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Effect of Heat Treatment on The Crystal Structur, Electrical Conductivity and Surface of $Ba_{1.5}Sr_{0.5}Fe_2O_5$ Composite

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Abstract. The Composite of $Ba_{1,5}Sr_{0,5}Fe_2O_5$ has been synthesized by using powder metallurgy technique. The $Ba_{1.5}Sr_{0.5}Fe_2O_5$ were prepared from $BaCO_3$, $SrCO_3$ and Fe_2O_3 raw materials with a specific weight ratio. The three materials were synthesized by powder metallurgy under heat treatment at 800 °C, 900 °C, and 1000 °C for 5 hours. All the three samples were characterized by using X-ray Diffraction (XRD) to determine the crystal structure and crystal size, LCR meter to determine the conductivity, and Scanning Electron Microscope (SEM) to observe the morphological of the composites. The phase analysis result showed that the composite consists of several minor phases such as BaO_2, SrO_2, and Fe_2O_3. The Crystal size of composite $Ba_{1.5}Sr_{0.5}Fe_2O_5$ decreased while increases the strain of crystal with increasing of sintering temperature. The crystal size of the $Ba_{1.5}Sr_{0.5}Fe_2O_5$ composite is 3.55 nm to 7.23 nm and value of strain is 8.47% until 3.90%. Based on the conductivity measurement, it was obtained that the conductivity of the $Ba_{1.5}Sr_{0.5}Fe_2O_5$ composite decreased with increasing sintering temperature. It was also noticed that the conductivity increased with increasing of frequency. The conductivity ranged from 6.619×10^{-7} S/cm to 65.659×10^{-7} S/cm. The energy dispersive spectroscopy (EDS) analysis showed that several dominant elements were a good agreement with the phase analysis.

Keywords: Composite, $Ba_{1.5}Sr_{0.5}Fe_2O_5$, X-ray diffraction, conductivity, surface.

1. Introduction

The magnetic properties to determine with characteristic include field strength magnet, the magnetic moment is saturated and has a stable chemical properties [1-3]. Impurity phases within the magnet depending on the synthesis process and are associated with homogeneity and heat treatment [4,5]. Ferrite magnetic material contained in the iron in the form of iron oxide (Fe_2O_3) called hematite and magnetite (Fe_3O_4). Hematite is used as a raw material while the hard magnetic magnetite as soft magnetic materials [6]. Effect of temperature on the magnetic properties and crystal structure and electrical properties have been studied [7]. Properties of ionic conductivity in solids as well as the theoretical approach of the ionic conductivity [8,9].

The synthesis of the $Ba_{1.5}Sr_{0.5}Fe_2O_5$ magnetic composite so far has been proposed to study the materials for an electromagnetic absorber, electrical conductivity, crystal size and surface properties. These properties are very useful for the designing for an absorber of electromagnetic especially for of $Ba_{1.5}Sr_{0.5}Fe_2O_5$ magnetic composite. So far, many reports of $Ba_{1.5}Sr_{0.5}Fe_2O_5$ related compound can be found in the literature [6-9]. Unfortunately, a comprehensive study of this compound as a function of

weight ratio, sintering temperature, porosity, and electrical conductivity, as well as the absorbtion response of electromagnetic wave have rarely been informed. Therefore, we proposed to synthesize a composite of $Ba_{1.5}Sr_{0.5}Fe_2O_5$. The preparation of the composite was also proposed by utilizing of powder metallurgy method from preparing from several starting materials i.e. Fe_2O_3, $BaCO_3$, and $SrCO_3$. Furthermore, in this work, we discuss the structure and electrical properties of the composite.

2. Experimental Method

The synthesis of composite $Ba_{1.5}Sr_{0.5}Fe_2O_5$ was conducted by using powder metallurgy method. The materials used were $BaCO_3$ (Aldrich 99.9% purity), $SrCO_3$ (Aldrich 99.9% purity), and Fe_2O_3 (Aldrich 99.9% purity). The weight compositions of the starting materials were $BaCO_3$ = 6.300, $SrCO_3$ = 0.845, and Fe_2O_3 = 2.855 g. The chemical equation for the composite is:

$$(2\text{-}x)BaCO_3 + (x)SrCO_3 + Fe_2O_3 \rightarrow Ba_{(2-x)}Sr_{(x)}Fe_2O_5 + 2CO_2$$

The starting materials consist of $BaCO_3$, $SrCO_3$, and Fe_2O_3 were mixed into a container vial and milled for 2 hours. After milling process, the materials were heated for 5 hours at a temperature of 800, 900, and 1000 °C. The samples were characterized using X-ray diffractometry to investigate the phase and crystallite size. Using LCR-meter and SEM, the conductivity and morphology of the samples were respectively characterized. Finally, the electrical properties of the samples were investigated by using LCR-meter.

