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Shrinkage, Density and Hardness of Hard Magnetic Material (BaFe₁₂O₁₉) Based on Iron Sand Produced by Conventional Solid-State Reaction Process

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Abstract. This paper presents shrinkage, density and hardness number of hard magnetic (BaFe₁₂O₁₉) based on iron sand produced by conventional solid-state reaction process. Iron sand was mechanically filtered using permanent magnets 35 times. The filtered iron sand was heated at temperatures of 900°C for 5 hours in the furnace and after it was cold and produce Fe₂O₃ phase. Powders of Fe₂O₃ and BaCO₃ were mixed and milled in a shaker ball mill up to 3 hours. The powder mixture compacted at a pressure of 2.5, 5 and 7.5 tons and followed by sintering at temperature of 1100, 1150 and 1200°C for 1 hour in the furnace. Shrinkage measurements include diameter and height uses vernier caliper, while density measurements use the Archimedes method. Hardness number obtained with pass vickers hardness testing methods. Barium ferrite 's maximum shrinkage and bulk density values were at 7.5 tons compacting pressure and 1200°C sintering temperature where the shrinkage value was 7.44 percent, average shrinkage was 3.49 percent, and density was 4.397 g/cm³. In barium ferrite with a compacting pressure of 7.5 tons and a sintering temperature of 1200 °C which is equivalent to 741 HV the highest hardness value is found. The higher the compacting press and sintering temperature, the greater the importance of bulk density and hardness of the materials.

1. Introduction

The large amount of iron sand found in the south coast of Java has the great potential to be a special hard magnetic base material and is generally adopted as a base for electronic components [1-3]. Iron sand in general has a dominant chemical compound such as magnetite (Fe₃O₄), maghemite (γ-Fe₂O₃) and hematite (α-Fe₂O₃) [4-7]. Magnetite (Fe₃O₄) is the mineral that has the highest iron content, that hits up to 72.4% [8]. This mineral is known to have the strongest magnetic properties. Thus, it is also known as Lodestone (magnetic stone) [9]. Magnetite is used as the main ingredient of iron ore in the manufacture of steel and iron [8]. At the nanometer scale, the material with this magnetite compound has superparamagnetic properties and has other properties such as saturation magnetization, biological compatibility and environmental stability which are better when compared to the size on the bulk scale [10-14].

Maghemite (γ-Fe₂O₃) is a strongly ferrimagnetic which has the same crystal structure with magnetite and similar chemical composition with hematite but has a different crystal structure where the maghemite has a crystal cubic structure and hematite has a hexagonal crystal structure [15-20]. Maghemites in natural environment are typically formed by the process of oxidation of magnetite at low temperatures [15,17]. Compared with magnetite the presence of maghemites in natural environment is very small. The cycle of oxidation (addition of oxygen) by giving heat to magnetite at certain temperatures becomes maghemite and hematite [21-25]. Maghemite produced through the magnetite oxidation process is characterized by a color shift from black to reddish brown [26-29]. Maghemite is widely applied in the biomedical field [30-



35], magnetic recording media [36] and nanoparticle technology, namely in the treatment of cancer cells in hyperthermia [37-38].

Hematite is the mineral form of iron (III) oxides ($\alpha\text{-Fe}_2\text{O}_3$) which are antiferro magnetic or in other words hematite is not magnetic and should not respond to a common magnet. However, many specimens of hematite contain enough magnetite that they are attracted to a common magnet [39]. Iron (III) oxides ($\alpha\text{-Fe}_2\text{O}_3$) can be synthesized using different techniques including co-precipitation method [40], thermal decomposition [41-43], hydrothermal synthesis [10, 14, 44, 45], microemulsion [46, 47], and sonochemical synthesis [48, 49]. Iron (III) oxides ($\alpha\text{-Fe}_2\text{O}_3$) are used more commonly for the manufacture of pigments, products for radiation control, ballasts, solar cells, microwave absorption, catalysis, environmental protection, gas sensors, magnetic storage, clinical diagnosis and treatment, etc [39, 50-54]. Iron (III) oxides ($\alpha\text{-Fe}_2\text{O}_3$) are more commonly used as a base material for ferrite magnets that have hard magnetic properties [55]. The magnetic properties of iron (III) oxides ($\alpha\text{-Fe}_2\text{O}_3$) were demonstrated to depend on their size, shape and microstructure [45, 56]. Iron (III) oxides ($\alpha\text{-Fe}_2\text{O}_3$) are an enticing category of products, ranging from antiferromagnetic, superparamagnetic and bulky ferromagnetic to ferrimagnetic [57, 58]. When the particle size decreases, the magnetic properties of iron (III) oxides ($\alpha\text{-Fe}_2\text{O}_3$) have unusual properties that vary from those of bulk equivalents due to nanoscale concentration and surface impact [56, 59].

