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Microstructure and Hysteresis Analysis of Ba - Hexaferrite

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Abstract- The single-phase Z-type Ba hexaferrite were elegantly synthesized by Co-precipitation method at 900°C and phase identification of powder were confirmed by X-ray diffraction method with lattice parameter a = 5.883 Å, c = 52.972 Å resembles with standard data. SEM gives the surface morphology of the prepared compound conformed hexaferrite structure of the particles. Magnetic study was carried out by hysteresis loop tracer and obtained low coercivity shows Zn_2Z powder may be used in information storage recording media. Z-type hexaferrite is high temperature sintering material. Here chemical methods is used to reduce this temperature, thus this compound can be used for modern cost-driven applications to form inductors,chips beads, LC filters, beads arrays etc.

Keywords- Co-precipitation, Z-type hexaferrite, Low temperature sintering, SEM, Hysteresis, Coercivity

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1. Introduction

Now a days, rapid information culture has been changing and various electronic devices, mobile phone, portable computer, have been dominant and becoming cordless. This required improvements in the device integration technology as well processing signals with much higher frequency in the range of MHz to GHz). Electromagnetic interference from such devices must be also in GHz region. Z-type hexaferrite $Ba_3Co_2Fe_{24}O_{41}$ is a promising EMIabsorbent material because of its high permeability even at high-frequency region of GHz [1,2,3].

Though, it is hard to prepare the Z-type hexaferrite material as a single phase. Impurities of Y-($Ba_2Co_2Fe_{12}O_{22}$) and W- ($BaCo_2Fe_{16}O_{27}$) phases canform and lower resultant permeability as their intrinsic permeabilities are lower than Z-type hexaferrite [4].

Z type ferrites are the complex compounds in the family of hexagonal ferrites with magnetoplumbite structure and have complex spin re-orientation. Thus, it requires higher sintering temperature to prepared single phase Z-type hexaferrite by convectional method [5]. The generalformula of Z type hexaferrite is $A_3Me_2Fe_{24}O_{41}$ where A is a large divalent cation such as (Ba, Ca, Sr), Me is a small divalent transition metal cation such as (Ni, Zn, Co) etc. The complexity of structuremainly results from large radius of Ba^{2+} ions in comparable to O^{2-} radius, it prefers the oxygen position rather than the interstitial site. Metal ions (Fe^{3+}, Fe^{2+}, Co^{2+} and Cu^{2+}) are located at non- equivalent interstitial sites.

In present paper, we are presenting a Z-typehexaferrite prepared at relatively low temperature bychemical method and its microstructural and magnetic properties.

2. Methodology

Here, BaZ type hexaferrite is prepared by co-precipitation method. Chemicals used are $BaCl_2 2H_2O$, $FeCl_3 6H_2O$, $Zn(CH_3COO)_2$, $Ba(CH_3COO)_2$, and NaOH. Using a magnetic stirrer, the solutions were made by dissolving $BaCl_2$ and $FeCl_3$, $Zn(CH_3COO)_2$ and $FeCl_3$, $Ba(CH_3COO)_2$, ingredient in 50 ml of distilled water. NaOH was used as a precipitating agent. A, B, and C-precursorare formed and combining to obtain a mixture precipitate. This precipitate was then wash with ethanol & dried at 60^0C.

To prepare pellet, the sample prepared is ground to fine particle size in an agate motor and mixed with 5% polyvinyl acetate solution made in AR grade acetone, as binder. About 2 ml of binder per gram of sample powder is found to give well- molded pellets. This compound is then transferred toa die and pressed under pressure of 5 tons per cm^2 using a hydraulic press. The pellets so prepared are then heated in a furnace up to 600ºC for 5 to 6 hoursto remove the binder. The temperature of the furnace is increased to 900ºC for 8 hours. After that the pellets are slowly cooled to room temperature. In this way crack free pellets of cylindrical shape of small height are obtained.

In the present work diffraction pattern wererecorded by using Philips PW – 1710 diffractometer. The Cu-K radiation with wavelength 1.54056 A^0 was used. The sample was scanned within the ranges 2θ between 10^0-100^0. The result gives the chart counts per second Vs diffraction angle 2θ. For the scanning electron microscopy (SEM), a Philips Cambridge Stereo scan was used to determine the morphology of barium hexaferrite particles. Magnetic studies were studied by Hysteresis loop tracer model HL-T-111.