3. Results and Discussion

3.1. X- Ray Diffraction of $Ba_{1.5}Sr_{0.5}Fe_2O_5$

Figure 1 is the X-ray diffraction peak of the $Ba_{1.5}Sr_{0.5}Fe_2O_5$ composite with variation sintering temperature. The identification results in Figure 1 looks that the sample consists of other small phases of BaO, SrO, and Fe_2O_5. The diffraction peaks of $Ba_{1.5}Sr_{0.5}Fe_2O_5$ composite then analyzed by using Igor Lorentzian program. The result of the analysis was the Brag's position (2 θ) and full width half maximum (β) as depicted in Table 1.

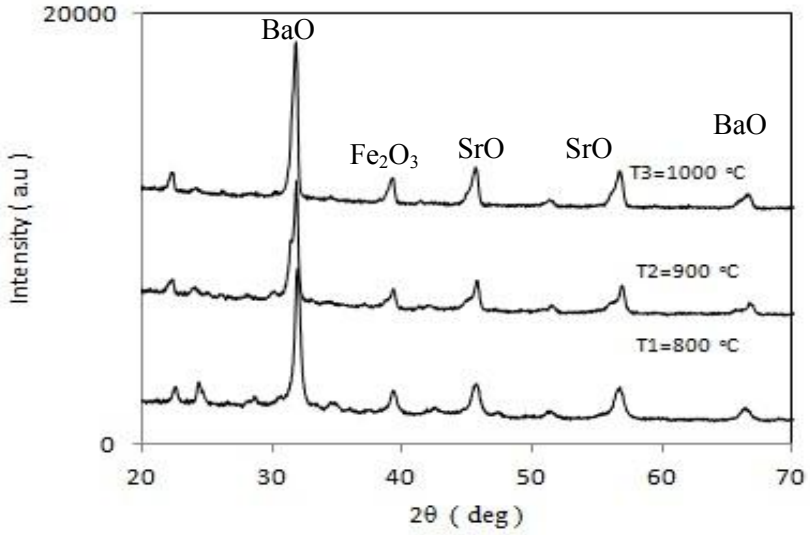

Figure 1. Phases identification of $Ba_{1.5}Sr_{0.5}Fe_2O_5$ X-ray diffraction peak.

Table 1. The calculated of 2θ and full width at half maximum (FWHM) on the composite of $Ba_{1,5}Sr_{0,5}Fe_2O_5$.

T_{sinter} (°C)	2θ (°)	β (rad)	sin θ	$\beta \cos \theta$
800	22.270	0.083	0.193	0.082
	31.761	0.106	0.274	0.102
	39.159	0.168	0.335	0.159
	45.509	0.320	0.387	0.295
	56.560	0.149	0.474	0.132
	66.310	0.256	0.547	0.215
900	22.255	0.026	0.193	0.026
	31.818	0.078	0.274	0.075
	39.300	0.048	0.336	0.046
	45.731	0.078	0.389	0.072
	56.887	0.047	0.476	0.042
	66.754	0.195	0.550	0.163
1000	22.320	0.038	0.194	0.038
	31.794	0.058	0.274	0.055
	39.199	0.095	0.335	0.089
	45.607	0.089	0.388	0.082
	56.688	0.133	0.475	0.117
	66.479	0.094	0.548	0.079

Table 2. Crystal size (D) and lattice strain (η) of $Ba_{1.5}Sr_{0.5}Fe_2O_5$.

T_{sinter} (°C)	D (nm)	η (%)
800	3.55	8.47
900	5.66	6.42
1000	7.23	3.90

From Table 1, it can be transformed curves between ($\beta \cos \theta$) against sin θ as shown in Figure 2. By using a model of Williamson-Hall [10] as written in Equation (1), it can be calculated a value of crystal size and strain.

$$B(\cos\theta) = 0.9\lambda / D + 4\eta \sin\theta \quad (1)$$

Where D is the crystal size, and η is the strain of crystal. From the curve, we calculated the crystal size and lattice strain. The results are shown in Table 2.

