Effect of temperature sintering on characterization of \( \text{BaFe}_{12-4x}\text{Al}_{4x}\text{O}_{19} \) on \( x = 0.001 \) prepared by powder metallurgy method

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Abstract The synthesis of \( \text{BaFe}_{12-4x}\text{Al}_{4x}\text{O}_{19} \) with \( x = 0.001 \) was done by using the powder metallurgy method with the raw materials such as : \( \text{BaO}, \text{Fe}_2\text{O}_3 \) and \( \text{Al}_2\text{O}_3 \). The raw materials were mixed and calcined at 1050 °C with holding time of 1 hour. After calcination, the powder was mixed with 3% wt. of celuna, then compacted with force of 8000 kgf. Then, the pellets were sintered at temperature of 1150 °C, 1200 °C, 1250 °C with holding time of 1 hour. The characterization of sintered samples was conducted such as : measurement of bulk density, porosity, XRD and magnetic properties. It shows that the highest value of bulk density about 4.379 g/cm\(^3\) and lowest porosity about 9.81 % were achieved for samples sintered at 1250°C. While in XRD test result shows that it was found some phases such as \( \text{BaFe}_{12}\text{O}_{19} \) as dominant phase and \( \text{Fe}_2\text{O}_3 \) phase as minor phase. The temperature sintering gives a significantly value of bulk density and porosity, whereas the increase in sintering temperature can cause a shift in the peak of \( \text{BaFe}_{12}\text{O}_{19} \), which in this case refers to the substitution of \( \text{Fe} \) atoms with \( \text{Al} \) atoms. Sintering temperature can influence significantly magnetic properties, samples sintered 1150 and 1200°C become hard magnetic and sample sintered 1250°C becomes soft magnetic.

1. Introduction
Ferrite also known as ceramic magnet began to be developed in 1950 and began production in 1952 by Philips with the name Ferroxdure production as one of the results of the Stoner-Wohlfarth theory [1]. Ferrite magnets are widely used, namely Barium Ferrite \( \text{BaO}_6\text{Fe}_2\text{O}_3 \)besides \( \text{SrO}_6\text{Fe}_2\text{O}_3 \) and \( \text{PbO}_6\text{Fe}_2\text{O}_3 \) [1,2]. Ferrite magnets have strong mechanical properties and are not easily corroded. Besides that, ferrite magnets have a high coercivity with a level of stability against the influence of external fields and a fairly good temperature [2]. Barium hexaferrite (\( \text{BaO}_6\text{Fe}_2\text{O}_3 \)) has a hexagonal crystal structure with lattice parameters \( a = 5.8920 \) Å and \( c = 23.1830 \) Å [3] is a permanent magnetic material that is widely used in industries such as part of : electronics, motor and sensor [3]. The manufacture of permanent magnets can be done in several ways, such as sol-gel and powder metallurgy, powder metallurgy methods are used more frequently because they are relatively economical and easy to do [4,5]. To overcome these problems, a lot of research has been done to improve its usability, namely the addition of additive materials such as \( \text{TiO}_2 \), \( \text{SiO}_2 \) and \( \text{Al}_2\text{O}_3 \) so that it is expected to be able to control grain growth and improve magnetic properties such as coercivity and remanence and toughness of materials [6]. With the addition of \( \text{Al}_2\text{O}_3 \), it is hoped that it can control...
grain growth and increase the ductility of the material because Alumina has a high melting point so it does not change the crystal structure [7]. In this study we used addition of Al2O3 about 0.1% (g / mol) on preparation of BaO6Fe2O3, the purpose of this research is to know the influence of addition of Al2O3 to physical properties, micro structure at variation of sintering temperature.

2. Research Method
The preparation of BaFe12-4xAl4xO19 begins with a mixture of BaCO3 from E-merck as BaO source, Fe2O3 from E-Merck and γ - Al2O3 from E-merck with the concentration of x = 0.1% (g / mol) . Then the raw materials were calcined at a temperature of 1050 °C. After the calcination process, the samples were crushed using hand mortar then added 3% celuna as a binder, and then put into the mold and pressed with 8 tonf force to form pellets. The sample pellets were sintered by electrical furnace and sintering temperature is varied at 1150˚C, 1200 °C and 1250 °C with holding time 1 hour. The characterization of sintered samples was conducted such as : measurement of bulk density, porosity, XRD to know the phase structure of the sample and magnetic properties by using Vibrating Sample Magnetometer (VSM).

