Synthesis and Characterization of Strontium Hexa Ferrite ($SrFe_{12}O_{19}$) Powder by using Powder Metallurgy Method

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Abstract: The Strontium hexa ferrite with formula $SrFe_{12}O_{19}$ or SrO $6Fe_2O_3$ is permanant magnet materials , it has high magnetic properties, high curie temperature (350ºC) and good corrosion resistance. This research was done in preparation of Strontium hexa ferrite by using raw materials : hematite (Fe_2O_3) and $SrCO_3$ at mole ratio SrO:Fe_2O_3 = 1 :6. The both raw materials were weighed and mixed by using HEM for 4 hours and aquadest as milling media. After that, the sample was dried at 110ºC for 4 hours by using drying oven. The dried sample was analyzed by using DTA/TG to know the calcination temperature. According DTA/TG curve, there are 3 peaks endothermic at 730ºC, 820ºC and 990ºC. After that , the sample was calcined at temperature 900ºC and 1000ºC for 2 hours. The calcined samples were analyzedcrystall structure by using XRD, measured magnetic properties by using VSM. According XRD results show that the first formation of $SrFe_{12}O_{19}$ phase at temperature 900ºC, With the increasing of calcination temperature can increasing of $SrFe_{12}O_{19}$ phase. The VSM results show that it is obtained a wide hysteresis curve with highest coercivity value 3000Oe.

1 INTRODUCTION

There are some of types materials as permanent magnet such as materials based on ferrite, metals alloy (SmCo and AlNiCo) and rare earth metals alloy (NdFeB), each of these materials exhibits different set of properties (Sebayang et al., 2011). Permanent magnets are widely used in various fields, among others, in the automotive industry, as components of divais energy (generators), sensor industry, components in medical equipment and others (Nowosielski et al., 2007). There are two types of ferrite permanent magnet namely Sr ferrite and Ba ferrite, which the magnetic properties of Sr ferrite are slightly higher than Ba ferrite, for example energy product of Sr ferrite is about 0.30 kJ/m^3 and about 27 for Ba ferrite (Slusarek & Zakrzewski, 2012). Sr ferrite with formula $SrFe_{12}O_{19}$ is one of type permanent magnet materials based on ferrite, and it is called as ceramic magnet. The Sr ferrite has hexagonal closed pack crystal structure and this magnet has large magneto ainisotropic, stable and good corrosion resitance also has high curie temperature about 450ºC (Nowosielski et al., 2007; Slusarek & Zakrzewski, 2012). The magnetic properties of permanent magnet based on ferrite is still lower than other types of permanent magnet, but the raw materials cost and manufacturing cost of ferrite magnet are lower than other types of permanent magnet, because the main raw materials such as hematite (Fe_2O_3) are abundant in nature. Among the class of permanent magnet materials the ferrite magnet are very important due to its moderate magnetic properties at lower cost. Therefore ferrite magnet is still used until now and many of the researches in this field are conducted to improve the physical and magnetic properties (Mahmood & Abu-Aljarayesh, 2016; Slusarek et al., 2013). The reaction mechanism of $SrFe_{12}O_{19}$ formation is through the solid reaction mechanism and there are two steps of forming reaction, as follow (Pullar, 2012):

$$SrO + Fe_2O_3 ======= SrO.Fe_2O_3 \quad (1)$$
$$SrO.Fe_2O_3 + 5Fe_2O_3 === SrO.6Fe_2O_3 \quad (2)$$

The first reaction step is an intermediate phase formation reaction ($SrO.Fe_2O_3$) that takes place at

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high temperatures, then continued with formation of $SrO.6Fe_2O_3$or $SrFe_{12}O_{19}$ at higher temperature. Mechanism of formation reaction dependson temperature , particle size and homogeneity of mixing (Pullar, 2012). The high temperature plays an important role in the formation of $BaFe_{12}O_{19}$ phase. If the temperature reaction is too high, that can lead to grain growth and can influence magnetic properties (Pullar, 2012; Takahashi et al., 2012). Ferrite magnetic particle powder can be prepared through several techniques including through wet chemical process (such as a cooprecipitation , sol gel and Freeze drying) and through solid-solid mixing which raw materials are in solid compound (Ghobeiti-Hasab, 2014; Nowosielski et al., 2008). The preparation of ferrite magnet using solid-solid mixing is called also a powder metallurgy process (Nowosielski et al., 2008). The powder metallurgy process is a simple process and it is needed a simple equipment, cheap raw materials. Some of parameters such as impurity, particle size and homogeneity of mixing give effect on physical and magnetic properties also on crystal structure (Moosa, 2013; Nowosielski et al., 2008). This research was conducted to synthesis of Sr ferrite ($SrFe_{12}O_{19}$) using powder metallurgy technique and the purpose of this study was to determine the effect of combustion temperature variations on changes in crystal structure and magnetic properties.