By using Equation 1, the crystal size size and lattice strain can be obtained by a straight line which is the slope of the line is a constant strain crystal and crystal size. It is seen that the crystal size increases with the increase of sintering temperature.On the other hand, the lattice strain decreases with the increase of sintering temperature. The increase of the crystal size effected by sintering temperatures indicated by the growth of granularity. We can expect that the conductivity may increase the grain size also increase. This phenomenon may depict as lowering the pore size. The lattice strain reduced as an increase of sintering temperature affected to the space between the grains get smaller. This is also driven to better electrical conductivity. Empirically, it is difficult to obtain information about the relation between the size and strain to the electrical conductivity of the compound.

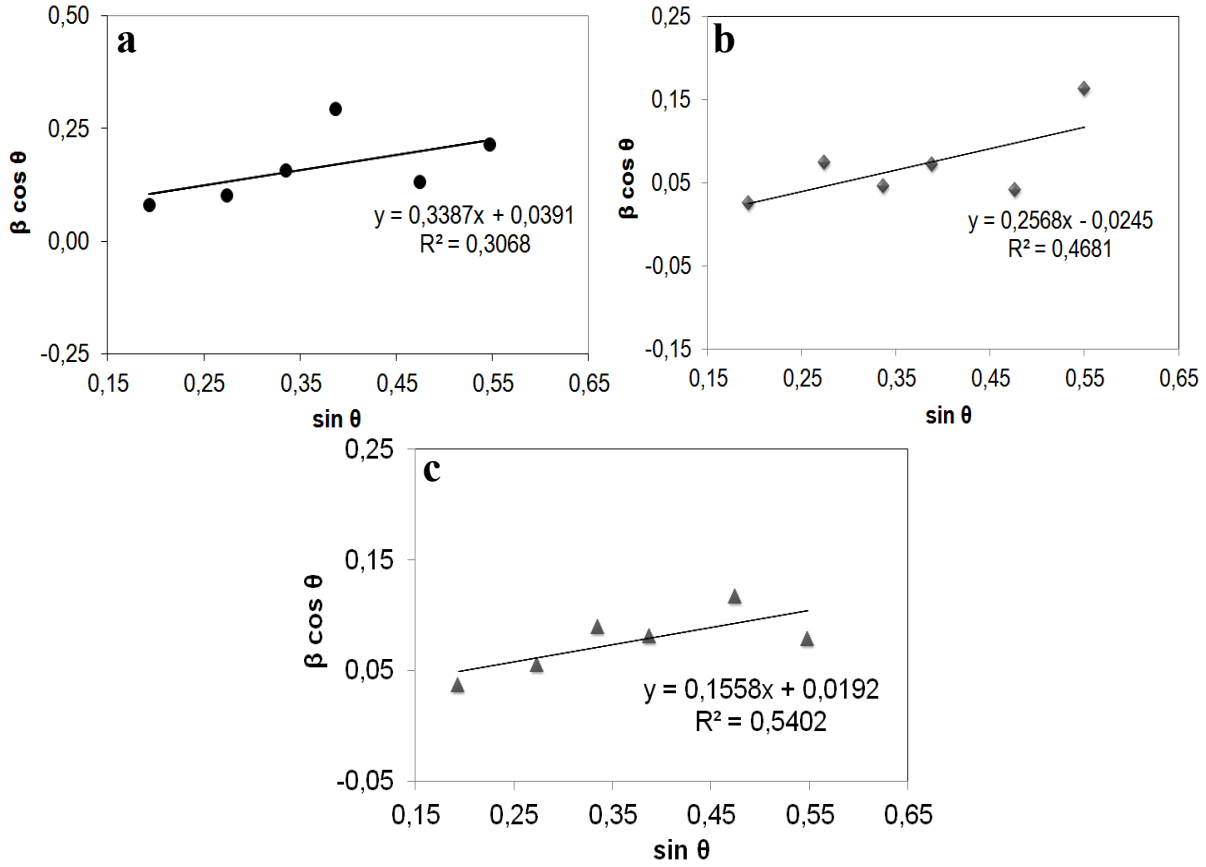

Figure 2. Relation curve of (β cos θ versus sin θ on composite of $Ba_{(1.5)}Sr_{(0.5)}Fe_2O_5$ with various sintering temperature (a) T_1 = 800 °C, (b) T_2 = 900 °C, and (c) T_3 = 1000 °C.