Iron (III) oxides ($\alpha\text{-Fe}_2\text{O}_3$) are more commonly used in ferrite magnets with hard magnetic properties as the base material. Ferrite magnets can generally be made using compound Iron (III) oxides added by BaCO_3 to manufacture barium hexaferrite magnets and SrCO_3 to produce strontium hexaferrite magnets [60-62]. The magnetic properties of ferrite are influenced by several factors including the level of purity and types of constituent compounds, stoichiometry, crystallite size, microstructure, preparation and manufacturing processes [63]. The process of preparing and producing ferrite magnets can be accomplished using many techniques or methods, including sol-gel, coprecipitation, salt-melting, citrate, hydrothermal, combustion, high-temperature self-propagation and normal ceramic techniques (solid-state reaction, mechanical milling and alloying) [63].

The process of making ferrite magnets, especially barium hexaferrite using standard ceramic techniques, where Fe_2O_3 and BaCO_3 powders are processed by mechanical milling and alloying followed by a sintering process. This process is also commonly referred to as the solid-state reaction process, where during the sintering process a reaction occurs between Fe_2O_3 and BaCO_3 resulting in barium hexaferrite ($\text{Ba}_0.6\text{Fe}_{12}\text{O}_{19}$ or $\text{BaFe}_{12}\text{O}_{19}$). Before the sintering process is carried out, the samples are given emphasis treatment (compacting) and some are not. Things that need to be considered before, during and after the sintering process include the heating rate, temperature variations, atmosphere, holding time and cooling rate which will affect the $\text{BaFe}_{12}\text{O}_{19}$ microstructure [63]. Microstructure material affects many things such as physical, mechanical, magnetic etc. Many solid-state reaction processes are carried out because they have advantages including mass production, are easy, simple, inexpensive and can produce nanoparticles [64, 65]. This research reports the effect of compacting pressure and sintering temperature on shrinkage, bulk density and hardness number on $\text{BaFe}_{12}\text{O}_{19}$ hard magnetic material based on iron sand by solid-state reaction process.

2. Experiments

Iron sand from Ketawang Indah beach with permanent magnetic rocks from Purworejo is mechanically extracted 35 times. The iron sand removed was then oxidized over 5 hours at a heating temperature of 900°C . Iron oxide and barium carbonate (BaCO_3) were weighed for based on stoichiometry calculations and then followed by mechanical alloying and milling treatment using a shaker ball mill for 3 hours.

After the mechanical alloying and milling process the mixture of Fe_2O_3 and BaCO_3 powders was molded with compacting pressure variation; 2.5, 5 and 7.5 tons where the diameter of the dies is 2 cm. The sample of compacting or green compact is measured in diameter and height dimensions using Mitutoyo Vernier Caliper Metric with an accuracy of 0.05 mm. Then the green compact sample was sintered with variations in temperature of 1100°C , 1150°C , and 1200°C for 1 hour using furnace (Nabertherm N31/H) until room temperature. The sample is measured shrinkage which includes shrinkage of diameter and height using vernier caliper and then calculated using the given equation 1 and 2.

$$\Delta d = \frac{d_1 - d_2}{d_1} \times 100 \% \quad (1)$$

$$\Delta h = \frac{h_1 - h_2}{h_1} \times 100 \% \quad (2)$$

Where, Δd = diameter shrinkage (%); Δh = high shrinkage (%); d_1 = diameter of the sample before sintering (mm); d_2 = diameter of the sample after sintering (mm); h_1 = sample height before sintering (mm); and h_2 = sample height after sintering (mm).

Bulk density sample measurements were done after the sintering process using the Archimedes method [66]. Whilst the material's hardness value was measured using the Vickers method using the M800 microhardness tool with a load of 100 gf held for 10 seconds.

3. Result and discussion

3.1. Shrinkage

Depreciation testing on samples is done on the dimensions of diameter (Δd) and specimen height (Δh). The results of diameter shrinkage test (Δd) with variations in compacting pressure and sintering temperature as shown in the Figure 1.