3. Results and Discussion
From the results of the study found that the value of the bulk density and porosity after sintering can be seen in table 1.

| Suhu(°C) | Densitas(g/cm³) | Porositas(%) |
|---------|----------------|--------------|
| 1150    | 3.958          | 13.17        |
| 1200    | 4.055          | 11.67        |
| 1250    | 4.379          | 9.81         |

From table 1 above, a graph can be made on the effect of sintering temperature on the density and porosity of the sample as seen at figure 1.

![Graph of the effect of sintering temperature on density and porosity](image)

From figure 1 above, it can be seen that the greater the sintering temperature variation given, the greater the density value of the material and the smaller porosity. It is possible that the greater the temperature causes the density of the structure in the sample to become denser so that the density is greater. With a large density, the pores in the material get smaller. The highest bulk density value was reached at 4.379 g / cm³ after being sintered at a temperature of 1250°C. Theoretically, the BaFe12O19 density is about 5.10-5.56 g/cm³ [8]; however, according to the comparison value between the experimental and theoretical density, the densification sample after sintering at 1250°C is achieved about 82.16% , and this is still classified as a low level of
densification, due to particle size or compression pressure used, or to be able to increase densification, the sintering temperature can still be increased again. Meanwhile, the results of the x ray diffraction (XRD) test to analyze the structure and phase and the size of the crystals have been shown in figure 2 below. In figure 2 above, it can be seen that the xrd patterns for each sample at different sintering temperature is dominated by the major phase BaO₆Fe₂O₃ and there are some patterns as minor phase Fe₂O₃. From the results of the XRD analysis shows that the Al₂O₃ phase does not appear, this is the possibility that Al atoms enter the crystal structure of BaO₆Fe₂O₃ by substituting part of the Fe atom with the Al atom.

![Figure 2](image1)

**Figure 2.** Graphs of material diffraction patterns for sintering temperature variations

Analysis magnetic properties of all samples were conducted by using Vibrating Sample Magnetometer (VSM), the hysteresis loop of sample after sintering at temperature 1150, 1200 and 1250 °C are shown at figure 3. According hysteresis loop at figure 3, magnetic properties namely : magnetic saturation, remanence, coercivity for each sample can be seen at table 2.

![Figure 3](image2)

**Figure 3.** Hysteresis loop of Sample after sintering 1150, 1200 and 1250°C
Table 2. Magnetic properties for sample after sintering process

| Sintering Temperature, °C | Magnetic Saturation, emu/g | Remanence, emu/g | Coercivity, kOe |
|--------------------------|-----------------------------|-----------------|---------------|
| 1150                     | 57.44                       | 27.63           | 2.544         |
| 1200                     | 56.79                       | 26.68           | 2.399         |
| 1250                     | 122                         | 39.40           | 0.178         |

Based on the results of measurements with VSM, shows that the sample with sintering temperatures of 1150 and 1200°C produces the same hysteresis loop pattern, which is a wider hysteresis loop than the hysteresis loop of the sample which is sintered 1250°C. This shows a significant decrease in the coercivity value, that is from order kOe to Oe as can be seen in table 2. Changes in the magnetic properties of the sample sintered at 1250°C were due to changes in the crystal structure as seen in the XRD pattern in figure 2, where there was a reduction in the peak of BaO_6Fe_2O_3 and increased the intensity of Fe_2O_3, so the magnetic properties changed to soft magnetic.

4. Conclusions
From the results described above it can be concluded that the addition of additive Al_2O_3 and sintering temperature variation affects significantly to the density, porosity and crystal structure changes and magnetic properties in the material. The highest value of bulk density about 4.379 gr/cm^3 and lowest porosity about 9.81 % were achieved for samples sintered at 1250°C. Based on XRD results show that it was found some phases such as BaFe_{12}O_{19} as dominant phase and Fe_2O_3 phase as minor phase. Whereas the increase in sintering temperature can cause a shift in the peak of BaFe_{12}O_{19}, which in this case refers to the substitution of Fe atoms with Al atoms. Sintering temperature can influence significantly magnetic properties, samples sintered 1150 and 1200°C become hard magnetic and sample sintered 1250°C becomes soft magnetic.

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