3.2. Conductivity of $Ba_{1.5}Sr_{0.5}Fe_2O_5$.

Curves conductivity on the composite of $Ba_{1.5}Sr_{0.5}Fe_2O_5$ is shown in Figure 3. From Figure 3, then calculating value conductivity of $Ba_{(1.5)}Sr_{(0.5)}Fe_2O_5$ by using a model [9], with a equation:

$$\sigma = \sigma_0 f^s \tag{2}$$

where σ is a conductivity (S/cm), σ_o is conductivity that does not depend on the frequency and s is an exponential factor. From Equation (2), it can be converted to the logarithm form, as in Equation (3)

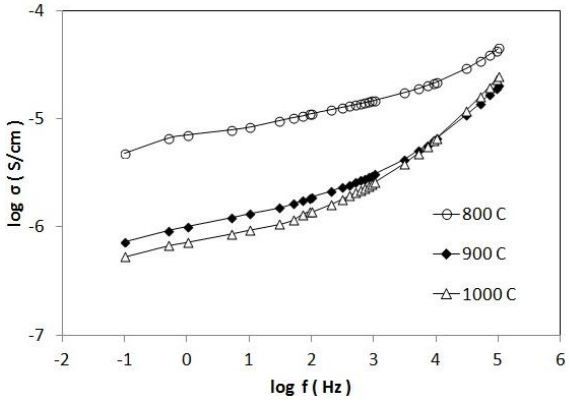

Figure 3. Conductivity value of $Ba_{1.5}Sr_{0.5}Fe_2O_5$.

Table 3. Electricl conductivity of $Ba_{1.5}Sr_{0.5}Fe_2O_5$.

$T_{sintering}$ (°C)	σ_0^a (S/cm)
800	65.659×10^{-7}
900	9.399×10^{-7}
1000	6.619×10^{-7}

σ_0^a = conductivity at the frequency of 0 Hz

$$\log \sigma = \log \sigma_0 + s \log f \quad (3)$$

By using Equation (3), it can be calculated the electrical conductivity of $Ba_{1.5}Sr_{0.5}Fe_2O_5$. The result is shown in Table 3.

It is observed from Table 3 that the electrical conductivity of the composite is 6.619×10^{-7} S/cm until 65.659×10^{-7} S/cm. The conductivity decrease with increasing of sintering temperature. It is indicated in Figure 3 that the conductivity of the composite $Ba_{1.5}Sr_{0.5}Fe_2O_5$ increase with increasing frequency. Conductivity at an interval to begin frequency of 0.1 Hz to 1000 Hz, this shows the conductivity of $Ba_{1.5}Sr_{0.5}Fe_2O_5$ is dependent on the frequency. In the sintering process of thermal energy to be absorbed by the atoms, so that the vibration occurs between atoms. The thermal energy influences the arrangement of the grains, so that it can affect the diffusivity arrangement and homogeneity of the material during heat treatment process.

3.3. The surface of $Ba_{1.5}Sr_{0.5}Fe_2O_5$.

The surface morphology of $Ba_{1.5}Sr_{0.5}Fe_2O_5$ with magnification at 10.000×, shown in Figure 4.

