample is BaFe$_{12}$O$_{19}$ (Barium Hexferrite) and the minor phase in this sample is Fe$_2$O$_3$ and Fe$_3$O$_4$. It can be seen that the sample with a sintering temperature of 1150 °C dominant phase is dominated by BaFe$_{12}$O$_{19}$ at an angle of 2θ = 32.130° and its intensity is 260 and the crystal size is 266 Å. At the sintering temperature of 1150 °C, barium hexa ferrite magnetic material (BaFe$_{12}$O$_{19}$) has been formed but the intensity is still small at 217 while the minor phase is Fe$_3$O$_4$ and Fe$_2$O$_3$ and CO$_2$ in barium carbonate has evaporated at a temperature of 1150 °C.
Figure 4. Results of XRD analysis for sintering temperature 1200 °C

Figure 4 indicates that, there are two phases such as: BaFe$_{12}$O$_{19}$ phase and Fe$_2$O$_3$ phase at sample sintered of 1200 °C in its dominance phase at the highest peak by BaFe$_{12}$O$_{19}$ with an angle of $2\theta = 32.137$ ° and the crystal size is 269 Å and the minor phase is hematite (Fe$_2$O$_3$). The barium hexaferrite phase (BaFe$_{12}$O$_{19}$) shows a slight increase in view of the shift in angle $2\theta$ experiencing an increase and there is a phase that is lost at this temperature, namely magnetite (Fe$_3$O$_4$).

Figure 5. Results of XRD analysis for sintering temperature 1250 °C

Figure 5 indicates that, there are two phases such as: BaFe$_{12}$O$_{19}$ phase and Fe$_2$O$_3$ phase. In the picture above it can be seen that the sample with a sintering temperature of 1250 °C has the highest peak with dominant phase BaFe$_{12}$O$_{19}$ at $2\theta = 34.134$ ° and its crystalline size of 271 Å.

3.3. Vibration Sample Magnetometer (VSM)

The measurement of magnetic properties was done by using VSM to know the value of Remanence (Mr), Coercivity force (Hc), Magnetic saturation (Ms) and Energy product (BHmax) and the results are shown in figure 6.
Figure 6. Hysteresis curve of sample magnet with 1150 °C, 1200 °C and 1250°C.

In the picture above can be seen the effect of the addition of sintering temperature greatly affects the greater the temperature given, the smaller the magnetic properties, for more details can be seen in table1.

Table 1. Value of Remanence (Mr), Coercivity (Hc), Magnetic Saturation (Ms) and Energy Product (BH max).

| Temperature Sintering °C | Mr (emu/g) | Hc (kOe) | Ms (emu/g) | Bhmax (kOe) |
|--------------------------|------------|----------|------------|-------------|
| 1150                     | 27.63      | 2.544    | 57.44      | 20.129      |
| 1200                     | 26.68      | 2.399    | 56.79      | 20.085      |
| 1250                     | 39.40      | 0.178    | 122        | 20.054      |

Based on Table 1, the highest saturation value is 122 emu/g on the magnetic material of the sample with a sintering temperature of 1250 °C, and this sample has coercivity value about 0.178 kOe and magnetic remanence about 39.40 emu/g. The coercivity (Hc) is the magnitude of the external magnetic field (H) needed to remove the magnetic field in the magnetic material of the sample which has the greatest value in the magnetic material of the sample with a temperature of 1150 °C, which is 2,544 kOe. Of the three samples tested, samples with a sintering temperature of 1150 °C have good conditions and magnetic properties greater than both. So that, the sintering temperature affects the value of each category of magnetic properties of the sample.
4. Conclusions
A permanent bonded magnet $\text{BaFe}_{12}\text{O}_{19}$ has successfully made from raw materials of by using raw materials barium carbonate $\text{BaCO}_3$ and hematite $\text{Fe}_2\text{O}_3$. The raw materials such as $\text{BaCO}_3$ and $\text{Fe}_2\text{O}_3$ were mixed according to the composition mole ratio $\text{BaO}:\text{Fe}_2\text{O}_3=1:6$ and its properties were sintered at a temperature of 1150°C, 1200°C and 1250°C with holding time one hour. The sintered samples were characterized such as bulk density value = 1.33 – 1.43 g/cm$^3$, Porosity values = 4.16-11.11%, flux magnetic value = 1150 - 1170 Gauss, the remanence value = 27.63-39.40 emu/g, magnetic saturation value = 57.44-122 emu/g, coercivity value = 0.178-2.544 kOe and energy product = 20.054-20.129 kOe. based on the most dominant phase XRD analysis test at each temperature is $\text{BaFe}_{12}\text{O}_{19}$.

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