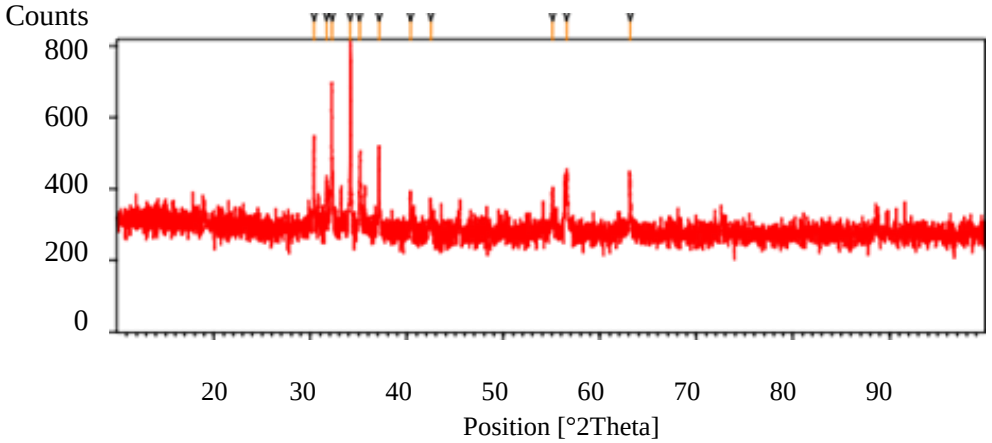

Figure 1. X-ray diffraction pattern of $Ba_3Zn_2Fe_{24}O_{41}$ at 900°C

3. Result and Discussion

3.1 XRD

Usually, the pure Ba Z phase is hard to obtain usingthe conventional solid state-reaction method at relatively low sintering temperature. The synthesized powder is frequently a mixture of Z andM or Y type phase. Consequently, the co- precipitation synthesis process was used in the preset study. $Ba_3Zn_2Fe_{24}O_{41}$ diffraction data revels that Z-type single phase is formed which was also conformed with JCPDS file No. 19-0097. Lattice parameters a and c are found to be a = 5.883 Å and c = 52.972 Å. These values are in good agreement with the earlier reported value viz a = 5.88 Å while c = 52.31 Å (JCPDF 019-0097) for similar compound prepared by other techniques. On theother hand, the density of the sample reached 5.3102 g/cm^3 at 900°C corresponding to 99% of theoretical density (5.36g/cm^3) conforms the formation of Z- type hexaferrite. Average crystalline size wascalculated for the sample from X-ray diffraction datausing Scherrer's formula [1] is found to be 17.76nm.

3.2 SEM Analysis

The morphological analysis of Z-hexaferrite sample was carried out using SEM. SEM images of the $Ba_3Zn_2Fe_{24}O_{41}$ hexaferrite sample is presented inFig. (2) The surface morphology of the compound was found to be

of pallet form shows hexagonal crystallites are grown well. The observed grains arein wide range of size. The minimum grain size is 0.015609 µm while maximum size is 2.70556 µm. Average grain size of the sample is found to be 0.299µm. It is to be noted here that in the compound prepared at temperature 900ºC, the porosity arises which results in decrease in the density of the compound. Thus 100% theoretical density is not achieved during sintering process.

$$D = \lambda / \beta \cos\theta \quad \text{.....(1)}$$

Where, D – Crystalline size K – Scherrer Constant (0.9)

λ - X-ray wavelength (1.5418 Å)

β – Full width half maximum (FWHM)Θ – diffraction angle

Thus, the formation of compound was confirmed from X-ray diffraction studies which show the hexagonal structure hence it is agreed thatwhen the compound was prepared by co- precipitation method, XRD data is in perfectagreement with the compound prepared by other methods and the data investigated in past.

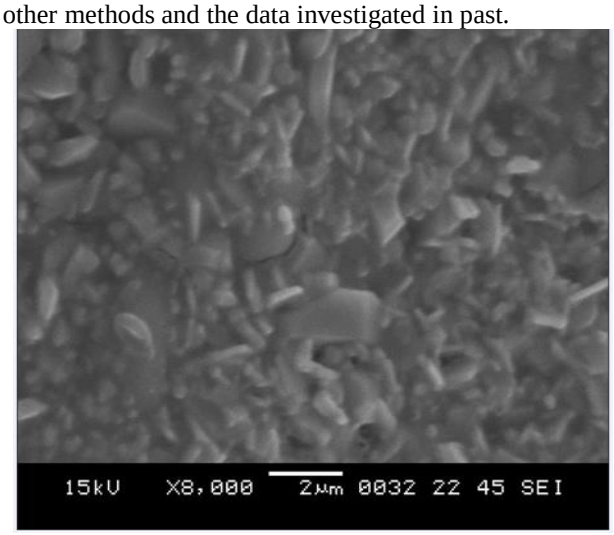

Figure 2. Morphology of $Ba_3Zn_2Fe_{24}O_{41}$ hexaferrite

3.3 Magnetic studies

To study the magnetic properties of the $Ba_3Zn_2Fe_{24}O_{41}$ hexaferrite prepared by co- precipitation method, we recorded hysteresis loop for the sample. It comes to our attention that the sample shows small hysteresis loop characteristics of soft magnetic material. High coercivity is required when one needs a hard magnet while low coercivity is required when one needs a soft magnet.The earlier is required for energy storage applications. The latter is required for information storage recording media. While coercivity of Zn_2Z powder is found 77.719 Oe *(HC)* which is low. This low coercivity shows Zn_2Z powder is magnetically soft. Saturation magnetization (μ_S) and Retentivity magnetization *(μ_r)* were obtained 1019.24 gauss and 566.246 gauss respectively for sample sintered at 900 °C.

The samples exhibited a Curie point (T_C) of 383°C above which hexaferrite materials lose their ferrimagnetic properties and behave like paramagnetic. The phenomenon is reversible.

Table 1. Hysteresis study of $Ba_3Zn_2Fe_{24}O_{41}$ Z type hexaferrite

Sample	Coercivity	Saturation Magnetization	Retentivity Magnetization
$Ba_3Zn_2Fe_{24}O_4$ 1	77.719Oe	1019.24 gauss	566.246 gauss

4. Conclusion

Here, single phase BaZ type hexaferrite is successfully prepared by co-precipitation method for first time with crystalline size 17.76 nm. SEM image shows hexagonal crystallites are grown well. Co-precipitation method found remarkable to reduce sintering temperature of Z type hexaferrite. 100% density was not achieved due to low sintering

temperature. Obtained hexaferrite showed low coercivity which is a characteristic of soft hexaferrite suggest the use of this material for information storage recording media in high frequency region.

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