2 EXPERIMENT WORKS

This research was done in preparation of Sr ferrite with formula $SrFe_{12}O_{19}$ by metallurgy method, Hematite (Fe_2O_3 E-Merck) and $SrCO_3$ (E-merck) were used as raw materials and mole ratio $SrO:Fe_2O_3$ = 1 :6 is applied for synthesis of Sr ferrite powder. The both raw materials were weighed and wet milled by using High Energy Milling (HEM) for 4 hours and used aquadest as milling media. After that, the sample was dried at 110ºC for 4 hours by using drying oven. The dried sample was analyzed by using DTA/TG to know the calcination temperature. According to DTA/TG curve, there are 3 peaks endothermic at 730ºC, 820ºC and 990ºC. After that , the sample was calcination at different temperature such as : 900 and 1000 ºC for 2 hours. The calcined samples were analyzedcrystall structure by using XRD, measured magnetic properties by using VSM.

3 RESULTS AND DISCUSSION

The mixed raw material after milling 4 hours using HEM was measured particle size distribution and the result is seen in Figure 1. Based on Figure 1, it can be obtained that the average particle diameter is determined at a cumulative point of 50 %, so that the average particle diameter value is 13.08 µm.

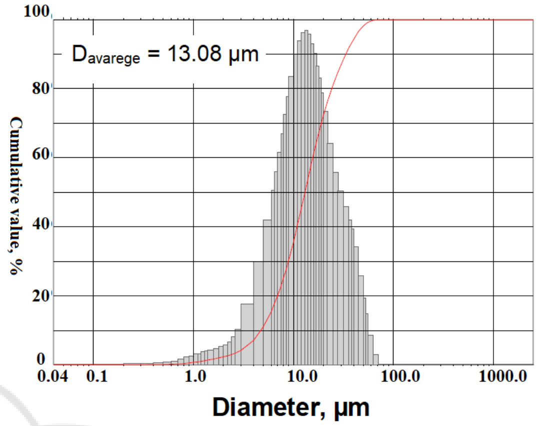

Figure 1: Particle size distribution curve after milling 4 hours.

The mixed raw material was measured using DTA/TG Analyser before calcination process and the result is seen at Figure 2.

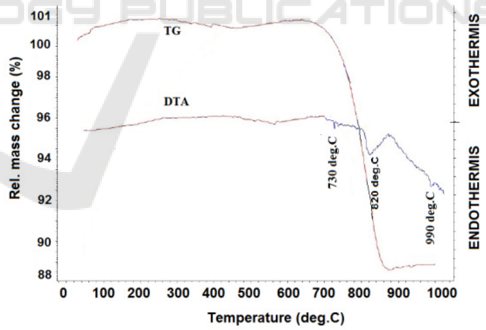

Figure 2: The DTA/TG curve of mixed raw material.

The mixed raw material consists of $SrCO_3$ and Fe_2O_3, according to DTA/TG curve , it was found that there are 3 peaks endothermic at temperature 730, 820 and 990 ºC. The reaction decomposition of $SrCO^3$ was occurred at temperature 730 and 820ºC to form SrO, which it indicates with sharply decreasing of mass from temperature 700ºC to 880ºC , then continued second reaction to form $SrFe_{12}O_{19}$ at temperature 990 ºC, where there are not mass change. Based on the DTA/TG results that the mixed raw material was conducted calcination using electrical furnace at temperature 900 °C and 1000°C. The XRD results of calcined samples are shown at Figure 3. The phase identification was done using Match soft ware through matching experiment data with reference data. The xrd result show that it is found two peaks namely hematite (Fe_2O_3) phase and $SrFe_{12}O_{19}$ phase at sample after calcination at 900°C. It is indicated that the starting of forming phase is at temperature 900°C, but the reaction of SrF formation at 900°C has not yet occurred completely.The xrd result of sample calcined 1000°C indicate single phase of $SrFe_{12}O_{19}$ with hexagonal crystal structure, in this case the hematite (Fe_2O_3) does not appear. The measurement of magnetic properties was done using Vibrating Sample Magnetometer (VSM) in evaluation of magnetic behaviour between xrd result with remanence and coercivity. The hysteresis loop from VSM is shown at Figure 4 for sample after calcination at 900 and 1000°C.