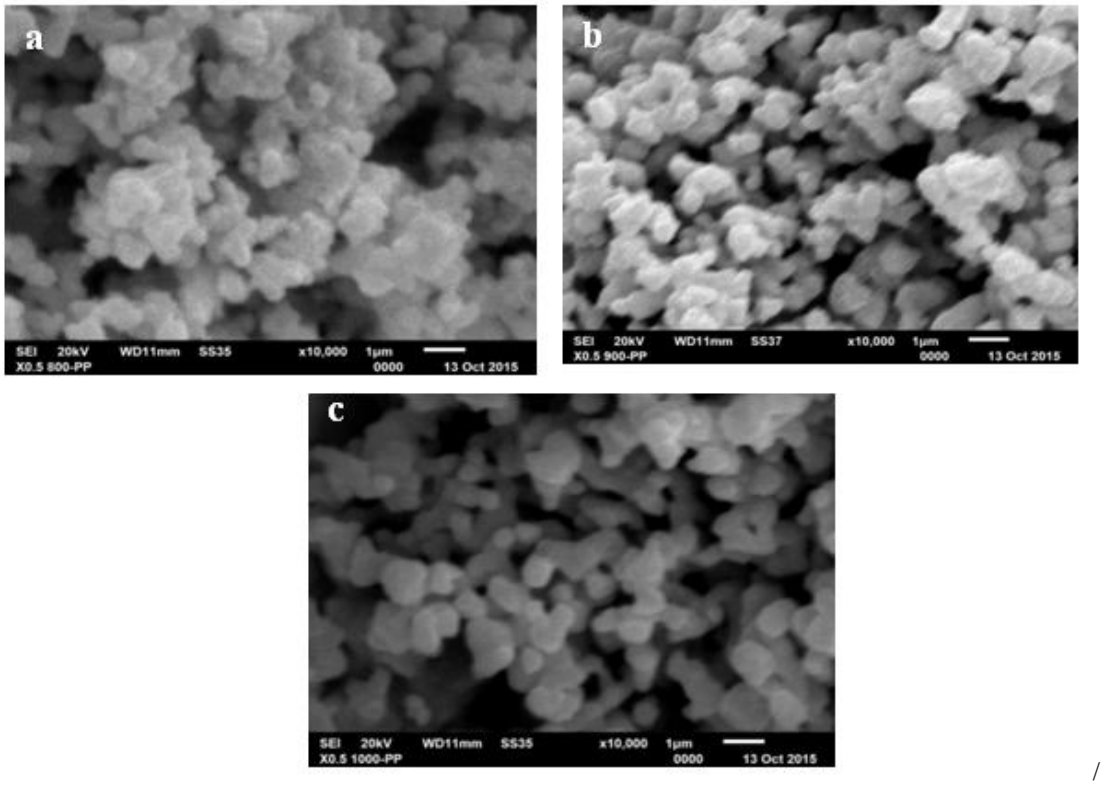

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Figure 4. Surface of $Ba_{1.5}Sr_{0.5}Fe_2O_5$ and Spectrum EDS with various sintering temperatures (a) T_1 = 800 °C, (b) T_2 = 900 °C, and (c) T_3 = 1000 °C.

Table 4. Analysis element EDS of $Ba_{1.5}Sr_{0.5}Fe_2O_5$ (% weight).

T_{sinter} (°C)	Fe	Ba	Sr	C	Cu
800	19.07	54.34	9.51	3.56	3.90
900	20.46	52.49	11.31	4.36	1.89
1000	21.94	56.87	9.38	2.56	1.96

The surface morphology of composite $Ba_{1.5}Sr_{0.5}Fe_2O_5$ is shown in Figure 4. The morphology of granules of the samples transforms to more ordered as the increase of sintering temperature. These SEM images showing the crystallinity of the samples also increase as the increase of the sintering temperature. This data clearly support the spectra of XRD patterns as depicted in Figure 1. As we cannot measure the shape of the grains quantitatively, so it is insignificant to relate of the conductivity to the surface morphology. The electrical conductivity is better described by the size and the strain of the crystal than to its morphology.

The result of EDS spectrum of all sintering temperature samples is presented in percent weight. Analysis of EDS spectrum of the samples can be obtained from the original data, and the results are shown in Table 4 excluded oxygen content. It is observed from Table 4 that increasing of sintering temperature increase Fe element but changes irregularly for other elements.

4. Conclusion

The composite of $Ba_{1.5}Sr_{0.5}Fe_2O_5$ has been synthesized by using powder metallurgy technique. The analyses of XRD spectra showed that the are other phases formed e.g. BaO_2, SrO_2, and $Ba_2Fe_2O_5$. The $Ba_{1.5}Sr_{0.5}Fe_2O_5$ composites revealed that the crystal size increase while the strain decrease with increasing of sintering temperature. The observation of the electrical measurement by LCR meter indicated that the conductivity decrease with increasing sintering temperature. On the other hand, the conductivity increase with increasing frequency. The changes of surface morphology of the $Ba_{1.5}Sr_{0.5}Fe_2O_5$ composite which is mainly originated from it grains is not significantly affected by sintering temperature. From these results, it can be concluded that heat treatment (sintering temperature) on the composite of $Ba_{1.5}Sr_{0.5}Fe_2O_5$ can affect the crystal size, strain, electrical conductivity, and surface morphology.

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