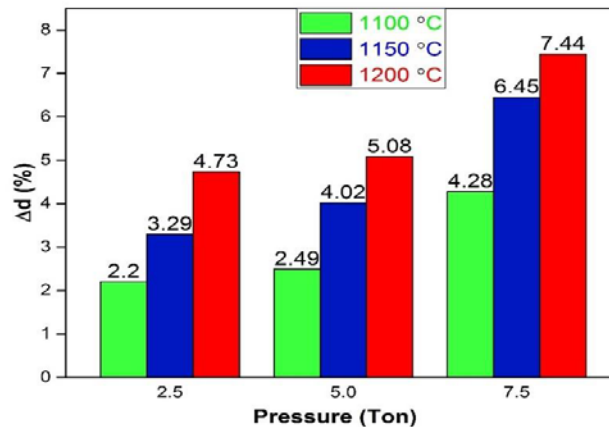


Figure 1. Shrinkage of diameter (Δd)

Figure 1 shows a significant increase in diameter shrinkage of the specimen when treated with variations in compacting pressure and sintering temperature. The test results show that the specimens treated with 2.5 ton compaction pressure and sintering temperature of 1100°C had the smallest diameter shrinkage value of 2.2%. In the sintering treatment 1150°C has a diameter shrinkage value of 3.29%. Whereas the 1200°C sintering treatment has a diameter shrinkage value of 4.37%. The increase in the value of diameter shrinkage that occurs at a pressure of 2.5 tons along with the increase in sintering temperature is around 1.09-2.17%. For specimens treated with 5 ton compacting pressure, the magnitude of the diameter depreciation is not adrift with diameter shrinkage values for specimens treated with 2.5 ton compacting pressure at each sintering temperature. The largest diameter shrinkage value in the specimen which was given a compacting pressure of 7.5 tons in each sintering treatment, especially in the 1200°C sintering treatment which has the largest diameter shrinkage value of 7.44%. The value of diameter shrinkage that occurs at a pressure of 7.5 tons at each variation of the sintering temperature is 2.17-3.16%. This indicates that the higher the compacting pressure and sintering temperatures cause the denser conditions of the specimen.

The results of high shrinkage test (Δh) samples with variations in compacting pressure and sintering temperature as shown in Figure 2.

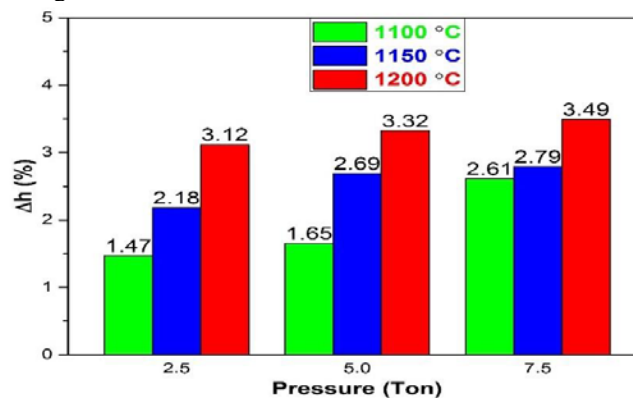


Figure 2. Shrinkage of high (Δh)

In Figure 2, it can be seen that the specimens given a 2.5 ton compacting pressure treatment and sintering temperature of 1100°C have the smallest high shrinkage value of 1.47%. While the highest high shrinkage value is owned by the specimen with a compacting pressure of 7.5 tons and a sintering

temperature of 1200°C which is 3.49%. The graph trend in Figure 2 is almost the same as the graph trend in Figure 1, where the cause of the phenomenon is that the higher the pressure and sintering temperature to 1200°C causes the denser conditions of the specimen and the lower the specimen's ability to return to its original dimensions after sintering. In microstructure this happens due to the diffusion process (mass transportation) of atoms between particles resulting in the growth of grains and eliminating pores in the specimen. High shrinkage speed can affect the characteristics of the sintering material, where the uneven distribution of sintering temperatures can cause residual stresses that are the source of cracks [67-70].

3.2. Bulk Density Measurement

The results of bulk density testing in samples with variations in compacting pressure and sintering temperature can be seen in Figure 3. In Figure 3 it is clear that the specimens which are compacted at 7.5 tons and sintered at 1200 °C have the highest bulk density values of 4.4 g/cm³. Overall the increase in the value of bulk density that occurs along with the increase in compacting pressure and sintering temperature is around 0.091-0.186%.