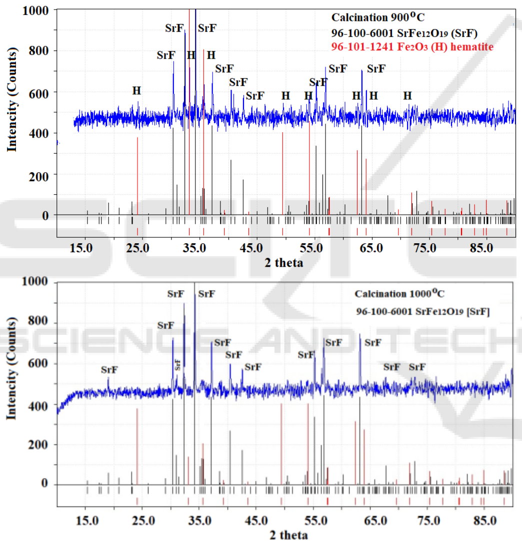

Figure 3: The XRD patterns of sample after calcination at 900°C and 1000°C.

The phase identification was done using Match soft ware through matching experiment data with reference data. The xrd result show that it is found two peaks namely hematite (Fe_2O_3) phase and $SrFe_{12}O_{19}$ phase at sample after calcination at 900°C. It is indicated that the starting of forming phase is at temperature 900°C, but the reaction of SrF formation at 900°C has not yet occurred completely.Thexrd result of sample calcined 1000°C indicates a single phase of $SrFe_{12}O_{19}$ with hexagonal crystal structure, in this case the hematite (Fe_2O_3) does not appear. The measurement of magnetic properties was done using Vibrating Sample Magnetometer (VSM) to evaluate magnetic behavior between xrd result with remanence and coercivity. The hysteresis loop from VSM is shown at Figure 4 for sample after calcination at 900 and 1000°C. Hysteresis loop curves as seen in Figure 4 for both samples show a hysteresis loop for permanent magnets material, which permanent magnet material has a wide hysteresis loop or has a coercivity value greater than 100 Oe.

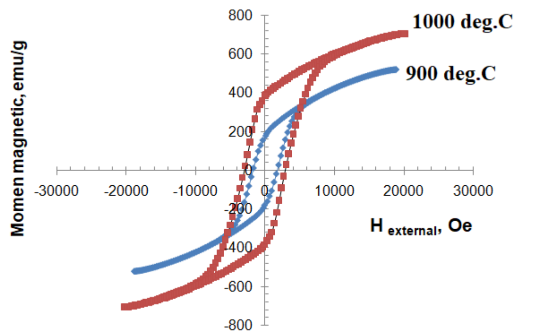

Figure 4: Hysteresis loop of samples after calcination at 900 and 1000°C.

Sample calcined at 900°C has magnetic properties (remanence, coercivity and magnetic saturation) lower than sample calcined at 1000°C, it is due to existing of hematite (Fe_2O_3) phase, which Fe_2O_3 is classified as soft magnet materials and sample calcined 1000°C has only single phase of $SrFe_{12}O_{19}$, The value of remanence, coercivity and magnetic saturation for both samples are shown at Table 1.

Table 1: Value of remanence (mr), coercivity (Hcj) and Magnetic saturation (ms).

Sample	mr, emu/g	Hcj, Oe	Ms, emu/g
Calcination 900°C	180	1900	500
Calcination 1000°C	390	3000	715

The highest value of magnetic properties is achieved at sample calcined at 1000°C i.e. the value of mr = 390 emu/g, Hcj about 3000 Oeand ms = 715 emu/g, because this sample has a single phase $SrFe_{12}O_{19}$. If the coercivity value from this experiment (3000 Oe or 239 kA/mm) compared to the theoriticalcoercivity for Sr ferrite is slightly lower (Slusarek & Zakrzewski, 2012).

4 CONCLUSSION

Magnetic powder of $SrFe_{12}O_{19}$ with hexagonal crystal structure can be made by powder metallurgy technique which the single phase of $SrFe_{12}O_{19}$ is achieved at calcination temperature about 1000^oC. Magnetic powder $SrFe_{12}O_{19}$is as a permanent magnet materials with remanence value about 390 emu/g, coercivity value about 3000 Oe and magnetic saturation about 715 emu/g.

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