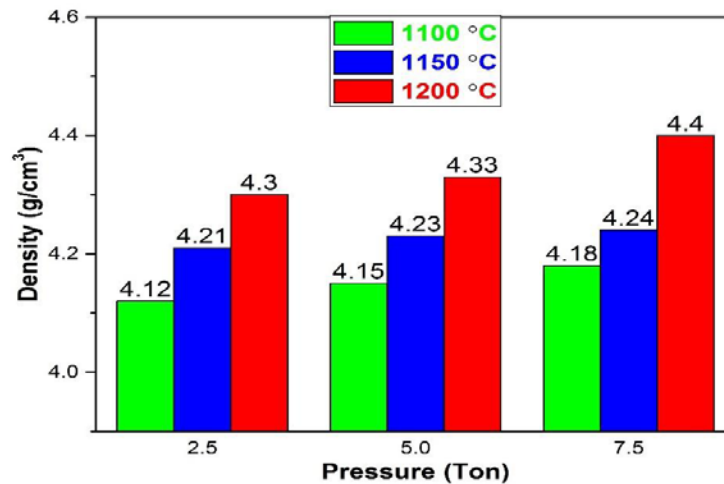


Figure 3. Bulk density

Research conducted by Kristiantoro et.al (2019) got a bulk density value of 4.65 g/cm³, where the increasing value of density also increases the value of remanence (Br) and maximum product energy (BHmax), but further decreases the value of coercivity (Hc) [71]. The highest barium hexaferrite bulk density value ever achieved in previous studies was reported at 5,295 g/cm³ [63], while the theoretical density of barium hexaferrite is 5.32 g/cm³ [72]. When compared with the highest bulk density value ever obtained and the theoretical density value of the results obtained in this study, the value is still smaller, this is due to the possibility that there are still many pores in the specimen even though this research has not been explored further on this subject. The application of higher compacting pressure and sintering at a temperature of 1200°C allows the particles in the specimen to disperse and the particle growth occurs during the sintering process thus reducing the pores in the specimen and this also allows the volume of the specimen to shrink [73].

3.3. Hardness testing

The results of hardness testing on the variation of compacting pressure and sintering temperature can be seen in Figure 4. Figure 4 shows that the test results show that the specimens have the lowest test hardness value of 255 HV despite a compacting pressure treatment and sintering temperature of 1100°C. Whereas the highest check hardness value belongs to the specimen with a compacting pressure of 7.5 tons and a sintering temperature of 1200°C which is 741 HV.

In the research of Kristiantoro et.al (2019) obtained a hardness value of 42.5 HRC or around 420 HV at a compacting pressure of 6 tons/cm² which is sintered 1250°C [71]. It can be concluded that the greater compacting pressure and sintering temperature would induce a higher hardness value. This is because the higher the compacting pressure applied to the specimen, the bonding of particle granules becomes stronger in order to increase or decrease the distance between particles. If the particle granules get thicker then the particles would be easier to bind during the sintering process, thus as to improve the material's hardness value.

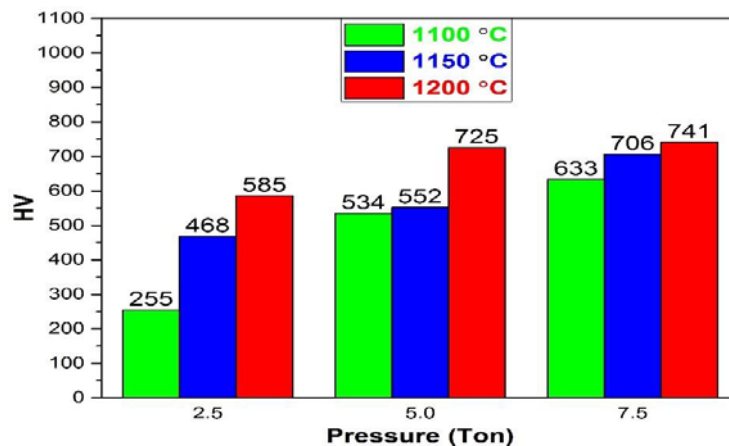


Figure 4. Vickers hardness number

4. Conclusion

Based on the research that has been done, the following conclusions can be drawn: Compacting pressure and sintering temperature affect the value of shrinkage and density of hard magnet barium ferrite-based iron sand, where the higher the compacting pressure and sintering temperature, the shrinkage value and material density will increase. The highest shrinkage and density values were found in barium ferrite with the treatment of compacting pressure of 7.5 tons and sintering temperature of 1200°C, with diameter shrinkage of 7.44%, high shrinkage of 3.49%, and density value of 4.397 g/cm³.

The compacting pressure and sintering temperature affect the hardness of barium ferrite magnets based on iron powder. The higher the compacting press and sintering temperature, the greater the significance of material hardness. For barium ferrite with a compacting pressure of 7.5 tons and a sintering temperature of 1200°C which is equivalent to 741 HV the maximum hardness value is found.

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